

Mechanical Properties of Powder Metallurgy Processed Biodegradable Zn-Based Alloy for Biomedical Application

Maruf Yinka Kolawole, Jacob Olayiwola Aweda, Farasat Iqbal, Asif Ali, Sulaiman Abdulkareem

Abstract—Zinc is a non-ferrous metal with potential application in orthopaedic implant materials. However, its poor mechanical properties were major challenge to its application. Therefore, this paper studies the mechanical properties of biodegradable Zn-based alloy for biomedical application. Pure zinc powder with varying (0, 1, 2, 3 & 6) wt% of magnesium powders were ball milled using ball-to-powder ratio (B:P) of 10:1 at 350 rpm for 4 hours. The resulting milled powders were compacted and sintered at 300 MPa and 350 °C respectively. Microstructural, phase and mechanical properties analyses were performed following American standard of testing and measurement. The results show that magnesium has influence on the mechanical properties of zinc. The compressive strength, hardness and elastic modulus of 210 ± 8.878 MPa, 76 ± 5.707 HV and 45 ± 11.616 GPa respectively as obtained in Zn-2Mg alloy were optimum and meet the minimum requirement of biodegradable metal for orthopaedics application. These results indicate an increase of 111, 93 and 93% in compressive strength, hardness and elastic modulus respectively as compared to pure zinc. The increase in mechanical properties was adduced to effectiveness of compaction pressure and intermetallic phase formation within the matrix resulting in high dislocation density for improving strength. The study concluded that, Zn-2Mg alloy with optimum mechanical properties can therefore be considered a potential candidate for orthopaedic application.

Keywords—Biodegradable metal, biomedical application mechanical properties, powder metallurgy, zinc.

I. INTRODUCTION

DEVELOPMENT of new biomaterial in orthopaedics implant for the restoration and correction of defected or fractured human hard and soft tissues are on the increase and has attracted research interest. This may probably be as a result of increasing accident rate, diseased bone, old ages and traumatic patient in Nigeria and other parts of the world.

Orthopedic biomaterials in form of internal implant are often used as remedies to structurally remodel, reconstruct and rehabilitate affected patients [1]-[3]. Conventional technique of osteosynthesis of the above complications usually involved the use of traditional bio-metallic materials such as CoCrMo, titanium, tantalum, gold and stainless-steel alloys [4] in the form of permanent bone plates and screws. This class of metallic implants has high corrosion resistance property and hence they remain stable without significant deterioration in physiological environments as permanent metallic implants (PMI) in patients [5]-[7]. PMI are characterized by high elastic

modulus (~200 GPa) which is far much higher than that of human natural bone (45 GPa) leading to stress shielding, chronic inflammatory response due to possible release of metallic ions from the implant whenever there is defect in surface oxides film [2]. Consequently, this defect may undermine the therapeutic function of the device which in most cases requires secondary surgery operation for removal of the implant [5]-[10]. Meanwhile, most bone implants in the form of plates, screw, pins, plugs and rods service functions are temporary and no longer required after the complete healing process. A novel approach to these nagging challenges is the use of biodegradable or bi-absorbable implants [8], [11]. Biodegradable implants permit the growth of new tissue in patient while gradually deteriorating harmlessly in the body at a rate matching the healing kinetics of the diseased bone without abrupt loss in mechanical integrity for complete healing of diseased bones [9], [11]-[13]. Till present, magnesium, iron and zinc base metals and their alloys constitute the newly research biodegradable metals (BDMs). The driving force for these metals was that they are all intrinsically corrosion susceptible and degrade safely in human body because they constitute essential bodily physiological mineral elements that partake in various metabolic and biological reactions in human body [14], [15]. However, due to unresolvable limitation of magnesium BDM in respect of its rapid corrosion rate associated with formation of large amount of hydrogen gas and premature loss of mechanical integrity in bodily fluid (*in vitro* and *in vivo*) degradation process resulting in wound interface cavitation and tissue necrosis had been a nagging challenge [16], [17].

Interestingly, zinc with electrode potential of -0.763 V has more desirable degradation rate when compared to magnesium and iron in a physiological environment. Moreover, zinc is an essential element for human beings required to degrade with various important physiological roles. In addition, it serves as a co-factor in six classes of enzymes, participates in nucleic acid metabolism as well as gene expression, wound healing, protein regulations and signal transduction in the body [9], [18], [19]. However, poor mechanical properties below the required benchmark of biomedical application of pure zinc have been its major limitation.

Several techniques such as casting, extrusion, rolling and alloying approaches with different metals have been explored to improving mechanical properties of zinc and its alloys. However, work on powder metallurgy processing of Zinc alloys are limited and rarely found in literature. Powder

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metallurgy (PM) technology of metal is a promising processing route that is continually and rapidly growing for the processing of various shapes of metal alloys and composites [20]. It is a high precision metal forming route resulting in near net pieces, cutting-off the extra cost of machining as experienced in the case of casting technique [20], [21]. One chief advantage of this technique was its flexibility giving room for easy tailoring of product properties to suit a specific application. Considering the dynamic parameters of material properties clinically required, PM glows out as a promising technique for the processing of BDM. Until present, work on PM processing of biodegradable zinc alloys and composites are rarely found in literature and are limited. Apart from the recent work of Krystýnová et al. [22] and Bagha et al. [23] on characterization of pure zinc and Zn-Mn alloy prepared by PM technique, no other work has been found on zinc based alloys to the best of the author's knowledge. In addition, most reported work on Zn-Mg alloy prepared by other methods had failed to consider wider range of magnesium addition in zinc. But, findings from literatures [22], [23] show that PM is a promising route for the processing of biodegradable zinc metals. Hence, this paper reports the mechanical properties of PM processed biodegradable Zn-Mg alloy for biomedical application.

II. MATERIALS AND METHODS

A. Materials and Equipment

Zinc (44 μm) and magnesium (73 μm) powder, Ney Centurion Qex vacuum furnace, digital weighing balance, Caver hydraulic press, $\phi 50 \times 100$ mm die with $\phi 8$ mm cavity, Planetary ball milling machine with alumina jar and balls, grinding machine, Aspex 3020 scanning electron microscopy machine incorporated with energy dispersive spectroscopy (SEM/EDS), Empyrean X-ray diffractometer, Universal

Testing Machine (UTM) and Micro-hardness Testing Machine all at Interdisciplinary Research Centre on Biomedical Materials, Comsats University, Lahore, Pakistan were used in this study.

B. Processing and Production of Zn-Mg Alloys

Zinc and Zn-Mg alloys of 0-6 wt% magnesium powder were produced using PM process following the sequence illustrated in Fig. 1. Zinc of 99 wt% and 1wt% of magnesium powders were weighed on a digital weighing balance. The resulting weighed powder mixtures were filled in alumina jar and balls. The Zn-1Mg powder mixture was then milled using planetary ball milling machine with ball to powder ratio of 10:1 according to [24] and [25] at 350 rpm for 4 hours. The milled powder was compacted at 300 MPa for 180 s retention time using a 25 tons capacity auto-series Caver hydraulic press, Model No: 3887.4NE0000 and Serial No:150608. The as-compacted samples were de-lubricated at 200 °C for 4 hours followed by sintering at 350 °C and 4 hours soaking time in Qex Centrium vacuum furnace at 20 °C/min. The samples were cooled by quenching in stagnant water to avoid unnecessary phase formation. Apparent green and sintered density of Zn-Mg alloy samples were determined using Archimedes' principle according to ASTM B962-17 standard as given in (1). The relative density of respective samples was then calculated by dividing apparent density by theoretical density of the corresponding samples as obtained from rule of mixture as reported by [26] and expressed in (2). The same procedure was replicated for 2, 3 and 6 wt% magnesium in Zn with 100 wt% zinc serving as control.

$$\rho_a = \frac{w}{v} \quad (1)$$

$$\rho_r = \frac{\rho_a}{\rho_t} \times 100 \quad (2)$$

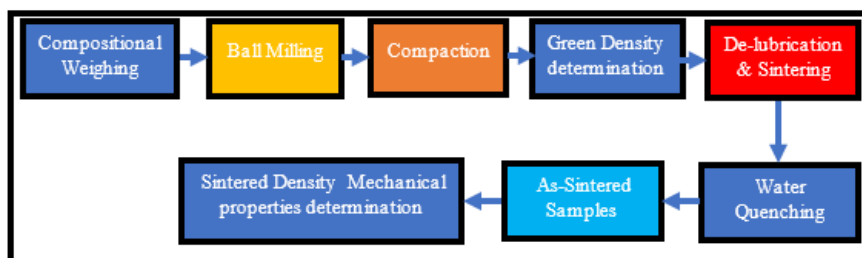


Fig. 1 Zn-Mg alloy production sequence

C. Mechanical Properties of Zn and Zn-Mg Alloy Determination

The compressive strength was determined according to ASTM E9-09 (2018) standard [27] with dimension of length (l) to diameter (d) ratio of 2, i.e. $\frac{l}{d} = 2$. The compression tests were performed in triplicates per variants using a Testometric 50 kN UTM machine at a constant cross head speed of 1mm/min with strain rate of 10^{-3} s^{-1} at room temperature as reported in [28], [29]. Elastic modulus and strain of respected samples were equally evaluated.

Micro-hardness property testing of as-sintered samples was performed using ASTM-E384-17 standard at a load of 100 kgf for 10s as reported by [24]. The average hardness value obtained from five indents on each sample was recorded as the hardness of each sample.

Morphology and phase examination were carried out as described in [5]. Microstructures of the prepared samples were carried on Vega3-XMU Tescan SEM/EDS (Oxford Instrument Inc, UK) after being etched in a solution containing 2% HNO_3 and 95% ethanol according to [30]. The phase identification of

the prepared sintered samples after ground up to 2000 grit size were examined on PANalytical X-ray diffractometer (XRD) using Cu α radiation generated at 40 kV and 30 mA at 0.02 step size on (1-5) s per step over a 2θ angular range of 5-100°. The phase identification was analyzed using X'Pert Highscore Plus software.

III. RESULTS AND DISCUSSION

A. Green and Sintered Density of Zn and Zn-Mg Alloy

Green and sintered densities of PM processed material are important parameters in determining the quality of samples produced [31]. For this purpose, the green and sintered densities of Zn-Mg alloy were examined and presented in this paper. Fig. 2 shows graph of green and sintered densities of Zn-Mg alloy at 0-6 wt% of magnesium compacted at 300 MPa. It was observed that, both green and sintered densities of Zn and Zn-1Mg alloy of 88.23, 89.71% and 91.08, 91.71% respectively were the highest in the alloy series. Above which, the green and sintered densities decreased with increase in magnesium addition. In all, there exists a marginal increase in sintered densities with respect to green densities as observed in Fig. 2. This increment may be added to densification of the fusion or bonded of alloy's grain due to sintering effect. The increased fusion of the grains collapsed and densified the inherited porosity and micro-pores from green compaction process to increase the sintered density. The marginal increase in sintered densities as noticed here was an indication of an efficient compaction process of the green samples with lesser porosity due to high relative green density [31]. Another factor that could be attributed to decrease in both densities as observed in this study was increase in magnesium percent in zinc bearing in mind that magnesium has a density of 1.41 g/cm³ compared to zinc with 7.14 g/cm³. Hence, increasing addition of magnesium in zinc may therefore may lead to a corresponding decrease in density of Zn-Mg alloy regardless of other parameters like compaction pressure and sintering temperatures. This result is similar and agrees with earlier findings [32], [33] where decrease in density of Al-alloy composites fabricated using PM were reported. Guleryuz et al. [33] attributed decrease in density to presences of oxides and porosity incurred in the sample during processing.

B. Microstructural and Compositional Phase Analysis

Microstructure and compositional phase analysis of Zn-Mg alloy were as illustrated in Fig. 3 and 4 respectively. Pure zinc samples possess single zinc eutectic phase (light-blue arrow) without any evidence of intermetallic phase formation (Fig. 3 (a)). But intermetallic phases were observed in all Zn-Mg alloys investigated. However, for Zn-1Mg and Zn-2Mg alloys, uniform and homogenous distribution of intermetallic phases (dark-blue arrow) without segregation of inter-metallic phases were observed. Also pores and micro-pores were not noticed in the microstructural morphology of the samples. This characteristic morphology features are indications of strength enhancer due to formation of strong bonded phases without stress raisers such as pores and delamination in the structure of

the samples. However, the addition of 3 and 6 wt% magnesium in zinc matrix not only resulted in intermetallic phases but also characterized with large lumps of soft magnesium particles (yellow arrow) (Figs. 3 (d) & (e)) filling up the entire matrix. This morphology may adversely affect the mechanical properties of the alloy by weakening the binding force and serving as stress raiser to which reduces the resistant to plastic deformation of the alloys.

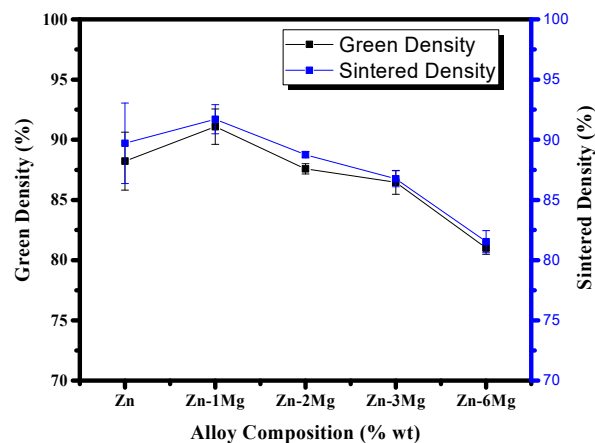


Fig. 2 Green and sintered densities of Zn-Mg alloys

Fig. 4 shows the XRD pattern of as-sintered zinc and zinc magnesium alloys. Only a single phase of pure zinc was observed in Fig. 4 while in Zn-Mg alloy, three phases were noticed. It was phase identification on Zn-Mg alloys revealed the presence of Zn eutectic-rich (light-blue arrow), Mg₂Zn₁₁ and MgZn₂ (dark-blue arrow) phases in the alloy's structure. These phases play an important role in the structure of the samples which in turn may increase the strength of materials by restricting the inter-atomic movement [23], [34].

C. Mechanical Properties of Zn-Mg Alloys

The mechanical properties of Zn-Mg alloy, with magnesium volume fraction ranging from 0-6 wt% were as illustrated in Figs. 5 (a)-(c). It was clearly shown that mechanical properties of Zn-Mg alloy depend on the volume fraction of magnesium content. Fig. 5 (a) shows graph of compressive strengths and strain against the wt% addition of magnesium. It was observed that the compressive strength increased from 99.67 MPa for pure zinc with increasing wt% of magnesium up to 210.23 MPa at 2 wt% Mg. This percentage increase in strength is equivalent to 111% with respect to pure zinc. Above this value, sudden reduction in compressive strength was noticed. meanwhile the maximum strain was observed at 1wt% Mg and after which it decreases with further increase in magnesium volume fraction. The increase in strength and strain as noticed in this study could be as a result of various factors such as homogenous dispersion of the magnesium in zinc matrix through the milling process as showed in Fig. 3. In addition, the formation of strong intermetallic phases as depicted in Figs. 3 & 4 can also be another contributing factor responsible for the increase in strength. However, further increase in magnesium volume fraction above 3 wt% leads to

corresponding decrease in strength and strain of Zn-Mg alloy. This could best be explained with the aid of microstructural and structural morphologies of Zn-Mg alloy as illustrated in Figs. 3 and 4 respectively. It will be observed that pure zinc has no intermetallic phases and hence low mechanical properties. But with addition of 1-2 wt% magnesium, homogenous distributed intermetallic phases were formed without any segregation within the phase and eutectic zinc matrix (Figs. 3 (a)-(c)) producing stronger bonds between different phases. The above observation constitutes the major factors responsible for the increase in strength as noticed in this study. However, formation of intermetallic phases with segregated lumps of magnesium particles within the eutectic zinc phase were conspicuously observed in Figs. 3 (d), (e). This segregation of different phases resulted in poor bonding and hence reduction in strength and strain as recorded in this

study. At the same time, inter-phase segregation can also be a source of stress raiser which reduces the resistance to fracture strength of the sample and hence lower strength. This result is in line to that reported by Kubasek et al. [35] where Zn-xMg alloys ($x = 0, 0.8$ and 1.6) were investigated. They reported that compressive strength increased with increasing magnesium volume fraction. The maximum compressive was recorded at Zn-1.6Mg meanwhile the peak elongation of Zn-Mg alloy was reached at 0.8 wt% Mg after which it decreases. Moreover, the results obtained by Vojtech et al. [36] on Zn-yMg ($y = 0, 1, 1.5$ & 3.5) indicated the highest strength at 1.5 wt%Mg while the lowest strength was recorded at 3 wt%Mg. The results of the present study show similar trend with optimum value of strength recorded at 2 wt% magnesium content in zinc.

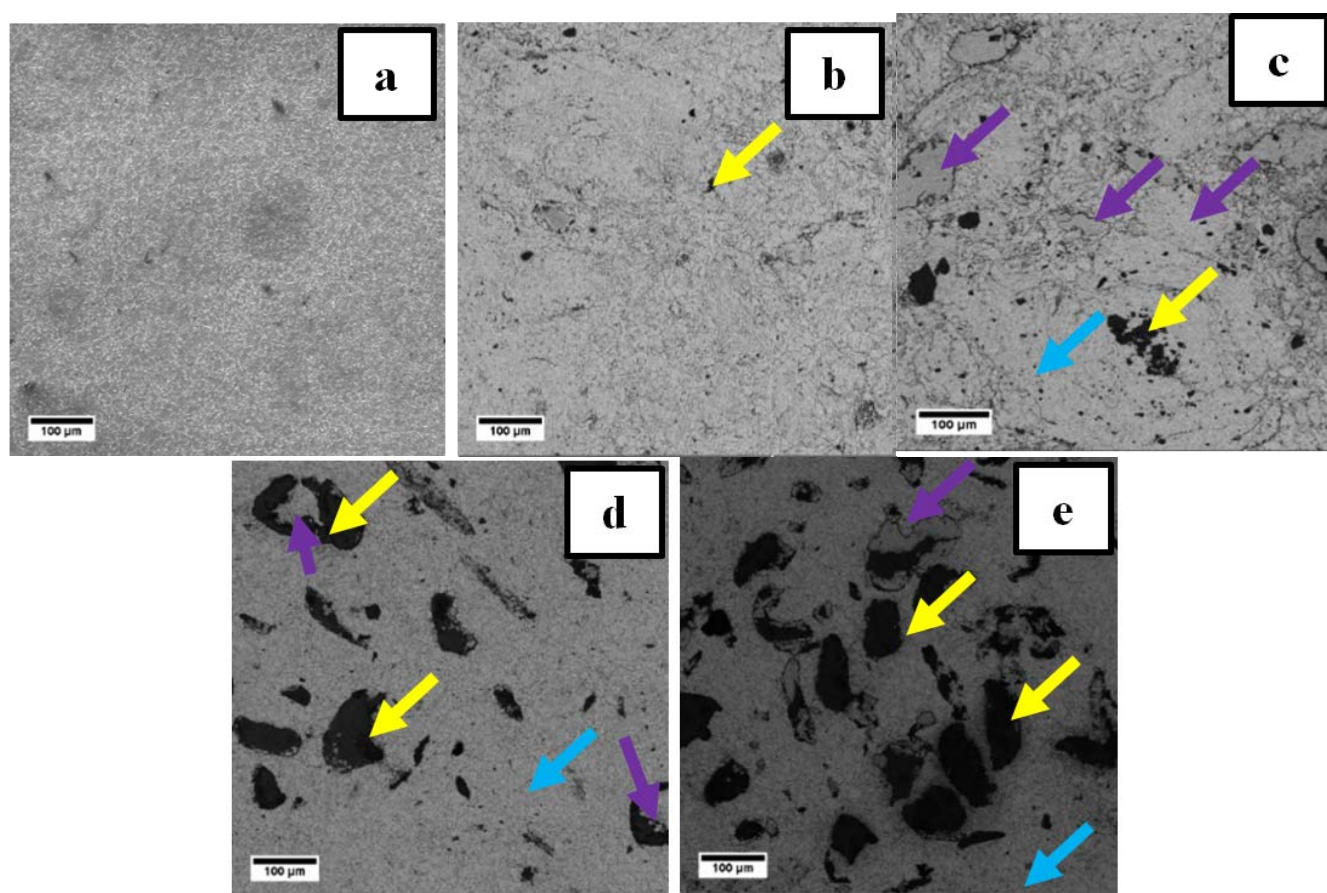


Fig. 3 Microstructure of Zn-Mg alloys

Fig. 5 (b) is a graph of elastic modulus against Zn-Mg alloy. This graph illustrated a parabolic behaviour of elastic modulus property with increase in volume fraction of magnesium. The elastic modulus increased from 24 ± 4.70 GPa at 0 wt%Mg to 45 ± 11.62 GPa at 2 wt%Mg. This value perfectly matches with natural bone elastic modulus and as a result will avoid issue of stress shielding and increase bone to implant interphase interaction. Above 2 wt%Mg addition, the elastic modulus decreases. In the same vein, similar pattern of

behaviour in hardness value was recorded with the alloy as showed in Fig. 5 (c). The reason for the decrease in hardness above 2 wt%Mg may be as a result of increasing formation of soft lump particles of magnesium and eutectic zinc phase in the alloy matrix. It is worthy of stating that, the optimum strength, strain, elastic modulus and hardness of 210.23 ± 8.88 MPa, $9.38 \pm 0.66\%$, 45.43 ± 11.62 GPa and 76.25 ± 5.71 HV as obtained in this study meet the minimum mechanical properties requirement for BDMs as reported by Gu et al. [37].

It is therefore suggested that, Zn-2Mg alloy produced using PM be considered a potential BDM for biomedical application.

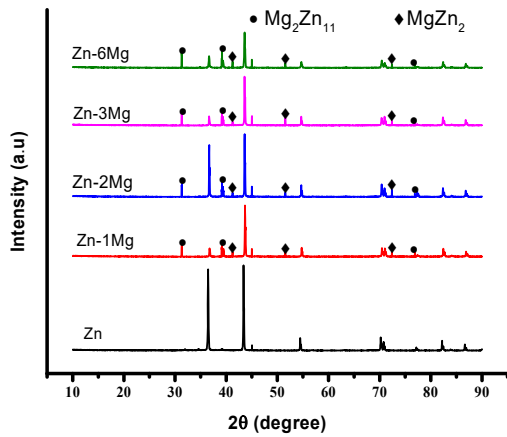


Fig. 4 XRD pattern of Zn-Mg alloy

IV. CONCLUSION

In the present study, biodegradable Zn-Mg alloy was successfully produced by PM route; mechanical and morphological properties were investigated. The findings were:

- i. Physical and mechanical properties of Zn-Mg are influenced by the volume fraction of magnesium in zinc matrix and absence of pores and porosity showed good compaction and sintering process.
- ii. The optimum compressive strength (210 ± 8.88 MPa), elastic modulus (45 ± 11.62 GPa) and hardness (76 ± 5.71 HV) equivalent to 110, 93 and 93% increments respectively were obtained at 2 wt% addition magnesium volume fractions.
- iii. The presence of high adhesive binding force between the hard intermetallics and zinc-rich phases were responsible for mechanical properties enhancement

The study therefore concluded that powder metallurgical route is promising route for the processing of Zn-Mg alloys. Zn-2Mg alloy possessing optimum mechanical properties meeting the minimum requirement of BDM can therefore be considered a potential candidate for biomedical application.

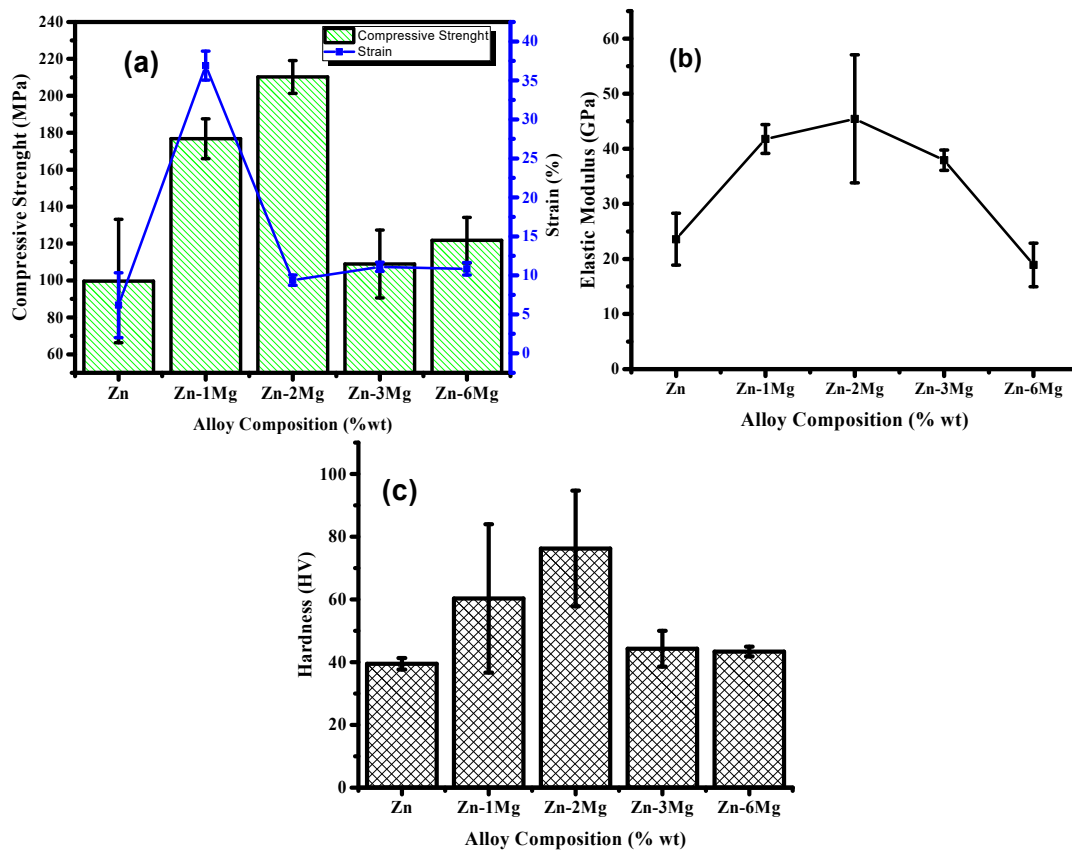


Fig. 5 Compressive strength and strain (a), Elastic modulus (b) and Hardness (c), of Zn-Mg alloy

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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