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Analysis of Tensile and Compression Strength on Magnesium Hydroxyapatite Composite for Biomedical Implants

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Abstract: This paper focuses on the biomedical implants of bone plates with biodegradable Magnesium composites. ZK30 Mgalloy metal matrix reinforced with five weight fractions ofHydroxyapatite (HAP), have been fabricated using powder metallurgy manufacturing process followed by hot extrusion process. Properties such as Tensile and compression tests were investigated. Scanning Electron Microscopy (SEM) techniques were used for surface fracture analysis. The results obtained shows that the maximum ultimate tensile strength was attained at ZK30 Mg alloy while the least was noted for ZK30/10wt%HAP. In addition, ZK30/2wt%HAP shows a higher ultimate compressive strength. The SEM images of tensile specimen displays ductile fracture for ZK30 Mg alloy and quasi cleavage fracture for Mg composite whereas the compressive specimen indicates ductile fracture for ZK30 Mg alloy and Mg composites. The composite exhibited excellent mechanical properties thereby it can be used for biomedical implants of Bone plates.

Keywords: Magnesium, Hydroxyapatite, Powder Metallurgy, Mechanical Properties, Implants.

1. INTRODUCTION

Magnesium alloys have gained increasing attention for biomedical applications due to their biocompatibility and the biodegradability. Hydroxyapatite (HA) is known to be highly bioactive because of its similar chemical and crystallographic structures to bone [1]. The bone repair materials can be segregated into bio inert and biodegradable materialsbased on their process of degradation [2, 3]. Bio inert materials or biomedical metals and alloys are permanent implant materials which comprise metals such as stainless steel, cobalt chromium alloys, pure titanium and its alloys. Due to the high strength, toughness, ductility and corrosion resistance these metals are used as substituents for defective bones and load bearing

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implants. [4] Biodegradable materials are preferredto bio inert materials because of its degradation property resulting in the prevention of a second surgery. [8]Metals are preferred over polymers and ceramics, for their load bearing applications due to their high strength and resistance to fracture [9]. The use of various reinforcement materials alsoincreases the yield strength and compressive strength of the Mg metal. Varying the ratio of the reinforcement material reduces the possibility of implant failure[10]. Magnesium and its alloys have gained a superior locus in orthopaedic application because of their biodegradable character, high stiffness and specific strength, excellent dimensional stability and damping capacities [11, 12].The Young's modulus of Mg and its alloys is around (40GPa)closely similar to that of human bone(10-30GPa) and reduces stress shielding effect [5]. Moreover, bioactive materials are capable of depositing Ca-P compounds on the surface for increasing properties of matrix materials [6].Mg alloy composites can be effectively used as the best implant material if they include mechanical properties to help the damaged tissue;provided, the degradation rate is relative to the healing process with proper stabilisationof ions. Mg ZK30 alloy has good shock absorbing capacity since the percentage of elongation at fracture is 9 andalso it has non-toxic elements such as Zn and Zr[13, 14].

This research work aims to prepare Mg matrix composites composed of ZK30 matrix reinforced with HAPparticles of different composition using powder metallurgy manufacturing process followed by hot extrusion as secondary process. The mechanical properties such as Tensile and Compression tests of ZK30 Mg/HAP composite are investigated and proved that the mentioned composition is appropriate for load-bearing applications of biomedical implants. .

2. EXPERIMENTAL PROCEDURES

2.1 Materials and Methods

The matrix composition of ZK30 alloy consists of 3wt% Zn, 0.6wt%Zr, and balance Mg, which were processed from high purity (Mg>99.95wt%), (Zn>99.9wt.%) [14]. The ZK30 Mg alloy and composites comprising five samples of different wt.% of HAP were prepared. The powder metallurgy route was used to synthesize ZK30 Mg alloy, ZK30/2wt% HAP,ZK30/4wt%HAP, ZK30/6wt%HAP,ZK30/8wt%HAP,ZK30/10wt% HAP composites. The blending process was performed in V blender. Blended powders of 150 grams of each sample were transferred to a cylindrical compaction chamber and pressed gradually by a hydraulic vertical pressing machine with a capacity of 150 tons. Billet of 68mm diameter and 28mm length were obtained. A muffle furnace of automatic relay was used for sintering operation to maintain a temperature of 450^0Cfor an hour to have desired recrystallization of atomic bonding between the ZK30 Mg alloy and HAP of billet in an argon atmosphere. Once the bonding was completed, the billet was cooled within the chamber by free convection. A turn table apparatus was used to polish the cold specimen. The sintered billet was further processed for hot extrusion process by placing the billet in extrusion die lubricated with colloidal graphite and then heated to a temperature of 450^0C.Prior to the heating process the entire die was insulated by ceramic wool to avoid heat loss and oxidation. A hydraulic pressing machine with a capacity of 150 tons was used for this process. Through this secondary process (extrusion),a cylindrical rod of 12mm diameter and 500mm length was obtained.

2.2 Tensile and Compression Study

The compression test was performed on computer controlled universal testing machine (UTM TUE CN-600) at room temperature with 60-ton load capacity and specimen for each samples were prepared from extruded bars as per the ASTM E9 standard. Cylindrical specimens with 9.53mm diameter and 25.53mm length were preferred. For tensile test the ASTM standard E8 was followed toprepare the circular specimen of each samples with 4.75mm diameter and gauge length of 25.40mm.The compressions and tensile specimens were machined from extruded bars with the axis parallel to extrusion direction. Tensile test was performed on computer controlled universal testing machine (MTS insight 100 kN) at room temperature with strain rate of 1 x $10^{-3}s^{-1}$.The mode of failure occurred at the fracture surfaces of the broken samples when the tensile and compression test were determined using SEM technique. Stress strain diagrams were referred for obtaining tensile yield strength.

3. RESULTS AND DISCUSSIONS

3.1 Tensile and Compression study

The tensile stress strain curves of ZK30 Mg alloy and the composites with different wt.% of HAP are shown in Fig.1and the results at room temperature of tensile test are tabulated

in Table 1.The ultimate tensile strength of 271MPa was obtained for ZK30 Mg alloy whereas the minimum was recorded for ZK30/10wt%HAP with 186.1MPa. The ZK30/4wt%HAP shows higher tensile yield strength of 155.066MPa on comparison with ZK30 Mg alloy and the composites owing to uniform distribution of HAP particles. The ZK30 Mg alloy and Mg composites show an excellent percentage of elongation in the range of (3.2%-7%) improving the ductility and shock absorbing capacity of the material. It has been observed that the tensile strength of human bone varies between 90-190MPa [16]. The tested ZK30 Mg alloy and the Mg composites show higher tensile strength than human bone. Thus the results designate that on increasing the wt% of HAP particles the ultimate tensile strength, tensile yield strength and elongation of the material decreases. This tendency of decrease in tensile yield strength due to the addition of HAP was also observed [17].Moreover, in [18] when the wt.% of HAP was more than 10%, the agglomeration of HAP particles deteriorated the ultimate tensile strength as well as the tensile yield strength ensuing in pores and defects formation. The presence and magnitude of all the obstacles is being controlled by the yield stresses and operates the dislocation motion in the matrix [19]. The tensile strength of Mg based composite can be improved by using reinforcement particles with micron dimensions, but it affects plasticity [20].If the reinforcement particles size is reduced to nanometre scale, the tensile strength as well as the plasticity can be improved [21]. Fig.2(a-f)depicts the SEM observations of tensile fracture surface of ZK30 Mg alloy, ZK30/2wt%HAP andZK30/8wt%HAP composites. In [22] it was observed that the quasi cleavage fracture was the cause for failure of Mg alloys.Fig.2 (a,b)describes the formation of cleavage fracture as river like patterns with the presence of fine micro voids as dimple markleading to ductile

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fracture. The dimple formation occurs due to the lack of coalescence between micro voids [23]. The river like pattern with fine micro voids disappeared in Fig.2(c,d) and the particles agglomerated with secondary cracks owed to brittle fracture for ZK30/2wt%HAP composites. The agglomeration of particles increased in ZK30/8wt%HAP composites with secondary cracks, thus increasing wt% of HAP results in the extension of brittle fracture in region Fig.2(e,f). The addition of HAP particles to Mg could cause agglomeration and the formationof brittle fracture was observed [17, 24].

Fig.3 illustrates the compressive stress strain curves of ZK30 Mg alloy and the Mg composites with different wt% of HAP and the compressive test results are summarized in Table 2.The ZK30 Mg alloy shows a higher ultimate compressive strength of 365.165MPaon comparison with ZK30/4wt%HAP, ZK30/6wt%HAP, ZK30/8wt%HAP,ZK30/10wt%HAP. The compressive strength of ZK30/2wt % HAP 388.848MPa was greater than that of the ZK30 Mg alloy, which happens due to the excellent inter atomic bonding between the ZK30 Mg alloy matrix and the reinforcement. The compressive yield strength of natural bone is in the range of 130-180MPa [25]. The tested ZK30 Mg alloy and the Mg composites show compressive strength higher than human bone.

Thus the result indicates that the ultimate compressive strength of ZK30 Mg composite decreases on increasing wt.% of HAP above 2% due to inadequate transfer of load between the ZK30 Mg alloy matrix and the reinforcement. In general, the mechanical properties of composite can be improved by considering factors such as grain size and dislocations [26]. The

Presence of solid particles dispersed in matrix material can enhance the strength of composite [27]. Fig.4 (a-f) depicts the SEM observations of compressive fracture surface of ZK30 Mg alloy ZK30/2wt.%HAPand ZK30/8wt.%HAP.The presence of miniature formation of void nucleation in cleavage planes was observed in fracture surfaces of Fig.4 (a-b)due to ductile fracture. The straight lines in Fig.4 (c-f) represents the formation of river like patterns with presence of micro voids. The river like patterns are formed when nearest step combine to form a single step of higher height [28]. Furthermore, the micro void locking with river like patterns was observedinFig.4(c-d) of ZK30/2wt%HAP. The river like patterns with micro voids formation increased in Fig.4 (e-f) of ZK30/8wt%HAPlinked to ductile fracture. The fracture surface of ZK30 Mg alloy matrix is changed due to the introduction of HAP particles, and is responsible for decline in ultimate compressive strength of composites above 2% HAP. A similar observation of ductile fracture was also recognised in Mg composites [29].

4. CONCLUSION

The ZK30 Mg alloy and Mg composite containing HAP reinforcements were developed using powder metallurgy route followed by hot extrusion process for amalgamation. The maximum ultimate tensile strength was attained in ZK30 Mg alloy while the least was noted for ZK30/10wt%HAP.In addition, ZK30/2wt%HAP shows a higher ultimate compressive strength. However, the tested Mg alloy and the composites show higher tensile and

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compressive strength than human bone. The fractured surface of ZK30 Mg alloy and Mg composites in tensile test shows a ductile and brittle fracture while compression test displays a ductile fracture for ZK30 Mg alloy and Mg composites. Hence increased wt.% of HAP particles decreased the tensile strength and compressive strength of Mg composites. However, up to 4wt%HAP composites increased the above mechanical properties and this may be due to uniform distribution of HAP particles. The results clearly indicate that the ZK30 Mg alloy/HAP composites have excellent mechanical properties and are suitable for load bearing degradable biomaterial implants.

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Table.1Tensile test results

Material	**Tensile Yield Strength (MPa)**	**Ultimate Tensile Strength (MPa)**	**Elongation (%)**
ZK 30Mg alloy	146.627	271	7
ZK30/2wt% HAP	135.225	250	4.60
ZK30/4wt% HAP	155.066	212.1	4.80
ZK30/6wt% HAP	122.129	208	3.60
ZK30/8wt% HAP	143.792	195.5	3.20
ZK30/10wt% HAP	136.497	186.1	3.60

Table.2Compressive test results

Material	**Ultimate Compressive Strength (MPa)**	**Elongation (%)**
ZK 30Mg alloy	365.165	2.730
ZK30/2wt% HAP	388.848	3.430
ZK30/4wt% HAP	362.110	1.960
ZK30/6wt% HAP	326.204	2.660
ZK30/8wt% HAP	289.535	2.060
ZK30/10wt% HAP	252.102	2.470

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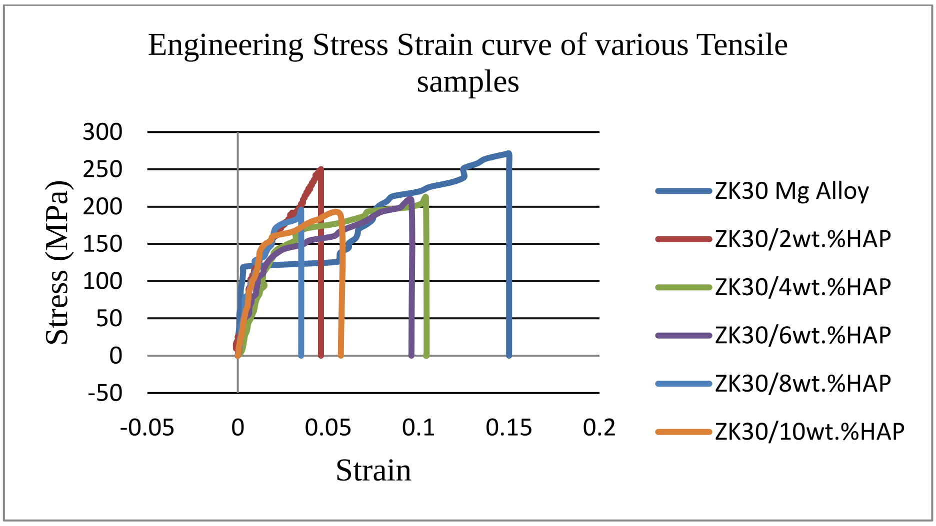

Fig.1 Engineering Stress strain curves of various tensile samples

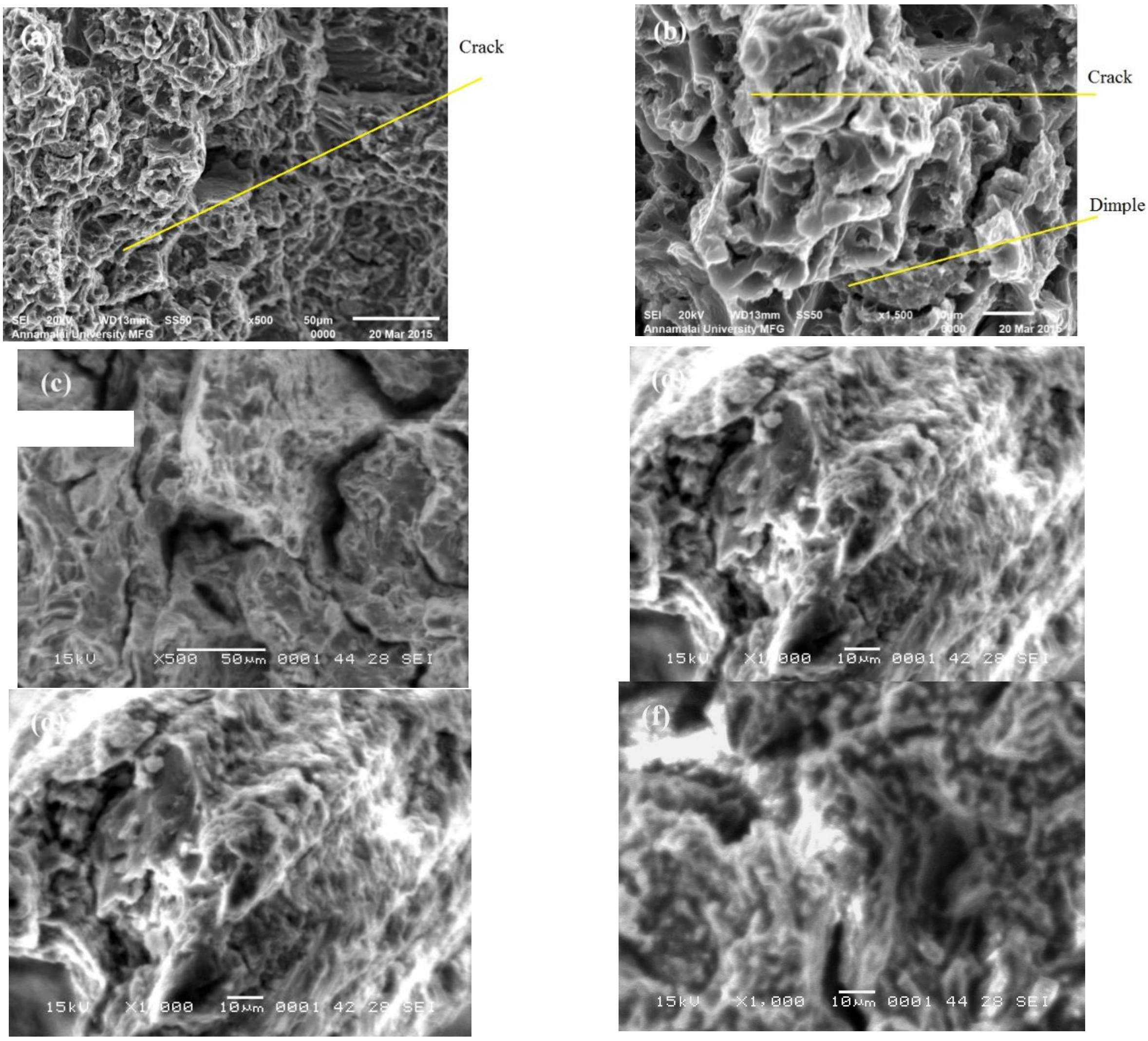

Fig.2 SEM Images of Tensile Fracture Surfaces of (a, b) ZK30 Mg Alloy, (c, d) ZK30/2wt%HAP, (e, f) ZK30/8wt%HAP

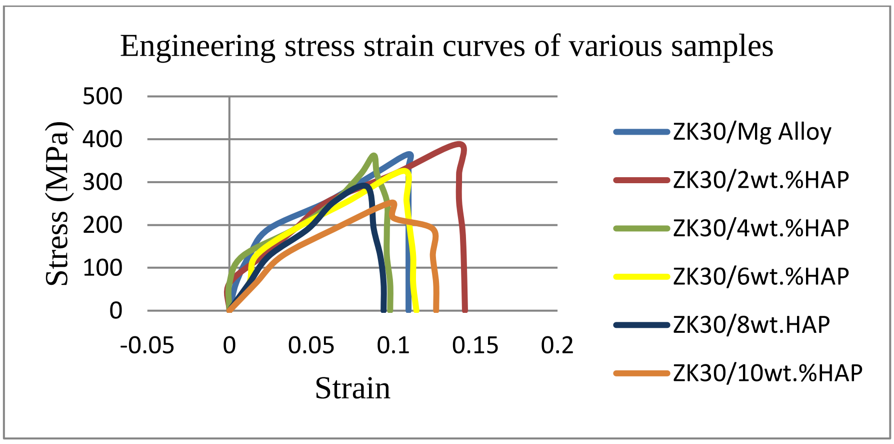

Fig.3Engineering Stress Strain curves of various compressive samples

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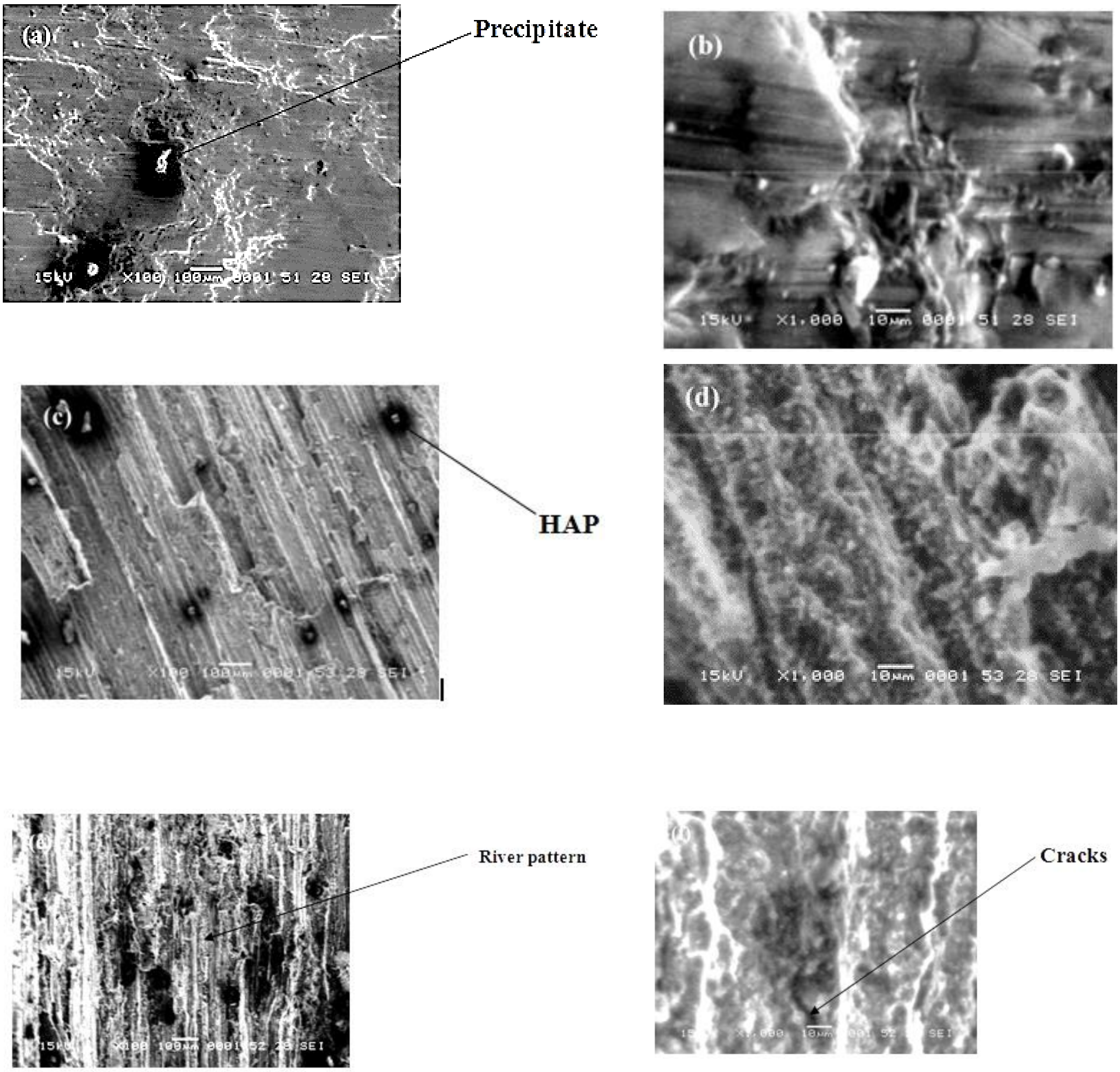

Fig.4 SEM Images of Compressive Fracture Surfaces of (a, b) ZK30 Mg Alloy, (c, d) ZK30/2wt%HAP, (e, f) ZK30/8wt%HAP