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Article

Strength-Ductility Synergy in Biodegradable Mg-Rare Earth Alloy Processed via Multi-Directional Forging

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Abstract

In this study, a biodegradable Mg-Zn-Nd-Gd alloy was processed via multi-directional forging (MDF) to evaluate its microstructural evolution, mechanical performance, and corrosion behavior. Electron backscattered diffraction (EBSD) analysis was conducted to evaluate the influence of grain size and texture on mechanical strength and corrosion resistance. The average grain size decreased significantly from $118 \pm 5 \mu\text{m}$ in the homogenized state to $30 \pm 10 \mu\text{m}$ after six MDF passes, primarily driven by discontinuous dynamic recrystallization (DDRX). Remarkably, this Mg alloy exhibited a rare synergistic enhancement in both strength and ductility with ultimate tensile strength (UTS) increased by ~59 %, yield strength (YS) by ~90 %, while elongation improved by ~44 % unlike conventional severe plastic deformation (SPD) techniques that often sacrifice ductility for strength. This improvement is attributed to grain refinement, dispersion strengthening from finely distributed Mg_{12}Nd and Mg_7Zn_3 precipitates, and texture weakening, which facilitated the activation of non-basal slip systems. Despite the mechanical improvements, electrochemical corrosion testing in Hank's balanced salt solution (HBSS) at 37 °C revealed an increased corrosion rate from 0.1165 mm/year in homogenized condition to 0.2499 mm/year (after six passes of MDF). This was due to the higher fraction of low-angle grain boundaries (LAGBs), weak basal texture, and the presence of electrochemically active fine Mg_7Zn_3 particles. However, the corrosion rate remained within the acceptable range for bioresorbable implant applications, indicating a favorable trade-off between mechanical performance and degradation behavior. These findings demonstrate that MDF processing effectively enhances the strength-ductility synergy of Mg-rare earth alloys while maintaining a clinically acceptable degradation rate, thereby presenting a promising route for next-generation biomedical implants.

Keywords: Mg-Zn-Nd-Gd alloy; multidirectional forging; grain refinement; mechanical properties; corrosion and bioimplants

1. Introduction

Biodegradable implants are the new generation implants, gained more attention due to their degradable properties without nontoxic nature [1,2]. The major advantages of biodegradable implants are to avoid secondary surgeries, thus patient feel comfort and reduction of hospital stay

cost [3]. From few decades, polymer based biodegradable implants are used for surgeries and implantations [4]. Various polymer-based materials are polylactic acid (PLLA), poly (lactic-co-glycolic acid) (PLGA) and poly(e-caprolactone) (PCL) and some other polymer-based composites are developing to enhance the biological properties [5]. However, the primary problem with polymer-based composites are failing due to their lower strength and bacterial infections. It has been reported that, the bacterial infections are causing and developing after few weeks of surgery and thus leading to damaging and loosening of implant from the surgery location [6]. Thus, its causing irritation, discomfort to patients and arising side effects due to this limitations and problems. On other hand, during surgery procedures, the doctors should apply some external forces on implants to fix at specific location to heal the fracture. While applying these forces on these polymer-based implants are failing due to their inherent property of lower strengths [7]. Therefore, researchers are looking for metal based degradable implants which possess high, acceptable strength and good biological properties. The major metal degradable materials are magnesium (Mg) and Iron (Fe) based alloys [8]. However, Mg and its alloys are widely used as biodegradable metal implants due to their unique properties, such as biocompatibility and a young's modulus similar to natural bone, which make them more suitable than Fe-based alloys [9,10]. Various Mg based alloys such as Mg-Ca, Mg-Al, Mg-Zn and Mg-rare earth element alloys are being developed to produce high quality metal biodegradable implants [11,12]. The alloys such as Mg-Y, WE43, etc., are the recent wrought alloys used in real time clinical applications [13,14]. However, some challenges are associated with the currently available commercial Mg based implants. One of the major limitations is low strength and early degradation [15,16].

Recent studies are focused on developing Mg based biodegradable implants with sufficient strength, controlled degradation rates, and excellent biocompatibility. Several methods such as alloying [17–19], composites [20–22], modifying the microstructure using severe plastic deformation (SPD) techniques such as equal channel angular pressing [23], MDF or multi axial forging (MAF) [24], friction stir processing [25], high pressure torsion, accumulative roll bonding [26] and cyclic extrusion and compression [27]. Out of these aforementioned processes MDF/MAF has been adopted for processing the Mg materials at different processing conditions (strain and temperature). Further, this process is found to be the most promising and unique for miniature applications such as biomedical devices which require both high strength and acceptable corrosion resistance [28,29]. Anne et al. [30] characterised the Mg-4Zn alloy processed at 280 °C upto 5 Passes. The results reported that the corrosion properties increased with increasing number of MDF Passes. This was attributed to the grain refinement and thus increase in length/area of grain boundaries which acted as a physical barrier. The samples processed at 5 Passes exhibited the very fine grain structure and mechanical properties of the alloy increased upto 3 passes and reduced further which was a result of strain hardening and inverse Hall-Petch mechanism [30]. Similar results were observed by Ramesh et al. [31] when Mg-6Zn alloy was processed upto 5 Passes and obtained the minimum grain size 5 µm possess with large number of dislocations and high angle grain boundaries (HAGB's). There was a significant improvement in the tensile properties and corrosion resistance upto 5 Passes, which was attributed to the grain refinement, high dislocation densities and strain hardening.

Similarly, Cao et al. conducted MDF of Mg-4Zn-2Gd-0.5Ca alloy at 350 °C. They observed that the corrosion resistance of the alloy varied under different processing conditions, which they attributed to the presence of second phase particles, specifically Mg_3Zn_6Gd and $Mg_3Zn_3Gd_2$. Homogenized sample exhibited better corrosion resistance compared to MDFed samples. Further with increase in number of MDF passes $Mg_3Zn_3Gd_2$ volume fraction increased which led to greater corrosion rates [32]. Bahmani et al. [24] investigated the effect of processing temperature in MDF on XM11 Mg alloy containing 0.59 wt.% Ca and 0.52 wt.% Mn and obtained better tensile properties when processed at 300 °C whereas better corrosion properties were found at 220 – 300 °C. However, with increase in the temperature led to the increase in corrosion rate and was attributed to the action of second phase particles [24].

Nevertheless, several investigations reported that the second phase particles plays a significant role in enhancing the corrosion resistance in the Mg-rare earth alloys. However, the combination of mechanical properties and corrosion resistance is a rare aspect of Mg-rare earth alloys. Indeed, the development of high strength and corrosion resistance biocompatible Mg alloys is a quite challenging. Therefore, in the current study the authors attempted to develop the high strength corrosion resistant Mg alloys for bioimplant applications. For this, a Mg- rare earth alloy i.e Mg-Zn-Nd-Gd alloy has been casted using stir casting technique and later carried out the MDF till 6 Passes. Further, the microstructural, mechanical and corrosion properties has been studied. Finally, the effect of MDF processes on microstructures has been corroborated with corrosion resistance of the Mg-Zn-Nd-Gd alloy.

2. Materials and Methods

2.1. Multi Directional Forging

Details of procurement of metal powders and casting process for fabricating the Mg-Zn-Gd-Nd alloy is mentioned in the authors previous studies [12]. The chemical composition of the alloy is obtained from spark optical emission spectroscopy (OES) and it is given in the Table1.

Table 1. Chemical composition of Mg-Zn-Gd-Nd alloy.

Element	Zn	Nd	Gd	Zr	Mg
Composition (wt.%)	3.07	1.70	1.40	0.95	Balance

The complete experimental set-up details of the MDF is shown in Figure 1((a)-(c)). As-received blocks were homogenized as per ASTM B 661 at 400 °C for 24 h followed by air cooling to room temperature. The homogenized Mg-Zn-Gd-Nd alloy blocks were sectioned in to small rectangular samples of dimension 30 mm × 30 mm × 20 mm obtained through wire cut EDM machining. The samples were forged at a uniform strain rate of 2 mm/min at 250 °C. After each pass of MDF, the samples were rotated to its next forging axis, and the same is repeated for the whole cycle. Figure 1(d) shows the samples processed after MDF at different conditions.

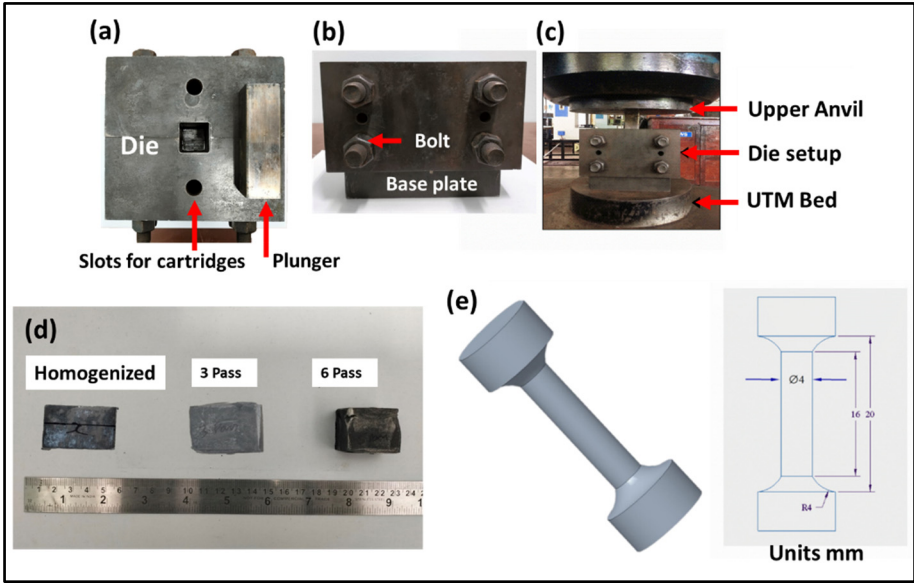


Figure 1. (a), (b), (c) Multidirectional forging components and (d) MDF samples processed at different conditions and (e) Tensile sample dimensions.

2.2. Microstructural Characterization

The samples for all the microstructure characterisations were extracted from the plane parallel to the final forging axis. The samples were sequentially polished by SiC abrasive papers with different granularities from 120 to 2000 grit size, followed by mechanical buffing using a velvet polishing cloth. To eliminate the scratches induced by mechanical grinding and to prevent the rise of temperature in this process, diamond suspension with a particle size of 1 μm – 3 μm along with kerosene was also utilized during elaborate polishing.

Samples were etched with a solution containing 10 g oxalic acid + 1 mL nitric acid + 4 mL acetic acid + 200 mL distilled water for 10 - 20 s. The surface morphology of homogenized, 3 Pass and 6 Pass samples were observed using an Optical Microscope (OM, Zeiss, Aixolab A1) and Scanning Electron Microscope (SEM, Carl Zesis) equipped with EDX. Samples for EBSD were prepared by sequential polishing using SiC abrasive papers from 120 to 2000 grit, followed by electropolishing at 16 V for 60 sec at -25 °C using Struers AC2 electrolyte as per ASTM E1558 – 09. The EBSD results were analyzed using HKL Channel 5 software. X-ray diffraction (XRD) analysis on the homogenized and MDF samples were performed using Empyrean 3rd Gen, Malvern PAN alytical diffractometer, within the range of 10° - 110° using Cu-K α source having a wavelength of 1.54 Å at a scan rate of 5 °/min. The linear intercept method has been employed to measure the grain size.

2.3. Mechanical Properties

Tensile samples were extracted from the blocks of homogenized and MDF samples as per ASTM E8 standards with a gauge length of 16 mm. The dimensions of the tensile samples were given in the Figure 1(e). Tensile testing of 3 samples from each condition (homogenized, 3 Pass and 6 Pass) was performed on the universal testing machine (Shimadzu AG-X plus™, 100 KN), at a cross head speed of 1 mm/min at room temperature. Here, the tensile testing axis is parallel to forging axis. The fracture surface morphology of the ruptured samples was analyzed through SEM. Microhardness test of homogenized and MDF samples were performed as per ASTM-E384 Standards on Omni Tech Microhardness testing machine. The samples were subjected to load 100 gf and dwell time 10 s were employed to calculate an average microhardness for all the samples.

2.4. Electrochemical Corrosion Testing

The electrochemical corrosion studies of homogenized and MDF samples of dimensions 10 mm $\times$ 10 mm $\times$ 4 mm in Hank's body solution at 37 $\pm$ 0.1 °C were investigated using a traditional three electrode system in an electrochemical workstation (OrigaFlex chemical workstation). Three electrode system had a saturated calomel electrode (SCE) as the reference electrode, platinum wire as the counter electrode and the sample as the working electrode, followed by which the whole three electrode setup was placed in a water bath at a temperature of 37 $\pm$ 0.1 °C. Prior measuring the open circuit potential (OCP) the samples were immersed for about 5 min in hank's solution to stabilize potential then OCP was measured for about 30 min from -100 to 1000 mV. Electrochemical Impedance Spectroscopy (EIS) measurements were made with a scan rate of 20 mV/sec at the frequency range of 100 KHz to 10 mHz. Potentiodynamic polarization (PDP) tests were then carried out with a scan rate of 1 mV/s after the EIS measurements. From the current density-potential data, tafel plots were generated. Corrosion rate (mm/y) is calculated according to ASTM G108. The corrosion rate is quantified using the following equation (1) [33] give below.

$$P_i = 22.85 I_{Corr} \quad (1)$$

Where, P_i is corrosion rate in mm/y and I_{corr} in mA/cm².

3. Results and Discussion

3.1. Microstructure and Phase Evolution of the MDF Samples

The OM and SEM microstructures of the homogenized and MDF (3 Pass and 6 Pass) samples are shown in Figure 2 and 3 respectively. The as-cast sample was homogenized at 400 °C for 24 h to obtain uniformity and to dissolve micro irregularities formed during casting and alloying. Homogenized sample exhibited equiaxed honey comb shaped grain structure (α -Mg) as shown in the Figure 2(a) with an average grain size of $118 \mu\text{m} \pm 5 \mu\text{m}$. Further, the grain boundaries were clearly visible and decorated with continuous eutectic network of second phase particles along the grain boundaries and fine particles in the grain interior. This can be clear from SEM microstructures given in Figure 3(a). After 3 Passes of MDF, microstructure exhibited a mixture of coarse and fine grains distributed in an uneven manner (Figure 2(b)) with average grain size of $40 \pm 10 \mu\text{m}$.

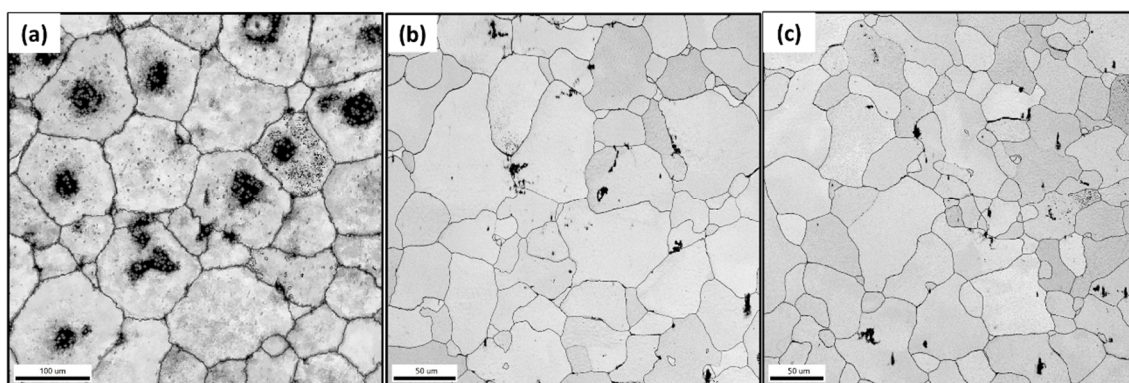
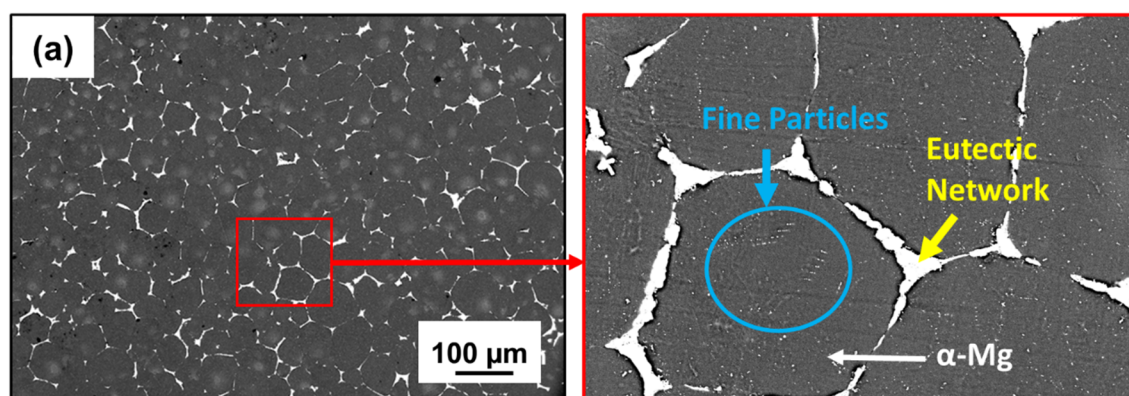


Figure 2. Optical Microstructures: (a) Homogenized, (b) 3 Pass and (c) 6 Pass samples.

A similar observations in the MDF processed Mg-rare earth alloys were reported by few researchers [34,35]. The obtained bimodal grain structure formed during MDF at a cumulative strain of $\epsilon_3=1.2$, driven by DDRX. This strain regime promoted partial recrystallization, resulting in a mix of fine DRXed grains ($\sim 1\text{--}5 \mu\text{m}$) and residual coarse grains ($\sim 20\text{--}110 \mu\text{m}$). After 6 Passes of MDF, further the grain size was reduced upto $30 \pm 10 \mu\text{m}$ due to subdivision of grains and the formation of new fine grains inside the grain. It has been observed that with increase in the cumulative strain ($\epsilon_6=2.43$) grain size decreases. The eutectic network of second phase particles observed along the grain boundary in the homogenized condition is fragmented during MDF process, dispersed and distributed non-uniformly through out the α -Mg matrix. This can be confirmed from the SEM microstructures provided in the Figure 3 (b) and (c). These second phase particles will enhance the DDRX through particle stimulated nucleation (PSN) leads to formation of fine grains along the grain boundaries and inside the grains [36]. Finally, the microstructure turns in to bimodal grain microstructure Figure 2(c).



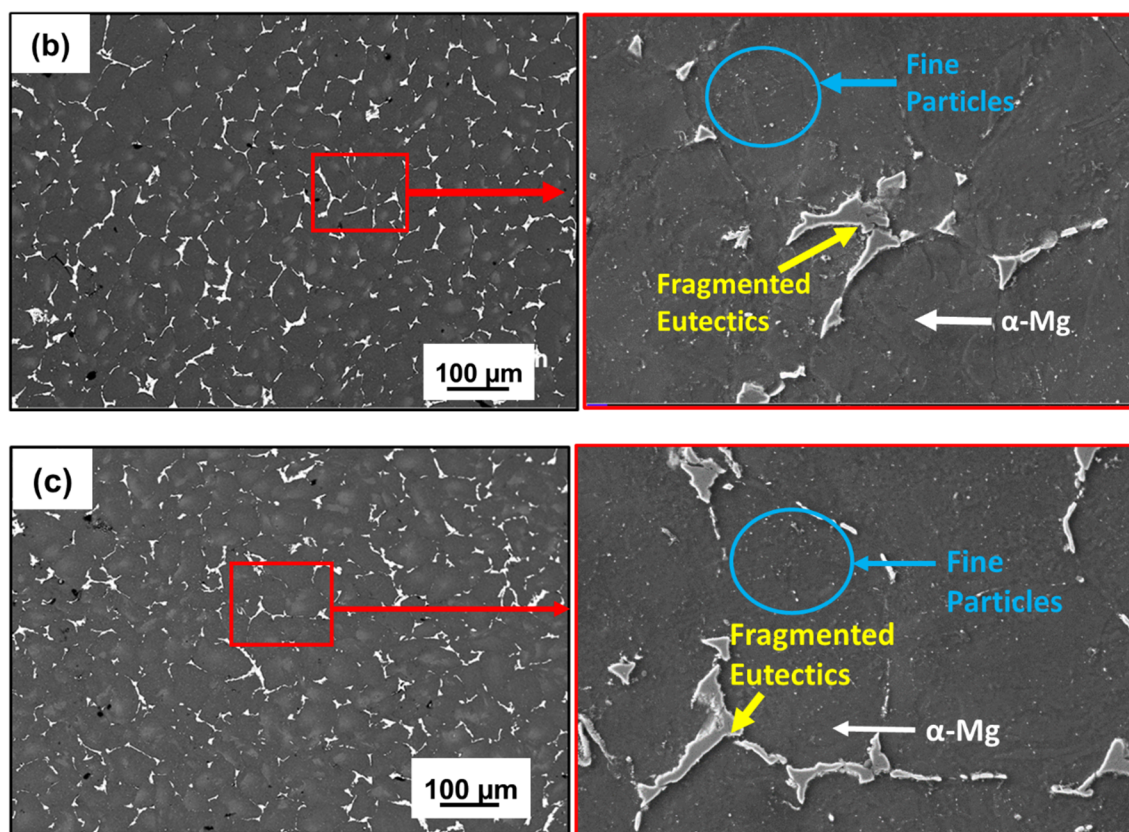


Figure 3. SEM Microstructures: (a) Homogenized (continuous eutectic network), (b) 3 Pass (semi-continuous eutectic network) and (c) 6 Pass samples (discontinuous eutectic network).

Figure 4 shows the EDS mapping of the homogenized and MDF samples. A higher percentage of α -Mg rich phases and Zn-Nd rich secondary phases were present in the microstructure of the Mg-Zn-Gd-Nd alloy, which was supported by the EDS mapping shown in the Figure 4(a) which revealed a distinct atomic contrast for the elements Mg, Zn, and Nd. The eutectic network of second phase particles in the homogenized sample had a continuous type distribution along the grain boundaries as shown in the Figure 3(a) and 4(a). After 3 Passes of MDF, second phase particles distribution was found to be semi-continuous in an uniform manner along the grain boundaries. The presence of very fine second phase particles near and inside the grain boundaries (Figure 3(b) and 4(b)) has also been observed. Further, after 6 Passes of MDF the second phase particle distribution was discontinuous along the grain boundaries and were distributed in a non-uniform manner (Figure 3(c) and 4(c)). The density of fine second phase particles were increased compared to 3 Pass and the same can be reflected in the Figure 3(c) and 4(c). This is mainly due to high cumulative strain during the MDF process and encourages to the formation of strain induced precipitation and similar observations were reported by Cao et al [32] and Yang et al. [37].

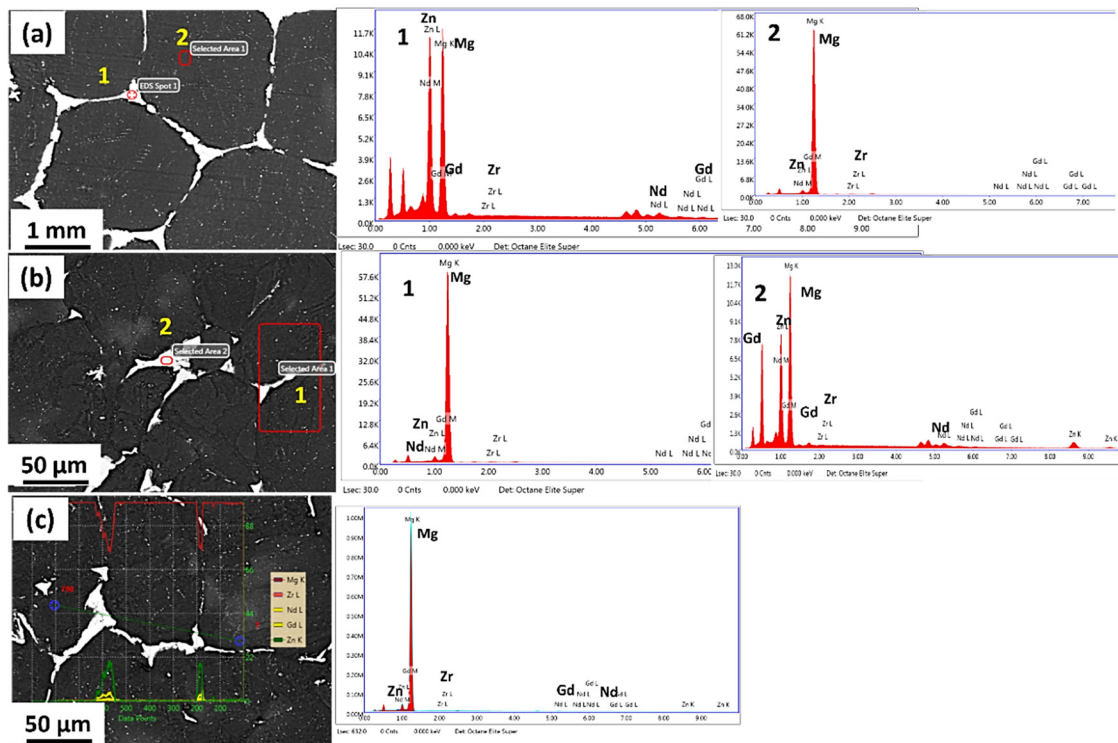


Figure 4. EDS Mapping: (a) Homogenized, (b) 3 Pass and (c) 6 Pass samples.

Figure 5 shows the XRD patterns of homogenized and MDF samples. For all the three conditions it has been exhibited α -Mg as the major phase, however after MDF Passes the peaks corresponded to different second phase particles such as MgZn , Mg_7Zn_3 and Mg_{12}Nd . The variation in the intensity of the peaks corresponding to Mg_{12}Nd and Mg_7Zn_3 at different processed conditions can be observed due to the dissolution of particles, further the major second phase particles of Mg-Zn-Gd-Nd alloy can be considered as Mg_{12}Nd and Mg_7Zn_3 as observed in XRD and the EDX composition, these observations were consistent with authors reported in [12,38]. Hence, through XRD phase analysis it is confirmed that the eutectic network is Mg_{12}Nd and fine second phase particles are Mg_7Zn_3 . Further, no peaks corresponded to GdZn phase in 6 Pass samples due to the dissolution of particles.

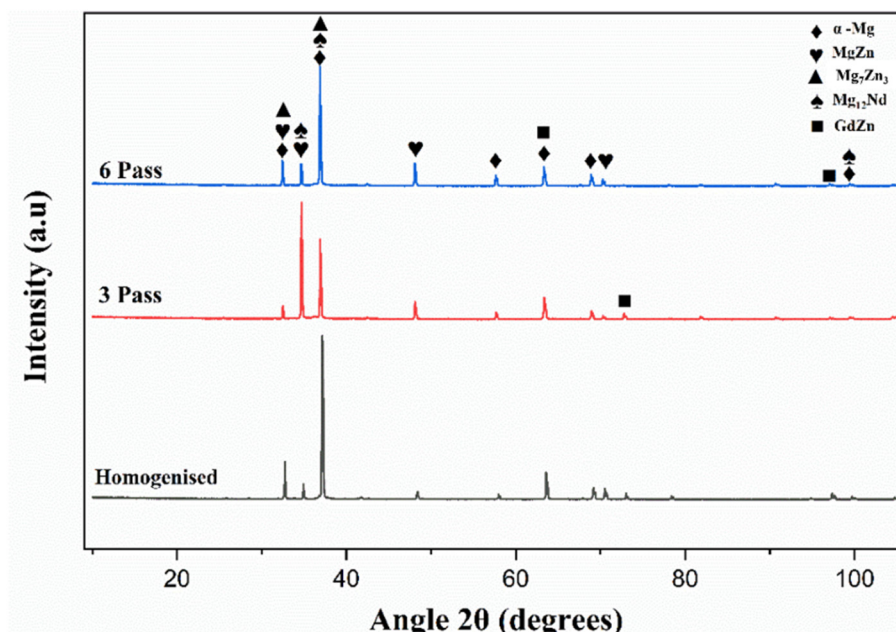
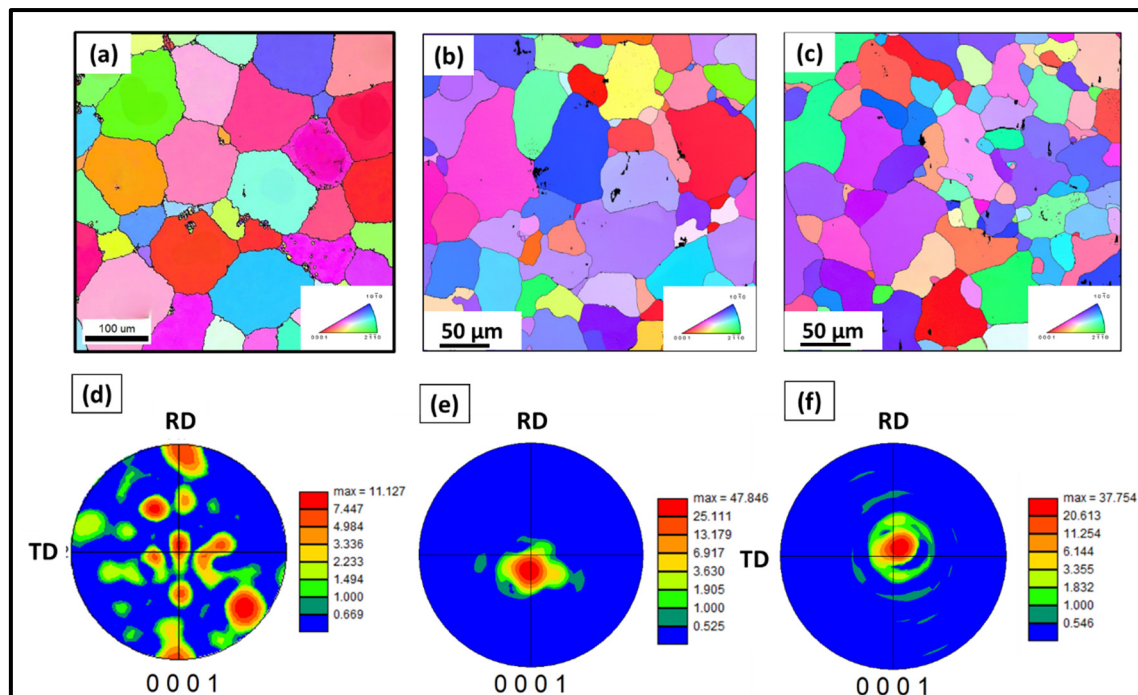


Figure 5. XRD patterns of the homogenized and MDF samples.

Inverse pole figure (IPF) and pole figure (PF) maps has been obtained through EBSD analysis of samples processed at different conditions are indicated in the Figure 6. It has been observed that, the homogenized sample exhibited an equiaxed honeycomb type grain structure with average grain size of $118 \pm 5 \mu\text{m}$ (Figure 6(a)). Upon MDF processing a mixture of both fine and coarse grains i.e., a bimodal grain structure has been observed in both 3 Pass and 6 Pass samples and further no traces of twins are generated (Figure 6(b) and (c)). This can be attributed to mainly three possible reasons: (i) the high temperature during MDF (ii) the orientation of grains during deformation such that restricted the twinning activation and (iii) reduction in grain size (for 3 pass $40 \pm 10 \mu\text{m}$ and for 6 pass $30 \pm 10 \mu\text{m}$) upon increase in strain accumulation with increase in MDF Passes. However, a significant reduction in grain size is observed after MDF Passes. Primarily, a random texture (Figure 6(d)) has been observed in the homogenized sample. After MDF, the random texture turned in to near-net strong basal texture. The basal (0001) PF's (Figure 6(e) and (f)) confirms the presence of texture but slightly weakened along the basal plane (0002) after MDF Passes. The presence of random texture in the Mg-rare earth alloys after SPD is quite common and widely reported [39–42].

The distribution of grain boundary misorientation angles is presented in Figure 6(g). A marked increase in the fraction of LAGBs ($1\text{--}15^\circ$) is observed in the MDF processed samples, rising from 48 % in the homogenized condition to 84 % post-processing. This increase is primarily attributed to the formation of fine sub-grains and a high density of dislocations generated during the MDF process. Concurrently, a significant reduction in the fraction of HAGBs ($15\text{--}180^\circ$) is observed, decreasing from 52 % in the homogenized sample to 17 % in the MDF processed samples. Notably, the maximum fraction of HAGBs was found in the homogenized condition. The higher fraction of HAGBs in the homogenized sample is known to enhance corrosion resistance in Mg-rare earth alloys [43], a point that will be further discussed in Section 3.3 on corrosion behavior.



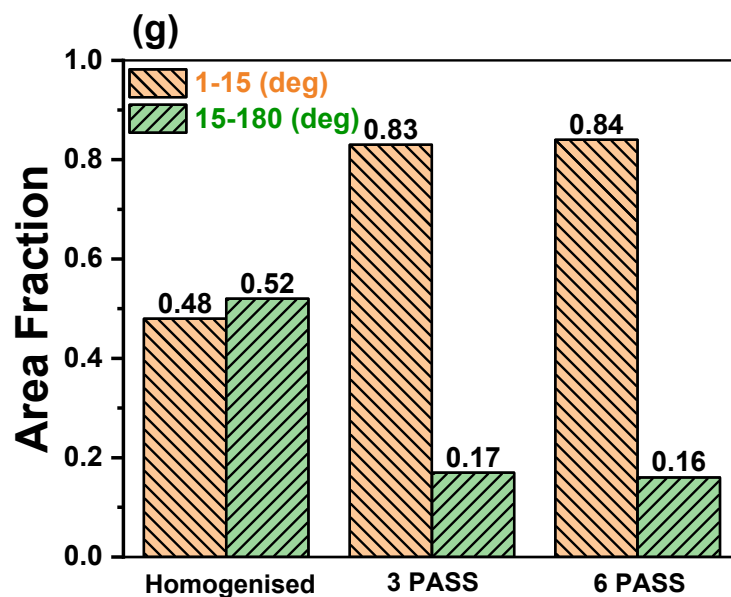


Figure 6. Inverse pole figures: (a) Homogenized, (b) 3 Pass and (c) 6 Pass samples; Pole figures: (d) Homogenized, (e) 3 Pass (f) 6 Pass samples, and (g) grain boundary misorientation distribution.

3.2. Mechanical Response

3.2.1. Tensile Behavior and Fracture Analysis

Engineering stress – strain curves of homogenized and MDF samples are plotted in the Figure 7(a). Figure 7(b) summarizes the mechanical properties of samples processed at different conditions. It has been observed that the YS, UTS and % of elongation has been increased with increasing number of MDF Passes. The YS and UTS of homogenized sample were 96 ± 3 MPa and 151 ± 4 MPa respectively. The maximum UTS is observed in the 6 Pass sample (250 ± 7 MPa), followed by 3 Pass (219 ± 6 MPa) and then homogenized (151 ± 4 MPa). Similar trend has been observed in the YS and % elongation of the 6 Pass sample. Indeed, it is noticed that overall 59 % increase in UTS and 92 % increase in YS and 40 % increase in elongation is observed in the 6 Pass sample compared to the homogenized sample. This unique combination of highest strength and ductility is significant and very special for bioimplant applications [44]. The increase in the YS and UTS after MDF can be primarily attributed to MDF induced grain refinement due to high strain accumulation and dispersion strengthening from second phase particles $Mg_{12}Nd$ and Mg_7Zn_3 [45,46]. While after 6 Passes of MDF, the continuous eutectic network had fragmented in to discontinuous particles and distributed non-uniformly in the α -Mg matrix. This could restricted the premature failure for the sample and enhances the ductility. Further, the significant enhancement in the elongation of the 3 and 6 Pass samples is primarily attributed to weak basal texture observed in the Figure 6(e) and (f) obtained after MDF process. The weakening of texture in Mg-rare earth alloys helps in enhancing the ductility of samples. This phenomenon is well addressed and documented by the researchers and authors previous findings [41,47]. In addition, the MDF samples exhibit outstanding elongation because activation of different slip systems that are introduced after texture randomization/weakend really boosted the elongation [48,49].

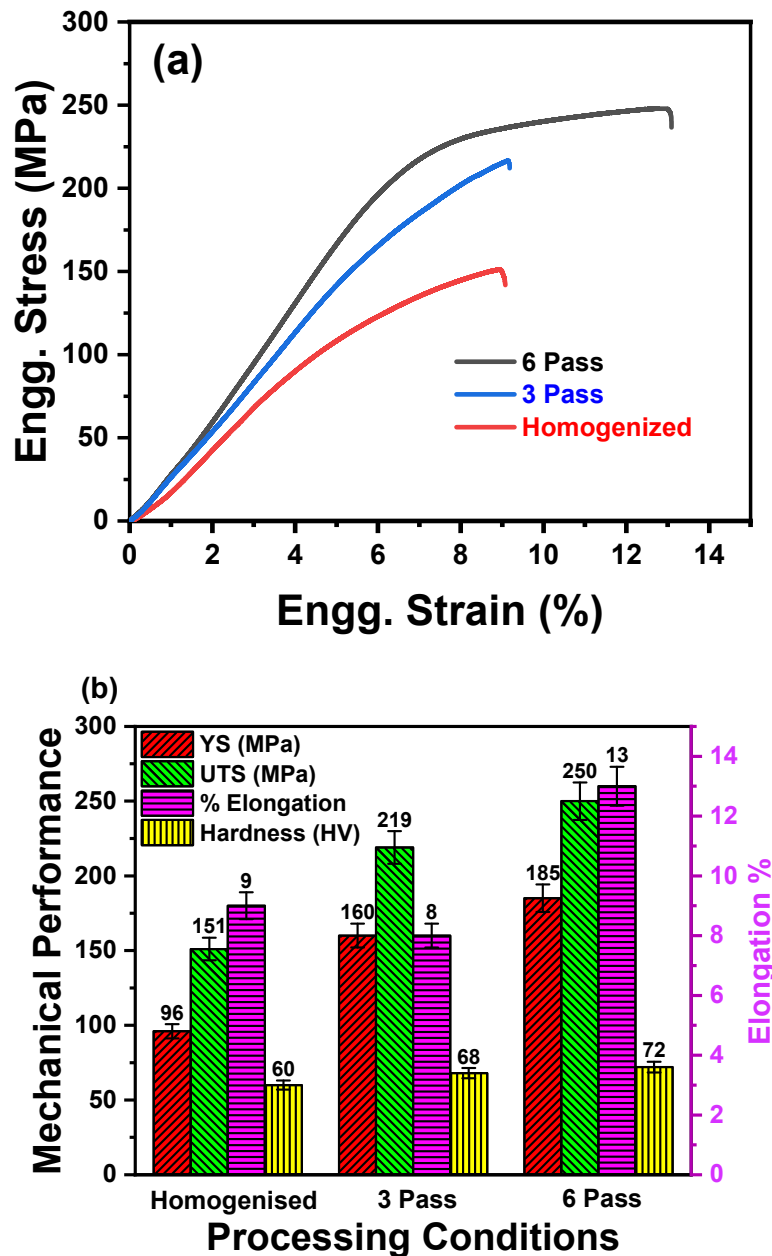


Figure 7. (a) Engineering Stress – Strain curve of samples processed at different conditions and (b) Comparison of UTS and elongation % of samples processed at different conditions.

3.2.2. Microhardness Evolution

The microhardness results of the samples processed under different conditions are presented in Figure 7(b). The homogenized sample exhibited an average microhardness of 60 ± 1 HV, which was the lowest among all conditions. A notable increase in microhardness was observed after MDF of the Mg-Zn-Nd-Gd alloy. Specifically, the samples processed through 3 and 6 passes exhibited average microhardness values of 68 ± 1.2 HV and 72 ± 1.7 HV, respectively. However, the increase from 3 to 6 passes was relatively modest, which can be attributed to the complete dissolution of the GdZn phase after 6 passes, as confirmed by XRD analysis (Figure 5). Overall, the MDF process led to an enhancement of approximately 20% in microhardness compared to the homogenized condition. This improvement can be primarily ascribed to grain refinement (Figure 6), strain hardening, and dispersion strengthening resulting from the presence of secondary phase particles such as Mg_{12}Nd and Mg_7Zn_3 along both the grain boundaries and interiors of the MDF-processed samples [50].

Figure 8 reveals the fracture surface of the homogenized and MDF samples. The homogenized sample fracture surfaces revealed tearing ridges and micro cracks leading to macro-ones (Figure 8(a)).

The presence of tearing edge and cleavage facets is an indication of brittle fracture, the converging of certain micro cracks to macro crack leading to the formation of tearing edge has been observed (Figure 8(a)). Mg alloys usually have brittle type of failure due to the restricted dislocation system; the formation of the cleavage facets can be attributed to the presence of large α -Mg grains [51]. However, the samples processed for 3 Pass exhibited the presence of dimples of different dimensions and also river like pattern as shown in the Figure 8(b). This indicates that the 3 Pass samples showed a mixed mode of fracture (brittle + ductile) with considerable percentage of elongation. This can be attributed to the grain refinement as a result of MDF processing. Reduction in tearing edges and the size of dimples was observed in samples processed upto 6 Pass as shown in the Figure 8(c). Compared to the 3 Pass and 6 Pass samples had a greater grain refinement in microstructure as a result the reduction in dimple size and increase in elongation percentage could be observed. Fracture of samples processed at 6 Pass is a combination of brittle and ductile fracture and more dominated by ductile nature. The depth of the dimple depends on microstructure and plasticity of material [52,53].

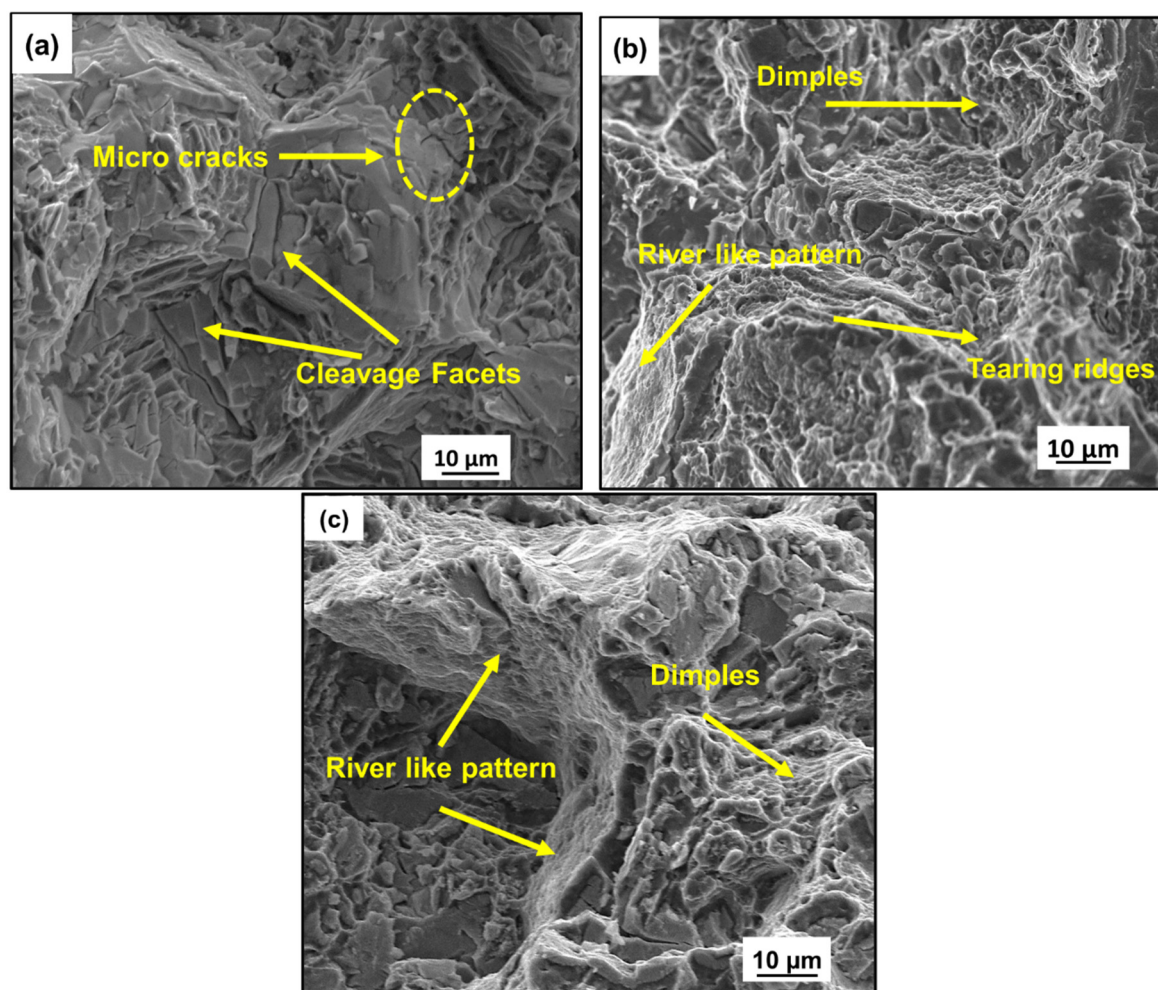


Figure 8. Fractography images: (a) Homogenised, (b) 3 Pass and (c) 6 Pass samples.

3.3. Electrochemical Corrosion Behavior

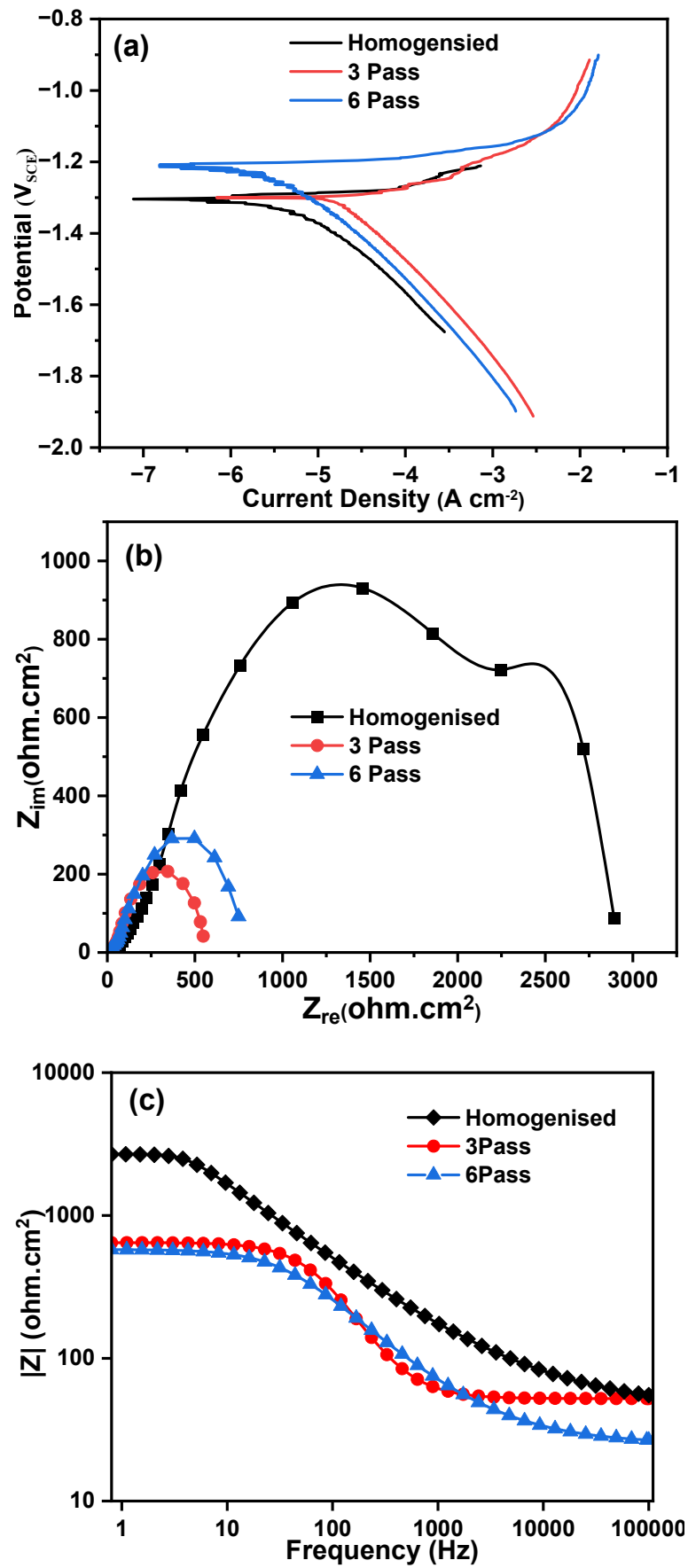
3.3.1. Potentiodynamic Polarization Studies

Potentiodynamic polarization curves of the homogenized and MDF samples tested at 37 ± 1 °C in hank body solution is plotted in the Figure 9(a). The measured parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic slope (β_a) and cathodic slope (β_c) were calculated from Tafel plots and tabulated in the Table 2. From the Table 2, it can be inferred that corrosion rate increased after MDF process. However, the corrosion current density is directly proportional to the corrosion density, with increase in corrosion current the corrosion rate also

increases. Cathodic curve represents the evolution of hydrogen when subjected to corrosion whereas anodic curve represents the dissolution of Mg. All the samples have similar polarization curve trends having a steep increase in anode curves due to the negative difference effect. Table 2. shows that the homogenized sample had a corrosion potential of -1.2799 V and corrosion current density of 0.0051 mA/cm², which is the lowest among all the samples processed in different conditions indicating a lesser corrosion rate of 0.1165 mm/yr. From the Figure 9(a), it is observed that, homogenized sample exhibited the filiform type of corrosion, and little part is affected by pitting corrosion. The area fraction of HAGB's is maximum (55 %) for homogenized sample. A passive layer is formed due to the high fraction of HAGB's, which could enhanced the corrosion resistance for homogenized sample [54]. After 3 and 6 Pass of MDF, corrosion potential shifted to -1.2967 and -1.1999 respectively which is decreased slightly with increase in corrosion current density compared to the homogenized sample, hence having a slightly higher corrosion rate of 0.4560 mm/yr and 0.2499 mm/yr for 3 and 6 Pass respectively. In strongly textured Mg alloys, Song et al.[55] noticed that surfaces that had higher {0002} basal planes exhibited more corrosion resistance than those with lesser basal planes. In addition, Xin et al. [54] had investigated the effect of texture on corrosion behaviour of AZ31 Mg alloy, and observed similar results which were noticed by the Song et.al. Here, the MDF samples exhibited near-net strong basal texture (weak texture) and the presence of high discontinuous distribution of LAGB's could lead to galvanic corrosion attack at the grain boundaries. Grain boundaries and LAGB's exhibit more active sites and energy compared to the substrate, which acts as a highly active site. Consequently, grain refining enhances the area of grain boundaries and LAGB's, resulting in an enhanced dissolution rate of atoms at the grain boundaries and augmenting the chemical reactivity of the alloy [56]. In addition, Cui et al. [57] studied, the effect of grain size and second phase particles on corrosion behaviour of Mg-3Al-5Pb-1Ga-Y alloy. They found that, the presence of high fraction of LAGB's and fine second phase particles enhanced the corrosion rate. After 3 pass, the bimodal grain structure (coarse unrecrystallized + fine DRXed grains) introduced moderate surface energy heterogeneity, with corrosion initiating preferentially at fine-grained regions (higher energy). The further refined microstructure increased the total grain boundary area and surface free energy after 6 pass, leading to a more pronounced corrosion rate. The presence of high fraction of grain boundary is further confirmed with the grain boundary distribution presented in the Figure 6(g). The weakened texture also reduced the fraction of corrosion-resistant basal planes, exacerbating material degradation [58].

Table 2. Corrosion properties of the samples processed at different conditions.

Sample Condition	I _{corr} (mA/cm ²)	E _{corr} (V)	β _a	β _c	CR (mm/y)
Homogenized	0.0051	-1.2799	0.0071	-0.1987	0.1165
3 Pass	0.0199	-1.2967	0.0285	-0.2557	0.4560
6 Pass	0.0109	-1.1999	0.0044	-0.2606	0.2499



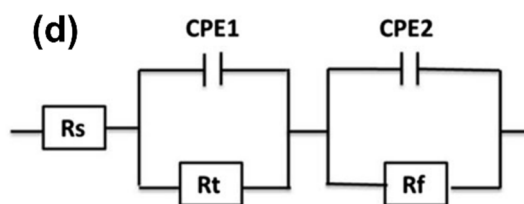


Figure 9. (a) Potentiodynamic polarization curves, (b) Nyquist Plot, (c) Bode plots of samples processed at different conditions and (d) Equivalent EIS Circuit.

3.3.2. Electrochemical Impedance Spectroscopy

Nyquist and Bode plots of Homogenized and MDFed samples in Hank's body solution at 37 ± 0.1 °C are shown in the Figure 9(b) and (c). EIS provides the inductive and capacitance loop and also the used to investigate the electrochemical interface between electrode and electrolytes. Nyquist plot depicts a semicircle which indicated the capacitance arc and the formation of protective film and degradation of it. The capacitance arc's diameter represents the corrosion resistance of Mg-Zn-Gd-Nd alloy, from the Figure 9(b) it can be observed that the all the samples exhibited depressed semicircles also homogenized sample had larger semicircle followed by 6 and 3 Pass samples indicating better corrosion resistance for homogenized sample. Equivalent circuit used to analyze the Nyquist is shown in the Figure 9(c). High frequency capacitive loop of electrode reaction process was described using a parallel circuit of electric double layer capacitance cell (Cdl) and charge transfer (R_t). Constant phase elements (CPE1 and CPE2) and a film resistance R_f were introduced to the circuit. If the metals exhibit better corrosion resistance, it is difficult to transfer charges between the solution and the sample surface, so R_t becomes higher. It is noticed that, R_t values for homogenized, 3 Pass and 6 Pass samples are 244.5 ohm.cm², 34.9 ohm.cm² and 144.5 ohm.cm² respectively. Therefore, the corrosion resistance for homogenized sample increased when compared to MDF samples.

The corrosion rate of MDF processed samples upto 3 Pass has been declined compared to homogenized sample. However, there is a slight decrease in corrosion rate of 6 Pass samples compared to 3 Pass samples and the primary reason can be attributed to the type, phase and distribution of the second phase particles present in the microstructure. However, the corrosion resistance depends on the electrode potential of the matrix and the adjoining phases, the majority of the second phase particles identified through EDX and XRD is Mg₁₂Nd and Mg₇Zn₃ (Figure 4(b and c) and Figure 5(b and c)), these phases will corrode first if the potential of the phase is lower than that of the substrate. From the Figure 11, it is noticed that, 3 and 6 pass samples have experienced the pitting corrosion. The corrosion potential of second phase particles are lower than Mg substrate, so that the second phase particles acted as anode and dissolved preferentially [59]. However, as reported in the microstructure results the distribution of the second phase particles Mg₁₂Nd was continuous among the grain boundaries in homogenized sample whereas semi-continuous with respect to 3 and 6 Pass samples. Therefore, it cannot provide better protection to the substrate if its distribution is discontinuous. The corrosion resistance of Mg alloy is closely related to the length of grain boundary which acts as a physical barrier [60]. The fragmented fine second phase particles acts as cathode near the LAGB's and α -Mg matrix acts as anode, which forms a micro galvanic couple. Figure 11(b) and (c), it is observed that pitting corrosion occurred at the grain boundaries region for the both samples due to the severe galvanic effect which could increased corrosion rate after MDF on Mg-Zn-Nd-Gd alloy.

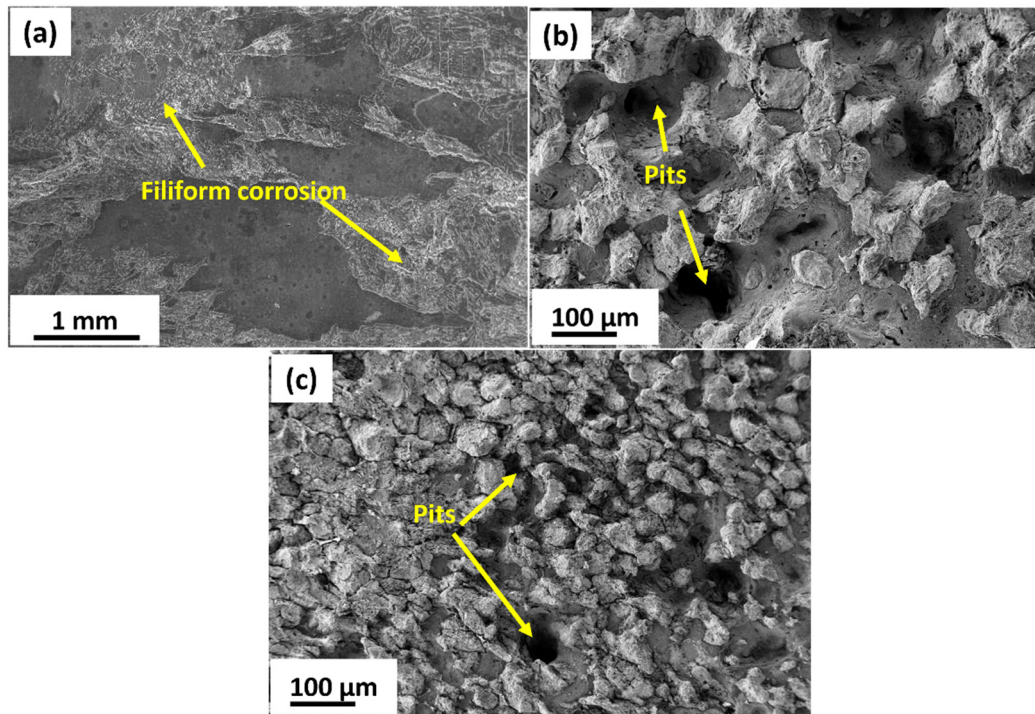


Figure 10. Surface morphologies after corrosion testing: (a) Homogenized, (b) 3 Pass and (c) 6 Pass samples.

4. Conclusions

The biodegradable homogenized Mg-Zn-Gd-Nd alloy was successfully processed through MDF up to 6 Passes at 250 °C with a cumulative strain of 2.43. Mechanical and microstructural characterizations were carried out to study the effect of MDF on the Mg-Zn-Gd-Nd alloy. To evaluate its suitability for biomedical applications, the corrosion behavior of the alloy was analyzed in Hank's body solution at 37 °C under various processing conditions. Key findings from the study are summarized below:

- Grain size reduced from $118 \pm 5 \mu\text{m}$ in homogenized condition to $30 \pm 10 \mu\text{m}$ after 6 passes with uniform distribution of Mg₂Zn₃ fine particles and semi-continuous Mg₁₂Nd eutectic network.
- The microhardness of the 6 Pass MDF sample increased by 20 %, while UTS and YS exhibited significant enhancements of 59 % and 90 %, respectively compared to homogenized sample. This is due to the fact that the grain refinement, strain hardening and dispersion strengthening from the second phase particles.
- Additionally, the ductility of the 6 Pass MDF sample improved remarkably, with a 44% increase in elongation, attributed to texture weakening and uniform distribution second phase distribution (resist premature failure).
- Homogenized sample exhibited better corrosion resistance compared to MDF processed samples. Although the corrosion rate of 6 Pass MDF processed sample (0.2499 mm/yr) is higher than that of homogenized samples (0.1165 mm/yr), this remains within an acceptable range for biodegradable implant applications.
- The slight reduction in corrosion resistance of the MDF processed samples can be attributed to high fraction of LAGB's and fine second phase particles; however, it does not compromise the alloy's suitability for biomedical use. Instead, the controlled degradation rate aligns well with the clinical requirements for biodegradable implants, where gradual material dissolution is essential for tissue regeneration.

Data Availability: The data that support the findings of this study can be requested from the corresponding author upon reasonable request.

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