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Improvement in electrochemical corrosion resistance of Mg–Al–Zn alloy by 250 keV carbon ions irradiation

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Abstract

Mg–Al–Zn alloy (AZ91D) was irradiated with carbon ions at different doses ($1 \times 10^{13}$, $5 \times 10^{13}$, $1 \times 10^{14}$, $5 \times 10^{14}$ and $1 \times 10^{15}$ ions cm$^{-2}$) by using Pelletron Accelerator. Ion energy was kept constant at 250 keV for all the irradiated samples. The range of carbon ions inside the alloy was found to be 0.67 μm. X-ray diffraction results indicated that the carbon ions irradiation of Mg alloy increases the intensity of Mg (002) plane. The average crystallite size of Mg alloy was decreased at $1 \times 10^{13}$ ions cm$^{-2}$ whereas an increase in the crystallite size was observed by further increasing the ion dose. The FTIR data indicated C–H, C–O, C=O and C-C vibrational modes in the irradiated samples. The electrochemical corrosion of Mg–Al–Zn alloy was investigated through potentiodynamic polarization tests in 0.9% NaCl solution. The open circuit potential (OCP) did not change at lower ion doses (up to $1 \times 10^{14}$ ions cm$^{-2}$), however, the OCP became more positive as the ion dose was increased to $1 \times 10^{15}$ ions cm$^{-2}$. Similarly, corrosion current density and corrosion rate were significantly decreased after carbon irradiation at $1 \times 10^{15}$ ions cm$^{-2}$. The SEM results revealed severe surface pitting of the unirradiated alloy that was reduced after the carbon ions irradiation.

1. Introduction

Magnesium (Mg) alloys are receiving tremendous attention these days due to their immense biomedical applications. The properties of Mg alloys are compatible with bone tissues, which makes them superior biomedical materials as compared to the stainless steel [1–3]. However, magnesium alloys deteriorate automatically in the physiological environment, unlike the passive alloys like stainless steel and titanium alloys, thus making the implant removal surgery redundant [4, 5]. Although, the deterioration rate is quite fast to fulfill the clinical requirements, particularly in the primary stage [6–8], however, due to the dynamic process of deterioration, it is critical to enhance the corrosion resistance of Mg alloys. Due to their natural biodegradable properties and good strength [9–11], magnesium alloys find a great prospect in the biomedical implants [12–14]. Mg alloys exhibit high strength to weight ratio, low density, fine dimensional stability, high creep strength, good vibration dampening, high electrical/thermal conductivity and high impact resistance. These days, with the moderate development in overall properties, particularly in the corrosion protection, magnesium alloys are being used progressively as structural materials in automotive, aerospace, and in biomedical applications [15–22].

Various surface modification techniques have somehow resolved the corrosion issues of magnesium alloys to some degree, however, the corrosion protection of Mg and its alloys is still limited due to their high chemical reactivity [23–27]. This restricts their applications to broader fields, particularly in aggressive environments [28, 29]. Hence, proper surface treatments are necessary to develop protective films, which play an important role in the corrosion protection of magnesium and its alloys. The surface modification techniques such as micro
arc oxidation, physical vapor deposition and ion implantation were used to improve the corrosion resistance of Mg alloys previously [30–47]. Research groups [32–47] proposed that the ion implantation is a suitable technique to enhance the corrosion resistance of magnesium alloys by developing a stable passive layer on its surface. Different ions including Cr, Ti, Al, Zr, Zn, Ce, Y, Ta, O2, and N2 were implanted in magnesium and its alloys [32, 36, 39–46]. The oxygen was implanted after Cr [36] and Ti [47] ion implantation to avoid galvanic effects and to develop surface oxide layers. Such ions were suggested to restrain fast deterioration of magnesium and its alloys. Compact surface oxide layers, developed by ion implantation, increased the corrosion resistance of the Mg alloys [48].

The carbon-based biomaterials with good biological characteristics like pyrolytic carbon [49, 50] and diamond-like carbon [51, 52] have established clinical acceptance because carbon is an important element which exhibits good biocompatibility. Carbon ion has favorable physical and chemical properties and these can react with the metallic element to form metal carbides [53]. Hence, carbon ion implantation is a good method to increase the corrosion resistance and biological properties of magnesium alloys. To the best of our knowledge, there is a limited work on the carbon ion implantation/irradiation on Mg and its alloys [54, 55]. Mumtaz [55] studied the mechanical properties and corrosion resistance of 500 keV carbon ion irradiated magnesium alloy AZ91D within the dose range of 1.26 × 1013 to 8.4 × 1015 ions cm−2. In this study, hardness of Mg alloy was increased whereas the sample mass (measured through weight loss method) was decreased after carbon irradiation. No open circuit potential, corrosion rate, corrosion current density and corrosion potential were studied in this work. There was insignificant change in mass loss of samples irradiated with 500 keV carbon ions within the dose range of ∼1013 ions cm−2. The ion energy and dose play an important role in controlling the corrosion rate of the Mg alloys. The penetration of ions inside the alloy surface can be controlled by controlling their energy. Lower the ion energy, lesser will be the ion penetration and vice versa. Small changes in the ion energy can significantly affect the surface properties of metallic materials [56]. The low energy carbon ions might be more effective in controlling the corrosion rate of metals and alloy as these can go only to few nanometers inside material’s surface [57]. Moreover, the use of high carbon dose irradiation (particularly of the order of 1015 ions cm−2) could be quite useful in improving the corrosion resistance of the Mg alloys. Therefore, in this work, the Mg–Al–Zn alloy (AZ91D) was irradiated with low energy (250 keV) carbon ions in a wide range of doses from 1 × 1013 to 1 × 1015 ions cm−2 and electrochemical corrosion rate and corrosion current density of unirradiated and irradiated samples were investigated through potentiodynamic polarization technique.

2. Methodology

Mg–Al–Zn alloy (AZ91D) was used in this study. The chemical composition of Mg alloy was about 91% Mg, 8% Al and 1% Zn. The alloy was obtained in the form of a solid bar that was cut into square pieces of dimensions 1.5 × 1.5 cm2 through a computerized numerical cutting (CNC) machine. The samples of the Mg alloy were grinded first with the help of silicon carbide (SiC) papers of grit size 1000, 1500 and 2000. After grinding, the samples were polished by polishing machine as well as with the diamond paste (0.3 μm). The polished samples were irradiated by carbon ions by using the Pelletron tandem accelerator. For irradiation, the samples were fixed inside the accelerator chamber under vacuum. The samples were irradiated with 250 KeV carbon ions at room temperature. In order to vary the ion dose, irradiation time was varied such that the desired doses of 1 × 1013, 5 × 1013, 1 × 1014, 5 × 1014 and 1 × 1015 ions cm−2 were obtained. After irradiation with carbon ions, both unirradiated and ion-irradiated samples were tested with x-ray diffractometer (for structural study), scanning electron microscope (for morphological features) and Gammy Potentiostat (for the electrochemical behavior). For the electrochemical testing, the unirradiated and carbon ions irradiated samples were mounted on copper plates having an exposed area of 2.31 cm2 while the remaining area was covered with the acidic resistance epoxy. Three electrode system containing Mg–Al–Zn (working electrode), Ag/AgCl (reference electrode), Graphite rod (counter electrode) was used. A distance of ~1 mm was maintained between the working and counter electrode with the help of luggage probe [58]. The NaCl (0.9%) was used as electrolyte for the electrochemical testing and the cell temperature was maintained to 37 °C during testing. Open circuit potential test was run for 1 hr to stabilize the potential and to maintain the equilibrium between working electrode and electrolyte. Potentiodynamic polarization was performed and potential-current scans were obtained by applying initial potential of −2 V versus Eoc to final potential of 1.5 V versus Eoc for each specimen.

3. Results and discussion

3.1. Structural study

Structural study of Mg–Al–Zn alloy (AZ91D) was performed by using x-ray diffraction (XRD) technique. The XRD graphs of Mg–AZ91D alloy are presented in figures 1(a)–(f) and 2(a). In figures 1(a)–(f), XRD patterns of
unirradiated and carbon ion irradiated Mg-AZ91D alloy at different doses are shown separately whereas the figure 2(a) displays a comparison of the XRD patterns of unirradiated and irradiated samples at various doses. As shown by the XRD graph of unirradiated sample, two phases of Mg such as $\alpha$-Mg (pure Mg) and $\beta$-Mg (Mg$_{17}$Al$_{12}$) are present in the alloy whose diffraction peaks exist along various planes. In the case of $\alpha$-Mg, diffraction occurs along $(100)$, $(002)$, $(101)$, $(102)$ and $(103)$ planes. For $\beta$-Mg phase, the diffraction peaks are formed corresponding to $(411)$, $(332)$, $(422)$ and $(651)$ planes. After irradiation with 250 KeV carbons ions of dose $1 \times 10^{13}$ ions cm$^{-2}$, intensity of Mg $(002)$ peak increases as compared to the intensities of other diffraction peaks of $\alpha$-Mg. In addition, slight increase in the full width at half maximum of $\alpha$-Mg peaks is also noticeable. On the other hand, the $\beta$-Mg peaks are found to exhibit slightly lower intensity as compared to the unirradiated

![Figure 1. X-ray diffraction results of Mg–Al–Zn alloy before and after carbon ion irradiation.](image-url)
sample. This shows the partial dissolution of Mg17Al12 intermetallic precipitates in the parent matrix. By increasing the ion dose to \( 5 \times 10^{13} \text{ions cm}^{-2} \), intensities of \( \alpha \)-Mg peaks almost remain constant. Only a small increase in the intensity of Mg (103) peak is seen from the figure 1(c). The \( \beta \)-Mg peaks are also not much changed by increasing the dose from \( 1 \times 10^{13} \text{ions cm}^{-2} \) to \( 5 \times 10^{13} \text{ions cm}^{-2} \). When the carbon dose is increased to \( 1 \times 10^{14} \text{ions cm}^{-2} \), intensities of Mg (100), Mg (101) and Mg (110) peaks increase while the intensity of Mg (103) peak is reduced. Intensity of Mg (002) peak almost remains same as observed at lower ion doses. Further, intensity of Mg17Al12 peaks are not much changed with increase of ion dose from \( 1 \times 10^{13} \text{ions cm}^{-2} \) to \( 5 \times 10^{13} \text{ions cm}^{-2} \). There is an insignificant change in the \( \alpha \)-Mg and \( \beta \)-Mg peaks intensities when the carbon ion dose increases to \( 5 \times 10^{14} \text{ions cm}^{-2} \). At the highest carbon dose of \( 1 \times 10^{15} \text{ions cm}^{-2} \), the diffraction from the Mg (002) peak is reduced, however, it is still higher than that of its intensity in the case of untreated sample. In addition, the intensities of the other peaks corresponding to \( \alpha \)-Mg and \( \beta \)-Mg are also changed.

Figure 2. (a) Comparison of XRD results of Mg alloy at different carbon doses. (b) Variation in average crystallite size of Mg alloy with ion dose. (c) Variation in average micro-strain of Mg alloy with ion dose.
The structural results show that the carbon ion irradiation of Mg alloy AZ91D enhances the growth of Mg (002) plane whereas the anomalous variations in the intensities and FWHM of the other α-Mg and β-Mg peaks are observed with increase of the carbon dose. The increase in Mg (002) peak intensity after carbon irradiation signals towards the diffusion of the atoms along (002) plane thus indicating the dominance of the (002) diffraction peak as compared to the other peaks. This happens because of the excessive thermal energy is transferred to the Mg matrix during the carbon ion bombardment. This thermal energy originates from the rise in the temperature of the localized region of the samples as predicted by the thermal spike model of radiations [56, 57].

When a carbon ion is incident upon the sample, it interacts with the atomic nuclei as well as the electrons of the target material. The elastic collision of the incident ion with nuclei displaces the atoms from their initial place and the displaced atoms further collide with the nearby atoms (cascade process) to produce the lattice defects in the form of vacancies, interstitials and their clusters. Moreover, during the ion-nuclei collisions, heat is produced due to the loss of ion energy during the collisions [56]. Similarly, for the ion-electronic collisions that are inelastic in nature, the energy of the incident carbon ion is lost in the form of heat due to a large number of excitations and ionizations produced in this process. By increasing the ion fluence, multiple number of collisions between incident ions with the target material take place that produce excessive heat energy. Consequently, a thermal spike is produced that raises the temperature of the localized region of the sample. The increase of Mg (002) peak after carbon ion irradiation of Mg-alloy AZ91D is attributed to the thermal energy gained by the Mg atoms and this favors the growth of Mg (002) plane. Moreover, the thermal energy deposition on the alloy surface is also responsible for the partial dissolution of intermetallic Mg17Al12 precipitates as corroborated by a decrease in their intensity after the carbon ion irradiation. However, the defects that are survived during the irradiation process produce widening of the diffraction peaks of both α-Mg and β-Mg phases that also causes a decrease in their intensity. In order to estimate the average crystallite size ‘D’ of α-Mg and β-Mg phases, the Scherer formula was used [59]:

\[ D = \frac{K\lambda}{\beta \cos \theta} \]

Here λ shows wavelength of x-rays, k represents shape factor (0.9), θ is Bragg angle and the β is peaks width at half maximum. The variation in the average crystallite size of α-Mg and β-Mg phases with carbon dose is shown in figure 2(b). As we see from this figure that the crystallite size of α-Mg phase decreases after carbon ions irradiation at \( 1 \times 10^{13} \) ions cm\(^{-2}\) and increases as ion dose increases. However, the crystallite size of Mg phase remains lower than that of the untreated sample. A similar trend of the change in crystallite size of the β-Mg phase is observed. The variations in crystallite size of β-Mg phase are lesser than that of the α-Mg phase. Overall, the crystallite size of the alloy decreases at lower dose (\( 1 \times 10^{13} \) ions cm\(^{-2}\)) and then marginally increases. The reduction in crystallite size is caused by radiation induced defects whereas the at higher dose the marginal increment in crystallite size occurs due to heat produced during the collisional cascades and electronic stopping process. This might recover some of the damage thus resulting in a small increase in the average crystallite size. The average microstrain ‘ε’ in the Mg alloy AZ91D in both the phases was evaluated by using the following relation [59]:

\[ \varepsilon = \frac{\beta \cos \theta}{4} \]

The microstrain variation in the α-Mg and β-Mg phases with carbon dose are shown in figure 2(c). As seen from the figure, the strain follows an opposite trend to that of the crystallite size. The structural analysis of Mg alloy AZ91D indicates that the both the thermal energy and the defects produced by the carbon ions control the structural properties of the alloy.

3.2. FTIR Spectroscopy results

Figure 3 shows Fourier Transform Infrared (FTIR) spectra of Mg–Al–Zn alloy before and after carbon ions irradiation. These spectra were collected in the transmittance mode within the wavelength range of 600 to 1800 cm\(^{-1}\). As seen from the figure, the spectrum of unirradiated sample does not contain any peak, whereas in the irradiated samples, carbon related peaks at 690 cm\(^{-1}\), 1028 cm\(^{-1}\), 1426 cm\(^{-1}\) and 1554 cm\(^{-1}\) are observed. The 690 cm\(^{-1}\) peak belongs to the characteristics of C–C stretching/Mg–C bond that becomes more prominent at higher carbon doses. The peaks at 1028 cm\(^{-1}\) and 1426 cm\(^{-1}\) correspond to C–O vibrations of carboxyl group and C–H stretching vibrations respectively. Similarly, the peak at 1554 cm\(^{-1}\) belongs to C=O carbonyl stretching vibrations. The generation of these peaks validate the presence of carbon inside the irradiated samples.
3.3. Electrochemical testing results

Electrochemical testing of untreated and carbon-ions irradiated samples was carried out in NaCl solution using Gammery potentiostat. Initially the open circuit potential was calculated and afterwards the potentiodynamic polarization technique was used to evaluate the corrosion rate as well as current density.

3.3.1. Open circuit potential results

The open circuit potential (OCP) results for untreated and carbon treated Mg alloy in 0.9% NaCl are given in figure 4. The untreated sample has more negative potential (active) relative to the carbon ion (C⁺) irradiated samples which shows its higher tendency towards the corrosion in 0.9% NaCl solution at 37 °C. The figure shows that the potential almost remains constant by irradiating Mg alloy with carbon ions up to the dose of 1 × 10¹⁴ ions cm⁻². However, above this dose, the OCP becomes less negative as compared to the unirradiated sample. The value of OCP before irradiation is found to be −1.627 V whereas after irradiation at 1 × 10¹³, 1 × 10¹⁴ and 1 × 10¹⁵ ions cm⁻², its value becomes −1.630, −1.631 and −1.594 V respectively. The less negative value of OCP after carbon ion irradiation at 1 × 10¹⁵ ions cm⁻² shows an improvement in the corrosion resistance of the Mg alloy. In unirradiated sample case, the higher tendency of hydrogen ions (H⁺) to initiate the reduction reaction (H⁺ + e⁻ → Hads) at the alloy surface, results in a relatively more negative reduction potential relative to the ion irradiated samples especially at 1 × 10¹⁵ ions cm⁻². Furthermore, the Cl⁻
ions present in the electrolyte also show more tendency to reach the surface and initiate pitting by disrupting the passive film at the surface of the sample [57, 58]. The results of OCP show that, up to doses $1 \times 10^{14}$ ions cm$^{-2}$, the insignificant change in the OCP value might be due to lesser resistance offered by the Mg/Al carbides that are formed during the irradiation process. However, by increasing the ion dose to $1 \times 10^{15}$ ions cm$^{-2}$, the amount of carbon inside the Mg alloy increases which results in the formation of more Mg/Al carbides. These carbides shift the potential towards less negative value than that of the untreated sample [57].

3.3.2. Potentiodynamic polarization results

The potentiodynamic polarization (PP) results for unirradiated and carbon-ions irradiated Mg–Al–Zn alloy AZ91D at $1 \times 10^{13}$ ions cm$^{-2}$, $1 \times 10^{14}$ ions cm$^{-2}$ and $1 \times 10^{15}$ ions cm$^{-2}$ are shown in figure 5.

The anodic polarization branch of the PP curves shows dissolution of samples in the solution whereas the cathodic polarization branch shows cathodic hydrogen evolution due to water reduction reaction. The anodic and cathodic reactions, represented by equations (3) and (4) respectively, are given as follows; [58].

$$\text{Mg} \rightarrow \text{Mg}^{2+} + 2e^- \quad (3)$$

$$2\text{H}_2\text{O} + 2e^- \rightarrow \text{H}_2 + 2\text{OH}^- \quad (4)$$

The upper part of the PP curves (in figure 5) shows the anodic polarization whereas the lower part represents the cathodic polarization. During the anodic polarization, the Mg alloy dissolves as depicted in the reaction given by equation (3). The increase in potential increases the current density. At a particular potential in the curves (that varies for each sample), the current density does not increase with further increase in the potential and it becomes almost constant. This suggests the barrier film formation on the sample’s surface. This can be demonstrated through the following reaction mechanism;

$$\text{Mg}^{2+} + 2\text{OH}^- \rightarrow \text{Mg(OH)}_2 \quad (5)$$

In the PP curves, the cathodic polarization curves represent the reduction reaction on the surface of unirradiated and ion irradiated samples as given by the equation (4). The figure 5 shows almost similar trend of the reduction curves in unirradiated and ion irradiated samples at $1 \times 10^{13}$ ions cm$^{-2}$, $1 \times 10^{14}$ ions cm$^{-2}$ and $1 \times 10^{15}$ ions cm$^{-2}$ during the cathodic polarization. This indicates that the carbon ions irradiation in Mg alloy up to $1 \times 10^{14}$ ions cm$^{-2}$ does not considerably affect the reduction reaction. When the ion dose increases to $1 \times 10^{15}$ ions cm$^{-2}$, the cathodic polarization curve also changes which results into the change in the polarization behavior of the sample.

The potentiodynamic polarization curves for unirradiated and ion-irradiated samples were Tafel fitted to obtain parameters such as corrosion rate, corrosion current density ($I_{\text{corr}}$), corrosion potential ($E_{\text{corr}}$) that are given in table 1. The corrosion current density ($I_{\text{corr}}$) and corrosion (C.R) rate of the Mg alloy are decreased after carbon ions irradiation. This decrease becomes more prominent at the ion dose of $1 \times 10^{15}$ ions cm$^{-2}$. The value of $I_{\text{corr}}$ for unirradiated sample is found to be 217 mA cm$^{-2}$ which reduces to 3.380 μA cm$^{-2}$ at $1 \times 10^{15}$ ions cm$^{-2}$. Similarly, the C.R. reduces from $181.6 \times 10^3$ mpy to $2.823$ mpy at the highest dose.

Further, the $E_{\text{corr}}$ value is also shifted towards less negative value $-900$ mV vs Ag/AgCl at $1 \times 10^{15}$ ions cm$^{-2}$ as...
compared to the unirradiated one. These results reveal that the higher dose of carbon ions (which is $1 \times 10^{15}$ ions cm$^{-2}$ in the present work) is more effective in increasing the corrosion resistance of the Mg alloy. At the dose $1 \times 10^{13}$ ions cm$^{-2}$, there are more chances of the formation of metal (Mg/Al) carbides due to increased number of carbon ions inside the sample’s surface [57]. This might be responsible for the improvement in the corrosion resistance of the Mg-alloy AZ91D. These carbides offer resistance against the current flow from the alloy surface thus reducing the samples oxidation and hence the corrosion rate.

3.4. Surface analysis of electrochemical tested surfaces

The scanning electron microscope (SEM) images of electrochemically tested surfaces of unirradiated and carbon ion irradiated samples are shown in figure 6. As one can observe that in the unirradiated sample case, the surface contains deeper pits and cracks that are generated in the NaCl solution due to corrosion of the sample. After carbon ions irradiation, the surface of the Mg alloy shows lesser corrosion damage as compared to the unirradiated sample. The pits density and depth decrease as a result of ion irradiation of the Mg alloy. At $1 \times 10^{13}$ ions cm$^{-2}$, the pits are significantly reduced that is an indication of a reduction in the sample’s corrosion rate. These results are consistent with the potentiodynamic polarization results as described above. The improvement in the corrosion resistance of the sample after carbon ion irradiation can be related to the formation of metal carbides inside the alloy that resist the electron flow and the oxidation from the samples’ surface, consequently, the corrosion rate decreases and hence the corrosion resistance is improved.

| Specimen        | $I_{corr}$ (A/cm$^2$) | $E_{corr}$ (mV) | CR (mpy)  |
|-----------------|-----------------------|-----------------|--------|
| Un-implanted    | $217.0 \times 10^{-3}$ | $-1520$         | $181.6 \times 10^{-3}$ |
| $1 \times 10^{13}$ | $1.360 \times 10^{-3}$ | $-2530$         | $1.137 \times 10^{-3}$ |
| $1 \times 10^{14}$ | $24.60 \times 10^{-6}$ | $-7700$         | $20.52$ |
| $1 \times 10^{15}$ | $3.380 \times 10^{-6}$ | $-900.0$        | $2.823$ |

**Figure 6.** SEM results of corroded Mg–Al–Zn alloy surface before and after carbon ions irradiation.
4. Conclusions

The conclusions can be from this work are;

1. Carbon irradiation of Mg–Al–Zn alloy (AZ91D) enhances the growth of hexagonal Mg along (002) plane.
2. The crystallinity of Mg–Al–Zn alloy decreases at lower carbon dose ($1 \times 10^{13}$ ions cm$^{-2}$), whereas at higher dose, the crystallinity improves.
3. The open circuit potential does not change at lower ion doses up to $1 \times 10^{14}$ ions cm$^{-2}$, whereas it becomes less negative with further increase of the carbon dose.
4. The electrochemical corrosion rate/current density of Mg–Al–Zn alloy decrease after carbon ions irradiation particularly at $1 \times 10^{15}$ ions cm$^{-2}$.
5. The pitting on unirradiated Mg alloy surface is reduced after carbon irradiation particularly at $1 \times 10^{15}$ ions cm$^{-2}$.

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