Effect of multi-axial hot forging process on mechanical, and corrosion resistance behavior of Mg-3Zn alloy for temporary orthopedic implants

Satish Jaiswal | Shubham Agrawal | Anshu Dubey | Debrupa Lahiri

Biomaterials and Multiscale Mechanics Lab, Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, Roorkee, India

Correspondence
Debrupa Lahiri, Biomaterials and Multiscale Mechanics Lab, Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, Roorkee, Uttarakhand 247667, India. Email: debrupa.lahiri@gmail.com; dlahifmt@iitr.ac.in

Funding information
Science and Engineering Research Board, Grant/Award Number: SB/SO/HS/138/2013; Department of Science and Technology

Abstract
Magnesium (Mg) is a load-bearing biocompatible material which has the ability to reduce stress shielding effect and facilitate osteocompatibility and biodegradation in the presence of body fluids. However, Mg is highly susceptible to corrosion in the physiological environment, hence leading to poor mechanical integrity. In this study, the multi-axial hot forging (MAHF) process is performed on Mg-3Zn alloys to study its grain refinement and possible improvement in mechanical, corrosion, and bioactivity behavior. The average grain size of the sample becomes significantly refined after the third cycle of MAHF. The yield and ultimate compressive strength of Mg-3Zn alloys are found to increase by 70% and 41%, respectively, after the third cycle of MAHF, which is potentially due to the grain size refinement. Accelerated corrosion studies show improvements in the corrosion resistance with the refined grain structure. Additionally, in-vitro immersion studies in the simulated body fluid for 14 days showed a reduction in the degradation rate after third cycle of MAHF, due to the increased grain boundary area, which offered more nucleation sites for apatite precipitation. This study underscores and lays the foundation for a new branch of severely deformed fine-grained Mg-3Zn alloy for temporary orthopedic implants.

KEYWORDS
degradation, grain-refinement, mechanical, Mg-3Zn, multi-axial hot forging, orthopedic

1 | INTRODUCTION

In recent years, Mg and its alloys are in the spotlight for a new generation of biodegradable materials for orthopedic applications. Besides biodegradability, Mg alloys, being metallic, are more suitable in load-bearing applications, as compared to ceramics and polymers. Mg offers sufficient strength and Young’s modulus close to the natural human bone. Additionally, the physical properties, like the density of Mg and its alloys (~1.74-1.90 g cm$^{-3}$), are quite comparable to natural compact bone (~1.75 g cm$^{-3}$).

Metals, such as Titanium (Ti) and its alloys, stainless steel (SS), and Platinum (Pt), are conventionally employed as temporary implants for bone fracture healings. These implants are non-biodegradable, and consequently, in most of the
cases, resurgery is required to take out these implanted accessories from the human bone after sufficient tissue healing. A second operation is not desirable because of the possibility of infection risk during surgery and higher costs for health care. In addition to that, after a specific time, bone growth and integration on these implants complicate their retrieval from the body, often making it impossible also. Thus, there is a need to design implants, which can demonstrate with a suitable degradation rate in the physiological environment, comparable to the rate of tissue healing. It should be non-toxic and metabolized by the human body.

It has also been reported that dissimilarity in elastic modulus of metallic implants and bone tissue caused the stress-shielding. The elastic modulus of Mg (~45 GPa) is close to the natural bone tissue (~30 GPa), which is approximately half of the elastic modulus of Ti6Al4V alloy (109-112 GPa). Mg has the great potential to replace the widely used Ti6Al4V if the corrosion rate can be adapted to simulate the bone growth rate. In addition, the issues with stress shielding would also be taken care.

In this context, the presence of active bivalent ions (Mg²⁺) in Mg-based materials supports the apatite layer formation in the physiological environment. Apart from that, Mg implanted alumina (Al₂O₃), by an ion implantation procedure, enhanced adhesion as well as differentiation of the human cells when compared to non-coated alumina. Mg²⁺ ion is extensively found inside the human body without any trace of harmful effects. In this regard, researchers have performed the initial in-vitro evaluation of cytotoxicity of bare Mg as well as surface-modified Mg alloys using bone marrow cells of mice. This initial assessment shows positive cell proliferation and viability even after 72 hours of incubation with Mg alloys, as well as no indication of growth inhibition. Additionally, the biodegradable nature of Mg and its alloys would be an extra benefit, as fracture-fixing accessories would be completely replaced by new growing tissue.

However, Mg and its alloys, despite having tremendous potential for orthopedic application, are not extensively used for clinical application till date. It is due to the major issue related to the high reactivity of Mg metal (standard electrode potential of ~2.13 V, as compared to a standard hydrogen electrode) in the physiological environment. Lower pilling-Bedworth ratio (~0.79) leads to poor capability of oxide layers toward the corrosion protection. For orthopedic application, as cast Mg metal has shown significant ability to induce the new bone formation; however, the lack of sufficient mechanical property and degradation rate is still a major concern. Alloyping is an important process to improve the corrosion resistance and mechanical properties of Mg. The advantages of alloys differ depending on the type and appropriate amount of alloying elements being added. Thus, alloying of Mg might be a suitable possibility to tailor the corrosion as well as mechanical properties. In connection to this, alloying elements like calcium (Ca), zirconium (Zr), aluminum (Al), zinc (Zn), and rare earth (RE) metals are generally found to be effective in reducing the corrosion rate of pure Mg as well as enhancing its mechanical characteristics. Out of all, Zn, Al, and RE elements are widely used in commercially available Mg alloys. In recent trends, it has been observed that many researchers have studied the effect of these commercially available elements in pure Mg in terms of corrosion and mechanical properties. In this regard, Witte et al. have reported in-vivo corrosion of four different Mg-based alloys and reported that degradation layer of all alloys contains biological apatites. Further, all the alloys showed enhanced bone mass surrounding the samples, as compared to polymers. As stated in this study, LAE442 (Mg-4%Li-3.6%Al-2.4%RE, in wt%) shows the lowest degradation rate, while WE43, AZ91, and AZ31 were claimed to corrode at the same rate. Additional of Al in the human body promotes neurotoxicity, and accumulation of Al is associated with much neurological disorder. Extreme hepatotoxicity has also been reported after the addition of RE elements in Mg materials. Although many studies have been carried out with these (RE metal and Al) as alloying elements in magnesium materials, but they are futile considering toxicity, as these alloys can never be used in orthopedic. Above-mentioned early studies suggested that Mg-based material system are biocompatible and help in bone tissue healings. Moreover, the degradation rate of as-cast Mg and its alloys are too fast to give significant time for bone tissue healing. At the same time, it is acceptable that the implanted fixtures sustain for minimum 8-12 weeks. To guarantee the biosafety of Mg-based alloys, selection of alloying element with no toxicity is a major concern. In this regard, Ca is found in the human bone and help in chemical signaling with cells. The major limitation of Ca as an alloying element is its solubility limit in Mg (~1.34%). With increasing the content of Ca, coarser Mg₃Ca phase precipitates out along the grain boundaries and eventually, lead to the poor degradation resistance and mechanical properties of Mg-Ca alloys. Zinc (Zn) is one of the other most abundant and nutritionally important element in the human body and generally found in all human body tissues. In addition to this, Zn helps in the formation of bone and prohibits the resorption of bone. Mg and Zn have a similar hexagonal closed packing (HCP) crystal structure, due to that, the addition of Zn strengthen Mg-based alloys by solid solution strengthening mechanism. Hence, considering the significant advantage of Zn as an alloying element for Mg-based materials, Mg-Zn alloy system could be coined as better biodegradable orthopedic temporary implants for fracture fixing accessories. Lotfabadi et al. studied the effect of the various composition of Zn (0, 1.5, 3, 6, and 9 wt%) on the corrosion behavior of Mg. It was found that the formation
of new intermetallic phase, that is, Mg$_{51}$Zn$_{20}$ along with the α-Mg and MgZn phases beyond 3 wt% composition of Zn in Mg, makes the alloy more prone to the galvanic corrosion, due to the formation of the anodic site. Linear polarization resistance ($R_p$) value was reported to decrease beyond 3 wt% of Zn. However, in the galvanic couple of Mg-3Zn, the intermetallic phase MgZn worked as the cathodic sites and α-Mg phase as the anodic site. Further, the formation of extra intermetallic phases after 3 wt% of Zn might reduce the ductility of the alloys. So, in this study, the addition of 3 wt% Zn in Mg was chosen as a possible candidate for orthopedic application.

In general, Mg and its alloys have poor mechanical strength and degradation resistance, when compared to the desired properties for the load-bearing application. To tailor this issue, grain refinement, produced via severe plastic deformation (SPD), is a constructive approach to enhance mechanical as well as corrosion properties of Mg alloys. It has been observed that homogeneous distributions of precipitates and Hall-Petch strengthening mechanism are the main cause for improving the mechanical and corrosion properties of Mg alloys. In connection to this, a couple of investigations have been done to see the effect of severe plastic deformation like equal-channel angular processing (ECAP) on as-cast Mg material. It has been reported that it offered decrement in grain size, which ultimately enhanced the corrosion resistance and mechanical strength of as-cast Mg. In another study by Dobatkin et al deformed WE43 alloy through different deformation techniques like rotary swaging, ECAP, and multi-axial deformation (MAD) and concluded that ECAP and MAD improved the tensile strength to 300 MPa, as compared with 220 MPa in, as-cast state. In addition to this, 12%-17% of improvement in ductility was also reported. Additionally, SPD of Mg alloys does not harm the resistance to galvanic corrosion. Borko et al characterized the AZ91 alloy as cast as well as ECAP treated in terms of corrosion resistance in NaCl solution. It has been observed that ECAP treated samples have shown better (~8%) corrosion resistance as compared to as-cast AZ91. Although samples showed better corrosion properties, but NaCl solution does not represent the real condition of body fluids and the presence of Al in these alloys may lead to Alzheimer disease. Apart from that, no mechanical study was reported about the load-bearing capacity of these alloys. In a previous study, the present authors have explored the effect of grain refinement on mechanical and corrosion behavior of Mg-3Zn alloys through the rolling and achieved ~77% improvement in tensile strength and ~264% enhancement in toughness. Grain refinement has helped in degradation resistance of these alloys for long term exposure in the physiological environment. These findings are encouraging. The rolling operation can provide only sheet-shaped materials, which are suitable for fabricating bone plates. However, it does not fulfill the requirement for different other shapes required in orthopedic fracture fixing application, for example, screws, pins, and nails. To get the bone-shaped implants, such as rod, screws, and nails, conventional rolling is not a suitable technique, and thus, forging is required to fabricate these shapes. Trivedi et al employed multi-axial forging (MAF) on Mg-2Zn-2Gd alloy and measured the grain refinement and its effect on the mechanical properties. After two passes, the average grain size reduced to ~1 μm, when compared to as-cast grain size (~25 μm). Yield strength of the alloy enhanced by ~320% after two passes of MAF, as compared to cast alloy. The increase in the mechanical properties is attributed to the high degree of grain fragmentation and the homogeneous distribution of fine precipitates throughout the alloy. However, the effect of MAF on corrosion and bioactivity of the Mg-2Zn-2Gd alloy was not explored by the authors. In another study, the mechanism of grain refinement and the relationship between the microstructure and mechanical properties was explored on the AZ61 alloy processed via isothermal MAD. After six passes, the initial grain size decreased from ~148 to 14 μm, and ultimate tensile strength increased by ~40%. The significant grain refinement was attributed to the continuous dynamic recrystallization. The corrosion and bioactivity of the alloys were not reported. Further, the presence of Al elements imposes a restriction on the use of this Mg alloys in biomedical application. In another effort, ultrafine grain structure was achieved by MAD of WE43 Mg alloy, and its effect on the mechanical, degradation, and biocompatibility was evaluated. The average grain size of the initial alloy (~64.9 ± 3 μm) reduced to the 0.93-0.29 μm after MAD. Reduction in the grain size of the alloy enhanced the degradation resistance. The relative mass loss after 6 weeks of in-vitro immersion (0.9 NaCl solution) was 83% and 19% for initial and processed alloys, respectively. However, 0.9 NaCl solution does not simulate the real ion compositions of the body fluid. Additionally, the presence of rare-earth elements in the Mg alloys is not preferable for orthopedic implants. In another study by Yurchenko et al, MAF was employed on the Mg-0.8Ca alloy and change in the mechanical properties was assessed as a function of grain refinement. The grain size of the Mg-0.8Ca alloy decreased from ~200 to ~2.4 μm after nine MAF passes. Ultimate tensile strength was enhanced by ~395% after nine passes of MAF when compared to homogenized alloy. No corrosion and the bioactive studies were performed in this study. Merson et al studied the influence of thermo-mechanical processing on the mechanical and corrosion properties of Mg-1Zn-0.2Ca alloy via multi-axis isothermal forging (MIF) at a relatively high homogeneous temperature (450°C, for 12 hours in argon) in combination with isothermal rolling. The grain size of the alloy reduced from 185 ± 44 to 2.9 ± 1.6 mm after five passes of MIF. The in-vitro immersion of the alloys...
showed the increment (∼320%) in the corrosion resistance as compared to cast alloy, after 168 hours of immersion in simulated body fluid (SBF). However, the immersion duration (168 hours) is too less to predict the real degradation behavior of Mg-1Zn-0.2Ca alloy. Furthermore, as per our best knowledge, no systematic investigation is reported in the literature on multi-axial hot forged (MAHF) treated Mg-3Zn alloy-based biomaterials for load-bearing orthopedic application. Attempts have been taken on the elucidation of the predominant steps of this field in this study.

In consideration of present scenario, the major objective of this study is to employ severe plastic deformation techniques to strengthen and development of more degradation resistance of selected Mg-3Zn alloy having potential use in the application of temporary implants/fracture fixing accessories. The severe plastic deformation process used in this study is multi-axial hot forging (MAHF) to develop homogeneous refined grain structure to tailor the strengthening behavior along with maintaining sufficient toughness and without significantly altering the elastic modulus of alloys. The strengthening is evaluated through compression testing. Further, the effect of grain refinement on the degradation mechanism of Mg-3Zn alloys is evaluated in depth using potentiodynamic polarization testing, as well as static immersion study to simulate real in-service state. In order to understand the corrosion and strengthening mechanism, in-depth, microstructural analysis has been performed at every stage of the experiments.

2 | EXPERIMENTAL MATERIALS AND PROCEDURES

2.1 | Material

The materials used in this study was Mg-3Zn (3 wt% Zn and balanced Mg) alloy, custom synthesized (Exclusive Magnesium Pvt. Ltd, Hyderabad). Gravity die casting route was followed to synthesize this alloys to get the homogeneous structure. Resistance type furnace was used to melt the starting materials, that is, Mg (purity—99.9%) and Zn (purity—99%) in the temperature span of 650°C-730°C using inert gas atmosphere. The molten materials were stirred for 3 minutes at a regular interval of 15 minutes to get the homogeneous structure followed by pouring in cast iron die mold of dimension 500×90×60 mm³ and cooled down. More details of the alloy synthesis are given in another article.13

2.2 | Multi-axial hot forging (MAHF)

As received Mg-3Zn alloy was cut into the cubical shape of dimension 13 mm × 13 mm × 13 mm for performing the deformation. MAHF of Mg-3Zn alloy was performed at a forging speed of approximately 12-15 mm s⁻¹, employing the forging press of 2000 kN load capacity (Birson Industries Regd., Punjab). Muffle furnace was used for soaking (400°C for 60 minutes) process to get the homogenized structure. After each press, the sample is again kept in the furnace for 10-20 minutes to maintain the forging temperature of the alloy (∼400°C) (so that slip system of alloy gets activated at high temperature) and further forging was performed on the sample by rotating it at 90°. This process was continued till a complete cycle (three passes each in x, y, z direction = 1 cycle). A total of four cycles of MAF was performed. Schematic diagram of MAF is shown in Figure 1. After each cycle, the sample was examined to get an insight into the physical properties and mechanism evolving with a given amount of plastic deformation. Nomenclature of the samples was done based on the number of cycles it has undergone (0C, 1C, 2C, 3C, and 4C).

2.3 | Microstructural and surface characterization

In order to evaluate the grain morphology of MAHF processed alloys, samples were thoroughly polished using silicon carbide (SiC) waterproof abrasive paper (up to 2000 grit size) and cloth polishing followed by 1 and 0.25 μm diamond paste. Further, to explore the grain boundaries of MAHF processed samples, electropolishing (−30°C, 12 V and 10 seconds) of specimens was carried out in the electrolyte solution made of 30% v/v nitric acid (HNO₃) in ethanol (C₂H₅OH). Methanol (CH₃OH) was used as a coolant during the etching process. Optical microscope (Leica DMI 5000 M, Germany) is used to capture optical micrographs of the samples. The morphology of degraded samples and apatite layer formed was characterized using field emission scanning electron microscopy (FESEM) (Quanta 200 FEG, FEI Netherlands) at 20 kV.
2.4 | Mechanical characterization

Mechanical characterization of homogenized and MAHF processed Mg-3Zn alloys was evaluated by uniaxial compression test and Vicker’s hardness test. Compression test specimens were machined as per the standard of ASTM E9-09 (height = 12 mm and diameter = 8 mm). Universal testing machine (H25K-S Tinius Oslen, USA) was used to performed compression testing (strain rate of $10^{-4} \text{ m s}^{-1}$) at room temperature. To assure the reproducibility, a minimum of three samples were tested in each cycle processed by MAHF techniques. Scanning electron microscopic (SEM) images of the fractured surfaces (homogenized and processed), after compression tests, were captured to analyze the failure behavior of alloys. To characterize the surface resistance property, Vicker’s hardness testing instrument (FIE-VM 50 PC, India) was used to measure the hardness of all the samples by applying 1 kg load for 15 seconds of dwell time at room temperature. At least 15 readings on every different cycle processed samples were taken to get the average hardness values.

2.5 | Corrosion behavior testing

2.5.1 | Electrochemical study

Potentiodynamic polarization experiment was performed to evaluate the accelerated corrosion behavior of MAHF processed Mg-3Zn alloy using a three-electrode configured electrochemical potentiostat (Gamry Instruments, USA). Parameters comprising of a voltage range of 0.25 V above and 0.25 V below the open circuit potential (OCP) and scanning rate of 0.3 mV s$^{-1}$ was set up to carry out this experiment. A constant temperature of $37 \pm 1^\circ\text{C}$ was maintained throughout the experiments. Samples were thoroughly polished up to 2000-grit size abrasive (SiC) paper before any accelerated corrosion study. A three-electrode cell, having platinum metal as the counter electrode, standard calomel electrode for reference, and samples as the working electrode, was used to complete the cell circuit. The electrochemical cell had fixed opening area (circular) of 0.785 cm$^2$ for the surface exposure into the electrolyte. SBF was used as the electrolyte in the cell and was prepared by following Kokubo’s protocol$^{38}$ which includes 8.035 g NaCl, 0.225 g KCl, 0.292 g CaCl$_2$, 0.355 g NaHCO$_3$, 0.072 g Na$_2$SO$_4$, 0.231 g K$_2$HPO$_4$·3H$_2$O, 0.311 g MgCl$_2$·6H$_2$O, 6.118 g Tris buffer and 39 mL of 1 M-HCl. In order to stabilize OCP, an initial stabilization time of 3600 seconds was given to the entire sample. The corrosion current density ($I_{\text{corr}}$) and corrosion potential ($E_{\text{corr}}$) were calculated through the linear fitting of the cathodic and anodic portions of the generated Tafel plots. Electrochemical tests were performed in triplicate to ensure the reproducibility of results.

2.5.2 | In-vitro static immersion study

The samples of dimension 10 mm $\times$ 10 mm $\times$ 4 mm were machined out from all different forged cycles. The samples were polished till 3000 grit size by SiC abrasive paper to get equal surface roughness of all the samples followed by
ultra-sonication to remove any contamination from the surfaces during polishing. In-vitro immersion test was performed in SBF at 37 ± 1°C. Each of the samples was immersed in 20 mL SBF solution in glass vials. Solution volume to the exposed surface area of all the samples was kept constant (∼55 μL/mm²). Dipped samples were retrieved after 3, 7 and 14 days of static immersion. Further, the retrieved samples were gently cleaned in the distilled water, followed by drying at room temperature and in the desiccator for next 24 hours. Weight of the samples was taken before and after the immersion studies to quantify the mass gain plot. The mass gain in samples was due to the apatite layer formation during immersion. The retrieved samples were thoroughly dried before taking the weights to avoid any mass gain due to the moisture or solution content. Separate set of samples were used for each immersion period. Degradation behavior of the samples (using mass loss) was reported after washing off the dried apatite layers from the surface of the samples using a solution of 180 g/L chromic acid. The morphology of apatite precipitation was examined by scanning electron microscopy.

2.6 | Statistical analysis

Statistical analysis was performed using an analytical tool pack from excel (MS office). Data were represented as mean ± SE. For statistical analysis, analysis of variance (ANOVA): single factor was used. The level of statistical significance was set at \( P < .05 \).

3 | RESULTS AND DISCUSSIONS

3.1 | Microstructure

In order to measure the grain size variation of homogenized Mg-3Zn and MAHF processed alloys, optical microstructures were captured, which is presented in Figure 2A-E. The optical microstructure of homogenized Mg-3Zn alloy is presented in Figure 2A. It can be clearly seen that as the number of MAHF cycle increases the grain size of the alloy decreases significantly (Figure 2). Figure 2F illustrates the quantitative variation of grain size measurement during all forging cycles. The grain size of the alloys was measured from the obtained optical microstructure after every cycle of forging using ImageJ software.\(^{39}\) Average value obtained with SD has been plotted, as shown in Figure 2F. Approximately 83% and 58% of decrement in the grain size of alloys was measured after the third and fourth cycle of forging respectively when compared to the homogenized Mg-3Znalloy. The average grain size of the alloy after the third cycle of MAHF (equiaxed grain) decreased by ∼83% as compared to homogenized Mg-3Zn alloy (Figure 2F). Further, during the transition from second to third cycle of MAF, a significant drop (∼68%) in the grain size was measured. It has been observed that coarse grains are being deformed, which leads to an increase in strain energy. Generally, these residual strains have a dual effect on microstructure. It might increase the rate of recrystallization due to high energy site and act as a nucleating site for recrystallization. It may also decrease the temperature of recrystallization. Further heating and increase in the strains led to the recovery and recrystallization phenomenon resulting in the release of strain energy and the formation of new small grains inside the parent grains (Figure 2B). In addition to that, higher stress concentration at the triple junction of grain boundaries led to the nucleation of small grains. From Figure 2A-E, original coarse grains and dynamically recrystallized grains can be clearly differentiated. With increasing the number of MAHF cycle, the volume fraction of fine crystallized grain is increased. The third cycle of MAHF represents the beginning of severe dynamic recrystallization. However, grain growth started after the third cycle of processes, which can be clearly observed in the fourth cycle of MAHF and can also be confirmed by grain size measurement (Figure 2F). Due to the recrystallization in the third cycle of forging, there is a chance for grain growth until residual strain becomes too high in the recrystallized grains to have further recrystallization. Further, heating of sample after third cycle of operation at 400°C for 1 hour may provide the activation energy for grain growth, resulting into grain growth of samples after the fourth cycle of MAHF. Figure 2D is the optical microstructure of MAHF alloy after the third cycle. It can be visualized from the given microstructure that, recrystallization of grains was exceeded after third cycle forging as compared to the second cycle which resulted into the grain size decrement (∼35 μm) (Figure 2F). This is expected to possibly affect the corrosion as well as mechanical properties of the alloy. Additionally, homogeneous refinement of grains at this high deformation rate is might be because of dynamic recrystallization. In addition to that, numbers of active slip planes at the high temperature (ie, 400°C) is comparatively more when compared with the room temperature deformation, which might help in the recrystallization of
FIGURE 2  Optical micrographs of (A) homogenized Mg-3Zn alloy, and forged ones after (B) first Cycle, (C) second Cycle, (D) third Cycle and (E) fourth cycle of multi-axial forging (F) grain size variation of Mg-3Zn alloy (0 cycles) and after different cycle (1, 2, 3, and 4) of MAHF

Grains in MAHF process. Slip planes developed through the lattice structure at some points, where the atomic bonds of attraction are weak; during high temperature (ie, 400°C) exposure there is a chance of the whole block of atoms displacement which might assist in the recrystallization. Generally, room temperature deformation is comparatively difficult due to high critical resolved shear stress of slip planes. Figure 2E is the MAHF processed micrograph of Mg-3Zn alloy after the fourth cycle of forging (average grain size ~86 μm), interestingly, the grain size is still lesser than the forged alloy after first and second cycle (~132 and 125 μm respectively) (Figure 2F). Formation of twin bands can also be seen in the microstructure taken after the third cycle of the process (Figure 2D). The number of twin bands decreases after the fourth cycle of forging (Figure 2E). In the polycrystalline materials, the twin shear in the grain is constrained by the surrounding grains, which result into the localized stress and strain state at twin and grain boundary junction and extend to the neighboring grains as well. Constraints posed on deformation by neighboring grain cause a stress concentration at the twin tips and a stress reversal on the twin lamella. As the grain size increases, these twin tip and grain boundary stress fields start decreasing and influence the degree of stress reversal in the twin lamella. Such a grain size constraint would
not resist thickening of twin lamella, and it gets disappeared in the comparatively larger grains. The decrease in the twin bands in the microstructure of the fourth cycle of MAHF indicates strain-free grains growth after recrystallization. The phase composition of the homogenized alloy was analyzed via energy-dispersive X-ray spectroscopy technique in the previous study by the author’s group. Composition of the precipitates at the grain boundary was found to be 78 wt% Mg and 22 wt% Zn and designated as the MgZn phase.

### 3.2 Mechanical properties

The load-bearing mechanism within the human body condition is quite complicated. To support the traumatized/fractured bone tissue, being quite tough and strong, implant materials should have similar mechanical performance. Different mechanical properties required for the design of fracture fixing accessories based on the application comprise of ultimate compressive strength (UCS), Young’s modulus, and toughness. Figure 3A shows the compressive stress-strain behavior of as-cast Mg-3Zn alloy and all other MAHF processed alloy. These plots were used for calculating compressive yield strength (YCS) and UCS of the processed alloys as depicted in Figure 3B. It was found that YCS and UCS value enhanced with decreasing the grain size of alloys. It has been seen that after the third cycle of forging the YCS of Mg-3Zn alloy surpasses the compressive yield strength of natural bone, which is 130-180 MPa. YCS of alloys was calculated at 0.2% of total strain through the stress-strain plot. Figure 3C represents the toughness and strain percentage of different alloys. The toughness of the Mg-3Zn alloy was improved by ∼9% after the second cycle of the MAHF process. For the first and second cycle of MAHF, toughness values were approximately the same. A slight decrement in toughness value of third cycle processed alloy was observed as compared to the second and first MAHF cycle. Approximately, 60% of decrement was seen in toughness value of Mg-3Zn alloy after the fourth cycle of MAF processing as compared to as-cast Mg-3Zn alloy.

Homogenized Mg-3Zn alloy showed comparatively lower compressive ultimate and yield strength due to its coarse-grained structure. According to the Hall-Petch equation, the lower grained structure gives higher YCS. This might be the possible reason for lower YCS of homogenized alloy. As the grain size of alloys decreases, having inverse relation with yield strength, the YCS of alloys increases till the third cycle of operation. In addition to that, dynamic recrystallization is promoted in the third cycle of forging, while it was absent in the homogenized alloy. Dynamic recrystallization may also play a significant role in improving the mechanical properties through the grain refinement of MAHF processed Mg-3Zn alloys. After the third cycle, due to the grain growth, YCS and UCS decreased. Additionally, the possibility of further recrystallization is reduced after the third cycle of MAF processing. A minimal change in the elastic modulus of MAHF processed alloys was observed as compared to the homogenized Mg-3Zn alloy (Figure 3A). In general, the elastic constants of materials depend on the interatomic forces. The presence of residual stress, microcracking, and variation in the dislocation structure caused by plastic deformation could be the reasons for such changes in the elastic modulus after plastic deformation. Further, the additional barriers generated near inclusions might hinder the elastic recoveries. Crystalline texture also can be expected to invoke significant degree of bulk anisotropy in severely deformed Mg-based alloys. Besides, the possible presence of pores and internal cracks in the homogenized Mg-3Zn alloy might be the reason for the change of elastic modulus after first cycle of MAHF.

Ductility of the alloy decreased significantly after the first cycle of MAHF, grain refinement of homogenized alloy might be the reason for decreased ductility (Figure 3C). But, afterward, it remained almost similar till the third cycle. Grain refinement from first to second cycle was comparatively lesser. Dynamic recrystallization took place during the third cycle of forging, which caused minor impact on the ductility. In the case of the fourth cycle of MAHF, ductility again decreased drastically due to the comparatively less dynamic recrystallization. The toughness of all the samples was measured by calculating the area under the compressive stress-strain diagram. Initially, the toughness of the alloys increased with the number of cycles, but it started decreasing after the third cycle of processing (Figure 3C). After the processing of the fourth cycle of MAHF, both the strength and ductility of the alloy decreased, which resulted in the lower toughness of alloys (Figure 3C). Micrograph of fractured surfaces for homogenized and third cycle processed alloy after compression tests were also taken and are presented in Figure 4. Typical shear bands structures were observed in the fractographs of homogenized and processed alloys (Figure 4A,B). However, the effect of severe plastic deformation on the fractograph of third cycle MAHF processed alloy can be observed (Figure 4B). The intensity of the shear band reduces after the third cycle of MAHF, which indicates the brittle fracture under compression stress.
3.3 | Corrosion properties

3.3.1 | Accelerated corrosion test

In spite of the immense potential of Mg-3Zn alloys for orthopedic implants, the less corrosion resistance of these biomedical implants in the physiological environment imposes severe restriction in many orthopedic applications. Consequently, extensive accelerated corrosion test has been carried out to evaluate corrosion resistance properties of as-cast Mg-3Zn
alloy, and the entire group of MAHF processed alloys. Figure 5 represents the potentiodynamic polarization test curves for all the alloys.

Cathodic polarization curve is generally associated with the hydrogen evolution phenomenon due to the reduction of the aqueous medium, while the dissolution of Mg alloy is represented with anodic polarization curve. The values of $I_{\text{corr}}$ and $E_{\text{corr}}$ were obtained from polarization curves using Tafel extrapolation method (Table 1). The corrosion current density ($I_{\text{corr}}$) decreases with an increase in the number of forging cycles (Table 1, calculated via Tafel extrapolation methods). In addition to that, corrosion potential ($E_{\text{corr}}$) also shifted toward the positive side with an increase in the number of forging cycles. Apparently, the processing of Mg-3Zn alloy up to third cycle of MAHF shifts the corrosion potential toward the noble direction. The corrosion rate is found to be decreasing with the number of forging cycles. The decrease in the corrosion current density ($I_{\text{corr}}$) and shifting of the corrosion potential ($E_{\text{corr}}$) toward positive value gives a clear indication of better corrosion resistance. Chlorine ions present in SBF can easily penetrate from any possible small cracks on exposed surfaces due to its smaller radius. As a result, it severely corrodes the surface, followed by the formation of large pits. SBF contains enough chlorine ions to breaks down the naturally formed Mg(OH)$_2$ layers from the exposed surfaces. Following reaction occurs during this phenomenon:

$$\text{Mg(OH)}_2 + 2\text{Cl}^- \rightarrow \text{MgCl}_2 + 2\text{OH}^-$$

$I_{\text{corr}}$ and $E_{\text{corr}}$ values measured from the Tafel curves (Figure 5) are summarized in Table 1. It is clearly deduced from Table 1 that all processed Mg-3Zn alloys exhibit superior corrosion resistance as compared to as-cast Mg-3Zn.

Refinement of grains plays a significant role in tailoring the corrosion behavior of Mg alloys. It has been observed that refinement of grain by ~82% as compared to homogenized Mg-3Zn alloy have caused ~52% of improvement in corrosion resistance after the third cycle of forging. However, ~42% of increment was seen in corrosion rate during the transition from third to fourth cycle, due to grain growth by ~58%. Refinement of grain size offers higher grain boundary area per unit volume, which ultimately provides more nucleation sites for apatite formation in the physiological environment. Grain boundaries are higher strain energy regions, which support for nucleation of apatite in SBFs. Formation of a thin apatite layer on the surfaces of alloys protects them from the severe corrosive environment. The native oxide film on Mg, which is supposed to offer considerable protection while exposed to the aqueous environment, does work for Mg due to their lower Pill-Bedworth ratio (<1). So, the formation of more apatite layer on the exposed surfaces helps in restricting

| Cycles | $I_{\text{corr}}$ (µA cm$^{-2}$) | $E_{\text{corr}}$ (V) | Corrosion rate (mpy) |
|--------|-------------------------------|----------------------|----------------------|
| 0      | 220.5 ± 2.05                  | 1.72 ± 0.057         | 221.9 ± 1.89         |
| 1      | 183.3 ± 1.6                   | 1.64 ± 0.048         | 187.6 ± 2.34         |
| 2      | 165.5 ± 1.8                   | 1.63 ± 0.065         | 166.9 ± 2.25         |
| 3      | 104.5 ± 1.5                   | 1.60 ± 0.049         | 105.3 ± 1.98         |
| 4      | 152.5 ± 1.4                   | 1.62 ± 0.045         | 149.2 ± 2.26         |
**FIGURE 6** Scanning electron microscopy image of corroded surfaces of homogenized Mg-3Zn alloy and MAHF treated alloys after the removal of apatite layers from 14 days of immersion (Image magnification: 200×)

**FIGURE 7** (A) Degradation rate of samples in SBF measured after the removal of apatite layers from the surfaces for different days of immersion. (B) Mass gain in the form of apatite layers during the immersion period. Data represent mean ± SE of three sets of an independent experiment.
FIGURE 8 Scanning electron microscopy images showing amount of apatite layer (A, C, E, G, I) after 3 days of immersion and (B, D, F, H, J) after 14 days of immersion in SBF (Image magnification: 2000×)
the severe attack of chlorine ions in the physiological environment. Generally, pitting corrosion happens when the oxide layer of Mg peels off locally, followed by severe corrosion initiation at these specific pitting locations. Figure 6 shows the scanning electron microscopy images of dried corrosion retrieved alloys surfaces after 14 days of immersion. Localized pitting corrosion can be clearly visualized from the given Figure 6A-E. Homogenized Mg-3Zn alloy shows bigger pits as compared to other processed alloy. Apparently, less significant pits can be observed on the alloy treated up to the third cycle of forging. The number of pits decreases with the forging cycles.

3.3.2 | In-vitro immersion testing

Immersion test was performed to measure the progress of degradation damage of alloys for different days of exposure (3, 7, and 14 days) to the SBF. Immersion testing of all the samples, including homogenized alloy, was conducted in SBF, which has similar ion concentration of human blood plasma. Figure 7A represents the variation of degradation rate over a period of 14 days of immersed in SBF. The degradation rate was measured after the removal of the apatite layers from the Cr₂O₃ solution and calculated as per the standard equation mentioned in previous studies.¹³,⁴²,⁴³

\[
\text{Degradation Rate} = \frac{(W \times K)}{(D \times T \times A)}
\]

where \( W \) is weight loss (g), \( T \) is the time of immersion (h), \( D \) is the density of the material (g cm\(^{-3}\)), and \( K \) is constant (8.76 \times 10^4).

It has been observed that corrosion resistance improved with the number of forging cycles, as well as the number of immersion days. The degradation rate for the initial period was quite high for all the samples. In addition to that, the evolution of hydrogen gas bubbles was also observed just after the immersion of these alloys in SBF environment. The intensity of hydrogen gas release declined with the time and finally got settled down after a few hours of immersion. The estimated degradation rate in mm/year for 14 days of immersion showed the fastest degradation rate for homogenized Mg-3Zn alloy (2.45 ± 0.07 mm year\(^{-1}\)). Whereas Mg-3Zn alloy treated up to the third cycle of forging exhibited a rate of 1.8 ± 0.054 mm year\(^{-1}\). The degradation rate of third cycle forging treated alloy showed ~31% of decrement after 14 days of static immersion. However, the degradation rate merges to approximately similar value after 14 days of immersion for all the samples. Reduction in the corrosion rate can be explained by precipitation of the protective apatite layer at the surface of the dipped samples. From Figure 7B, it can be observed, the amount of apatite layer formation increases.

![Energy-dispersive X-ray spectroscopy elemental mapping of the apatite layer formed on the Mg-3Zn alloy after immersing in the SBF solution](image-url)
Figure 10 Macroscopic image of immersed samples for 3, 7, 14 days in SBF (red dotted circles are denoting the high pitting corroded regions)
with the number of immersion days. It is evident from Figure 8B, D, F, H, J that apatite precipitation on the alloy surfaces becomes prominent after 14 days of static immersion. The apatite layers, formed on the surfaces of the alloy, was confirmed via energy-dispersive X-ray spectroscopy elemental mapping of a specific exposed surface of the Mg-3Zn alloy after 14 days of immersion in SBF (Figure 9). Presence of the elements such as Calcium (Ca), Phosphorous (P), and Oxygen (O) confirms the precipitation of apatite compound on the surfaces of alloys. The amount of apatite precipitation was comparatively higher in case of third cycle of forging due to its refine-grained structure, which offers more nucleation sites to precipitation of the apatite. Further, the unique property of Mg-3Zn alloy to precipitate out the apatite layer at surfaces, during immersion in the physiological environment; could offer as an impressive in-situ mechanism for degradation resistance. Figure 7B is showing the mass gain (apatite layer precipitation) during exposure in SBF. Grain size reduction resulted in the increment of grain boundary area/volume, in case of third cycle processed sample, has facilitated the highest precipitation of apatite layers after 14 days of static immersion. Precipitation of more apatite on refined grain structure restricts the negative principle of galvanic corrosion. Figure 8A-J represents the scanning electron microscopy image of retrieved and dried samples from immersion tests. It gives a clear indication of apatite layer formation on the alloy surfaces during the immersion period. Similar benefits of apatite layer precipitation were also seen in the previous studies.12,13

The extent of pitting corrosion (shown by red dotted circles) can be clearly seen in digital images (Figure 10A-O) of immersed samples, after removal of apatite layers through Cr2O3 solution. Degradation rates (Figure 7A) obtained via immersion studies are correlated with digital images of retrieved samples (Figure 10). In addition to that, the protection mechanism of apatite layers toward degradation can also be correlated by FESEM images (Figure 8). The homogenized Mg-3Zn alloy has shown severe pitting corrosion (Figure 10C) as compared to MAHF processed alloys for 14 days of immersion in SBF. From Figure 10C, it can be observed that the formation of pits was comparatively more on homogenized Mg-3Zn alloy for 14 days of immersion. Precipitation of more apatites on the forging treated sample made it more resistance for degradation in the physiological environment. This could be the possible reason for better degradation resistance of the third cycle treated forging alloy (Figure 10J-L). These trends fully support the accelerated corrosion test data, performed in SBF.

4 | CONCLUSIONS

The present study thoroughly explored the possibility of the strengthening and corrosion resistance of Mg-3Zn alloy through grain refinement by MAHF process and its compatibility in orthopedic fracture fixing accessories. The homogenized Mg-3Zn alloy severely deformed through the MAHF process shows grain refinement up to ~83% till third successive cycle of forging. The yield compressive strength and UCS of Mg-3Zn alloy were found to be improved by ~70% and 41% respectively up to third cycle of MAHF. Increase in the yield compressive strength can be attributed to grain refinement. Accelerated corrosion experiments have revealed high corrosion resistance for smaller grain size (~58% improvement for the third cycle of forging) due to the more grain boundary area/volume, which favors the nucleation of apatite layer. Further, in-vitro immersion test up to 14 days in SBFS also supports the trends found in accelerated corrosion studies. Based on these findings, it can be proposed that Mg-3Zn alloy treated with MAHF till third cycle has the potential to replace the current permanent implants for fracture fixing accessories. Future studies should address the advance biocompatibility tests of MAHF treated alloys in order to predict in-vivo performance.

ACKNOWLEDGEMENTS

This study is supported by the Department of Science and Technology (Funding reference no. SB/SO/HS/138/2013). The authors appreciate the efforts of Mr Manoj Kumar R for their technical supports throughout the experiments. The authors would also like to thank the staff of the fabrication lab in Metallurgical and Materials Engineering Department, IIT Roorkee.

AUTHOR CONTRIBUTIONS

Satish Jaiswal: Investigation; methodology; writing-original draft; writing-review and editing. Shubham Agrawal: Investigation; methodology; writing-original draft. Anshu Dubey: Investigation; methodology. Debrupa Lahiri: Conceptualization; funding acquisition; supervision; writing-review and editing.
PEER REVIEW INFORMATION

Engineering Reports thanks Hamdy Ibrahim and other anonymous reviewers for their contribution to the peer review of this work.

CONFLICT OF INTEREST

The authors declare no potential conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are partially available in the supplementary material of this article. The full data can be obtained from the corresponding author upon reasonable request.

ORCID

Satish Jaiswal https://orcid.org/0000-0001-5067-8774
Debrupa Lahiri https://orcid.org/0000-0003-3499-7307

REFERENCES

1. Prakash C, Singh S, Pabla BS, Sidhu SS, Uddin MS. Bio-inspired low elastic biodegradable mg-Zn-Mn-Si-HA alloy fabricated by spark plasma sintering. Mater Manuf Process. 2019;34:1-12.
2. Yin Y, Huang Q, Liang L, et al. In vitro degradation behavior and cytocompatibility of ZK30/bioactive glass composites fabricated by selective laser melting for biomedical applications. J Alloys Compd. 2019;785:38-45.
3. Bommala VK, Krishna GM, Rao CT. Magnesium matrix composites for biomedical applications: a review. J Magnes Alloy. 2019;7:72-79.
4. Dubey A, Jaiswal S, Haldar S, Roy P, Lahiri D. Mg-3Zn/HA biodegradable composites synthesized via spark plasma sintering for temporary orthopedic implants. J Mater Eng Perform. 2019;28:5702-5715.
5. Haghshenas M. Mechanical characteristics of biodegradable magnesium matrix composites: a review. J Magnes Alloys. 2017;5:189-201.
6. Sukhodub L, Panda A, Dyadyura K, Pandova I, Krenicky T. The design criteria for biodegradable magnesium alloy implants. MM Sci J. 2018;12:2673-2679.
7. Mahapatro A, Jensen K, Yang SY. Effect of polymer coating characteristics on the biodegradation and biocompatibility behavior of magnesium alloy. Polym Plast Technol Eng. 2019;59:301-310.
8. Jaiswal S, Kumar RM, Gupta P, Kumaraswamy M, Roy P, Lahiri D. Mechanical, corrosion and bio-compatibility behaviour of mg-3Zn-HA biodegradable composites for orthopaedic fixture accessories. J Mech Behav Biomed Mater. 2018;78:442-454.
9. Song MS, Zeng RC, Ding YF, et al. Recent advances in biodegradation controls over mg alloys for bone fracture management: a review. J Mater Sci Technol. 2018;35:535-544.
10. Howlett CR, Zreiaot H, O’dell R, et al. The effect of magnesium ion implantation into alumina upon the adhesion of human bone derived cells. J Mater Sci Mater Med. 1994;5:715-722.
11. Wang Y, Geng Z, Huang Y, et al. Unraveling the osteogenesis of magnesium by the activity of osteoblasts in vitro. J Mater Chem:B. 2018;7:1-3.
12. Liu F, Ji Y, Sun Z, Wang G, Bai Y. Enhancing corrosion resistance of Al-cu/AZ31 composites synthesized by a laser cladding and FSP hybrid method. Mater Manuf Process. 2019;34:1-9.
13. Nayak S, Bhushan B, Jayaganthan R, Gopinath P, Agarwal RD, Lahiri D. Strengthening of mg based alloy through grain refinement for orthopaedic application. J Mech Behav Biomed Mater. 2016;59:57-70.
14. Dubey A, Jaiswal S, Lahiri D. Mechanical integrity of biodegradable mg–HA composite during in vitro exposure. J Mater Eng Perform. 2019;28:800-809.
15. Kaesel V, Tai PT, Bach FW, Haferkamp H, Witte F, Windhagen H. Approach to Control the Corrosion of Magnesium by Alloying. Weinheim, FRG: Wiley-VCH Verlag GmbH & Co. KGaA; 2003:534-539.
16. Witte F, Kaese V, Haferkamp H, et al. In vivo corrosion of four magnesium alloys and the associated bone response. Biomaterials. 2005;26:3557-3563.
17. El-Rahman SSA. Neuropathology of aluminum toxicity in rats (glutamate and GABA impairment). Pharmacol Res. 2003;47:189-194.
18. Zhen Z, Xi TF, Zheng YF. A review on in vitro corrosion performance test of biodegradable metallic materials. Trans Nonferrous Metal Soc. 2013;23:2283-2293.
19. Müller WD, Nascimento ML, Zeddiies M, Córsico M, Gassa LM, Mele MA. Magnesium and its alloys as degradable biomaterials: corrosion studies using potentiodynamic and EIS electrochemical techniques. Mater Res. 2007;10:5-10.
20. Stroganov GB, Savitsky EM, Terekhova VF, Volkov MV, Sivash KM, Borodkin VSUS. Patent No. 3,687,135; 1972. Washington, DC: U.S. Patent and Trademark Office.
21. Ilich JZ, Kersetter JE. Nutrition in bone health revisited: a story beyond calcium. J Am Coll Nutr. 2000;19:715-737.
22. Li Z, Gu X, Lou S, Zheng Y. The development of binary Mg–Ca alloys for use as biodegradable materials within bone. Biomaterials. 2008;29:1329-1344.
23. Rad HRB, Idris MH, Kadir MRA, Farahany S. Microstructure analysis and corrosion behavior of biodegradable Mg–Ca implant alloys. *Mater Design*. 2012;33:88-97.
24. Tapiero H, Tew KD. Trace elements in human physiology and pathology: zinc and metallothioneins. *Biomed Pharmacother*. 2003;57:399-411.
25. Yamaguchi M. Role of zinc in bone formation and bone resorption. *J Trace Elem Med Biol*. 1998;11:119-135.
26. Friedrich HE, Mordike BL. *Magnesium Technology*. Berlin, Germany: Springer; 2006:788.
27. Gao JC, Sha W, Qiao LY, Yong WANG. Corrosion behavior of Mg and Mg-Zn alloys in simulated body fluid. *Trans Nonferrous Metals Soc*. 2008;18:588-592.
28. Lotfabadi AF, Idris MH, Ourdjini A, Kadir MRA, Farahany S, Bakhsheshi-Rad HR. Thermal characteristics and corrosion behaviour of Mg-Zn alloys for biomedical applications. *Bull Mater Sci*. 2013;36:1103-1113.
29. Trivedi P, Nune KC, Misra RDK, Goel S, Jayganthan R, Srinivasan A. Grain refinement to submicron regime in multi-axial forged mg-2Zn-2Gd alloy and relationship to mechanical properties. *Mater Sci Eng A*. 2016;668:59-65.
30. Ralston KD, Birbilis N. Effect of grain size on corrosion: a review. *Corrosion*. 2010;66:75005-75013.
31. Li Z, Huang N, Zhao J, Zhou SJ. Microstructure, mechanical and degradation properties of equal channel angular pressed pure magnesium for biomedical application. *Mater Sci Technol*. 2013;29:140-147.
32. Dobatkin SV, Lukyanova EA, Martynenko NS, et al. Strength, corrosion resistance, and biocompatibility of ultrafine-grained mg alloys after different modes of severe plastic deformation. *IOP Conf Series Mater Sci Eng*. 2017;194:1-8.
33. Borko K, Fintová S, Hadzima B. Linear potentiodynamic characterization of ECAPed biocompatible AZ91 magnesium alloy. *Mater Sci Forum*. 2017;891:404-408.
34. Xia X, Chen M, Lu Y, et al. Microstructural and mechanical properties of isothermal multi-axial forging formed AZ61 mg alloy. *Trans Nonferrous Met Soc Chin*. 2013;23:3186-3192.
35. Dobatkin S, Martinenko N, Anismanova N, Kiselevskiy M, Estrin Y. Mechanical properties, biodegradation and biocompatibility of ultrafine grained magnesium alloy WE43. *Materials*. 2019;12:3627.
36. Yurchenko NY, Stepanov ND, Salishchev GA, Rokhlin LL, Dobatkin SV. Effect of multi-axial forging on the microstructural and mechanical properties of Mg-08Ca alloy. *Mater Sci Eng A*. 2014;63:012075.
37. Merson DL, Brilevsky AI, Magalhães PJ, Ram SJ. Image processing with ImageJ. *Biophotonics Int*. 2004;11:36-42.
38. Cai S, Lei T, Li N, Feng F. Effects of Zn on microstructural, mechanical properties and corrosion behavior of mg–Zn alloys. *Mater Sci Eng C*. 2012;32:2570-2577.
39. Song GL, Atrens A. Corrosion mechanisms of magnesium alloys. *Adv Eng Mater*. 1999;1:11-33.
40. ASTM NACE TM0169/G31-12a. *Standard Guide for Laboratory Immersion Corrosion Testing of Metals*. Philadelphia, PA: ASTM International; 2012.
41. Zaludin M, Jamaludin S, Idris M, Llah N. Effect of 45S5 bio-glass particles on physical properties and corrosion resistance of the mg-5Zn matrix composite. *Open J Metal*. 2014;4:1-8.

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Additional supporting information may be found online in the Supporting Information section at the end of this article.

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**How to cite this article:** Jaiswal S, Agrawal S, Dubey A, Lahiri D. Effect of multi-axial hot forging process on mechanical, and corrosion resistance behavior of Mg-3Zn alloy for temporary orthopedic implants. *Engineering Reports*. 2021;3:e12286. [https://doi.org/10.1002/eng2.12286](https://doi.org/10.1002/eng2.12286)