

Article

Corrosion Behavior and Mechanism of Carbon Ion-Implanted Magnesium Alloy

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Abstract: Carbon ion implantation was conducted on an AM60 magnesium alloy with fluences between 1×10^{16} and 6×10^{16} ions/cm² and an energy of 35 keV. The microstructure and electrochemical properties of the samples were systematically characterized by X-ray photoelectron spectroscopy, X-ray diffraction, Raman scattering, scanning electron microscopy, transmission electron microscopy, and electrochemical methods. These studies reveal that a 250 nm-thick C-rich layer is formed on the surface and the Mg₂C₃ phase embeds in the ion-implanted region. The crystal structure of the Mg₂C₃ was constructed, and an electronic density map was calculated by density-functional theory calculation. The large peak in the density of states (DOS) shows two atomic p orbitals for Mg₂C₃. The main electron energy is concentrated between −50 and −40 eV, and the electron energy mainly comes from Mg (*p*) and Mg (*s*). The electrochemical experiments reveal that the E_{corr} is −1.35 V and I_{corr} is 20.1 μA/cm² for the sample implanted with the optimal fluence of 6×10^{16} ions/cm². The sample from C ion implantation gives rise to better corrosion resistance.

Keywords: magnesium alloy; carbon nanolayer coating; plasma ion implantation; first-principle calculation; corrosion resistance

1. Introduction

Magnesium alloys have some obvious advantages such as light weight and recyclability, and they are more and more widely used [1–5]. However, the corrosion resistance of magnesium alloys is not good enough [6,7]. To date, many methods have been carried out to improve the corrosion properties of magnesium alloys such as microarc oxidation [8,9], laser cladding [10,11], and ion implantation [12–16]. Ion implantation causes the substrate and coating have a good binding force compared to other methods. Many ions can be used in the ion implantation. For example, Ti [17,18], Al [19,20], Zr [21,22], and Cr [23,24] ion implantation has been conducted on Mg alloys to improve the corrosion properties. Compared with that of an untreated sample, the corrosion potential of a Ti/N-treated coupon increases by over 600 mV, the corrosion current density decreases by over two orders of magnitude, and the polarization resistance increases by 66.4 times [17]. The Al ion-implanted AZ31 magnesium alloy with an ion implantation dose of 6×10^{16} ions/cm² achieved a high pitting breakdown potential of about −480 mV (saturated calomel electrode (SCE)) [20]. The Zr ion implantation sample shows a smaller I_{corr} due to the formation of ZrO₂ in the implanted layer [21].

In addition to metals, some nonmetals are also used for ion implantation. A Mg-Nd-Zn-Zr alloy was modified with C_2H_2 gas, and a diamond-like carbon film (DLC) was synthesized. Most of the surface region was protected in the aggressive NaCl solution [25]. The corrosion resistance of the AZ31 Mg alloy formed by N ion implantation was studied [26,27]. The results indicate that a Mg_3N_2 film is formed. The corrosion potential shift was 100 mV. Besides the corrosion resistance, the cytotoxicity properties of Mg alloys modified by ion implantation have also been investigated [28–30].

The corrosion performance of the magnesium alloys can be improved to a certain extent by ion implantation. The C element was also implanted into pure magnesium, and the electrochemical properties were studied by Xu [31], but the microstructure of a C layer is not well known. As a very important element, C has many physical and chemical properties, such as good electrical conductivity and tribological properties. Many new carbon-based materials are being developed, for instance, nanoporous carbon [32] and carbon layers [33].

The application of AM60 materials is limited by poor corrosion properties. The effects of C ion implantation on the AM60 magnesium alloy and related mechanism have not been reported previously. In this work, the AM60 magnesium alloy was implanted with C with different fluences, and the structural and electrochemical properties were studied in detail. The atomic structure is discussed based on both experimental and theoretical results.

2. Experimental and Theoretical Calculations

Commercially available AM60 magnesium alloy samples were cut into $15\text{ mm} \times 15\text{ mm} \times 2\text{ mm}$ pieces and used as substrates in the experiment. Before ion implantation, the samples were finely ground with SiC paper of 400, 800, 1200, 1500, and 2000 grits, sequentially, followed by polishing with fine diamond paste (average grain size, $0.5\text{ }\mu\text{m}$) to a final roughness of approximately $0.08 \pm 0.02\text{ }\mu\text{m}$, and then they were cleaned in acetone by ultrasonication. Carbon ion implantation was performed on the PI-80A plasma ion implanter in the Plasma Laboratory of City University of Hong Kong, Hong Kong, China. During implantation, the base pressure in the vacuum chamber was $2 \times 10^{-3}\text{ Pa}$ and the acceleration voltage was 35 keV. The ion implantation fluences are shown in Table 1.

Table 1. Carbon ion implantation fluences.

Samples	BM	C1	C2	C3
Fluence (ions/cm ²)	0	1×10^{16}	3×10^{16}	6×10^{16}

X-ray photoelectron spectroscopy (XPS, PHI-5000 versaprobe, Kanagawa, Japan) was carried with Al $K\alpha$ irradiation at a sputtering rate of about 10 nm/min in order to analyze the depth of the implanted elements. The Raman scattering spectra were acquired on the HR LabRAM using a 514.5 nm argon laser, and the structure was confirmed by X-ray diffraction on the D2 Phaser X-ray diffractometer (XRD) (BRUKER, Karlsruhe, Germany) with Cu $K\alpha$ radiation. The cross-sectional microstructure of the samples was observed under a transmission electron microscope (TEM, JEM-2100, Tokyo, Japan). A TEM sample with a thickness of 3 mm was cut from the center of the ion implantation surface, with a plane size of $3\text{ mm} \times 0.5\text{ mm}$. Then, the 0.5 mm particles were further ground and thinned. The hole was thinned near the ion implantation surface, and the observation surface was perpendicular to the ion-implanted surface.

The corrosion tests were performed on an electrochemical workstation (CHI660E, Shanghai, China). A three-electrode configuration with a platinum sheet as the counter electrode, Mg sample as the working electrode (0.785 cm^2 exposed area), and saturated calomel electrode (SCE) as the reference electrode was adopted. The volume of the electrolyte was 1000 mL, the electrolyte was 3.5% NaCl solution, the temperature was $23 \pm 1\text{ }^\circ\text{C}$, and the experiment was conducted three times to improve the statistics. The polarization curves were acquired at a scanning rate of 5.0 mV/s from -500 to 400 mV with respect to the open circuit potential. The samples soaked in 3.5% NaCl for 24 h were observed by scanning electron microscopy (SEM, ZEISS SIGMA500, Dresden, Germany).

In order to calculate the electronic structure, the crystal structure of Mg_2C_3 was constructed by a first-principle calculation with the CASTEP module in the Materials Studio software [34]. The unit cell constant of Mg_2C_3 and each atomic lattice were completely relaxed until the unit cell energy converged to a fixed value before the calculation. The result of the calculation ignores the rotation effect.

3. Results and Discussion

Figure 1 displays the XPS depth profile of the ion-implanted AM60 alloy of Sample C3. A carbon-rich layer is formed after C ion implantation with a depth of about 250 nm. The high-resolution XPS spectra of C, O, Al, and Mg acquired after sputtering for different times are displayed in Figure 2. As the sputtering time increases, the C peak intensity decreases (Figure 2a) and the Mg intensity increases gradually (Figure 2d). The Al intensity also increases (Figure 2c), and O is observed at the sputtering times of 0 and 15 min (Figure 2b). The C binding energy is 284.5 eV (Figure 2a), suggesting a sp^2 hybridization composed of three σ bonds and one π bond perpendicular to the σ bond. This observation is consistent with previous results, and it is generally believed that sp^2 hybridization is formed by low-energy ion implantation [35,36].

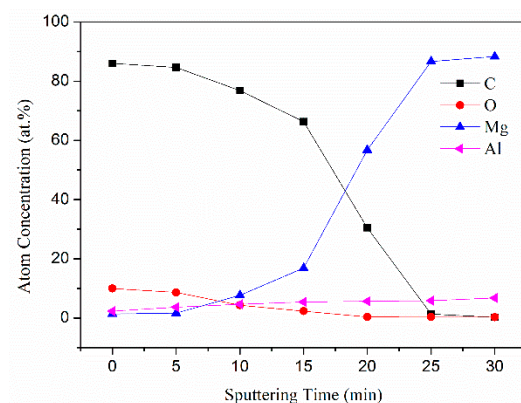


Figure 1. XPS element depth profile of the AM60 alloy after C ion implantation at 6×10^6 ions/cm² with a sputtering rate of 10 nm/min.

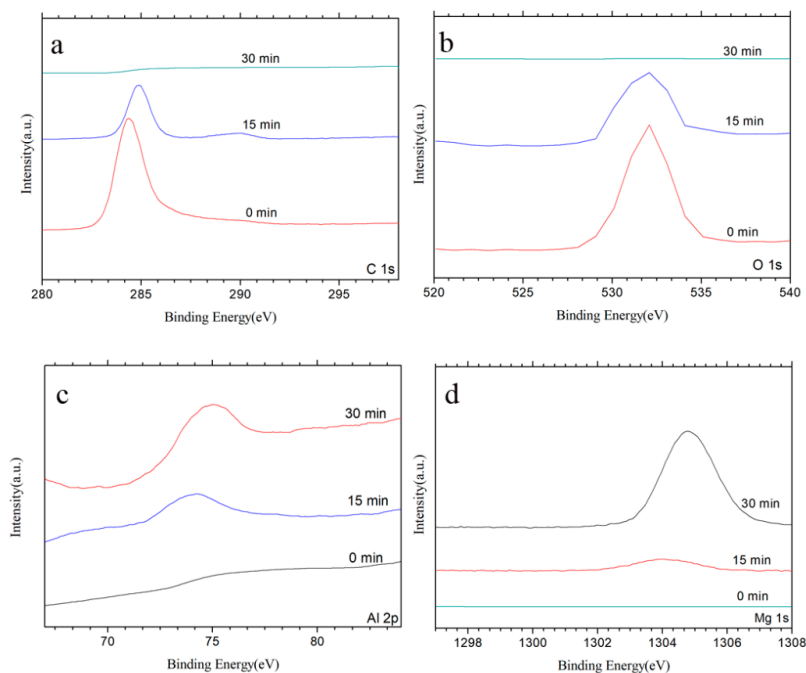


Figure 2. High-resolution XPS spectra of the treated AM60 alloy at 6×10^6 ions/cm² after sputtering for different times: (a) C 1s, (b) O 1s, (c) Al 2p, and (d) Mg 1s.

Figure 3 shows the Raman scattering spectra. Before ion implantation, there is no C peak, but after C ion implantation, a G peak at 1542.14 cm^{-1} and D peak at 1331.66 cm^{-1} arising from amorphous carbon with sp^2 and sp^3 hybridization appear, being consistent with the XPS data. Figure 4 shows the XRD patterns, and α -Mg and $\text{Mg}_{17}\text{Al}_{12}$ are present. A weak Mg_2C_3 phase was formed after ion implantation, especially for high fluences such as for Samples C2 and C3. Figure 5 shows the TEM images of Sample C3. Figure 5a shows that the carbon-implanted sample is about 250 nm thick, consistent with the results of XPS. The white line shows the interface between the C layer and substrate. The black objects are the compound formed during ion implantation. A typical compound is revealed in the near surface as shown in Figure 5b. Figure 5c is a higher magnification of part of the white circle area in Figure 5b. The distance of the lattice space of the compound is 0.37 nm. According to the EDS results, the percent of Mg is 34.6%, the percent of C is 61.3%, and the percent of O is 4.1%. The compound is mainly composed of Mg and C. The diffraction patterns of the compound are displayed in Figure 5d. The results of TEM and XRD suggest that the compound is Mg_2C_3 .

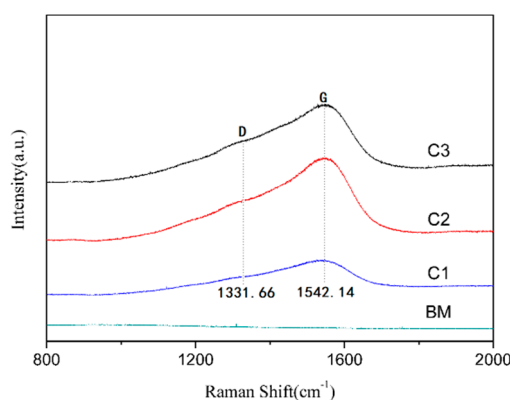


Figure 3. Raman scattering spectra of the carbon films.

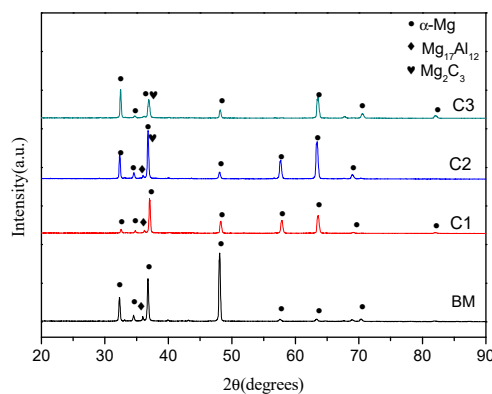


Figure 4. XRD patterns of different samples.

According to Find It soft, Mg_2C_3 has a complex cubic structure. The Mg_2C_3 space group is Pnnm (parameter code 58), and $a = 6.4108\text{ Å}$, $b = 5.2786\text{ Å}$, and $c = 3.7283\text{ Å}$, as shown in Table 2. Based on the data, a unit cell model and the electron density map are shown in Figure 6a,b, respectively. Figure 6c shows the density of states (DOS) of the Mg_2C_3 unit cell, and the large peak shows two kinds of atomic p orbitals. The main electron energy is concentrated between -50 and -40 eV , and the electron energy mainly comes from Mg (p) and Mg (s). In addition, the valence electron of C (s) and C (p) makes a small contribution. The structure is primarily responsible for the improved corrosion resistance, to be discussed later.

Table 2. Lattice parameters of Mg_2C_3 .

Crystal	Space Group	Lattice Parameters			Elements	Atomic Coordinates		
		<i>a</i>	<i>b</i>	<i>c</i>		<i>X</i>	<i>Y</i>	<i>Z</i>
Mg_2C_3	58	6.4108	5.2786	3.7283	Mg(I)	0.2903	0.3901	0
					C(I)	0.5	0	0
					C(II)	0.6188	0.2070	0

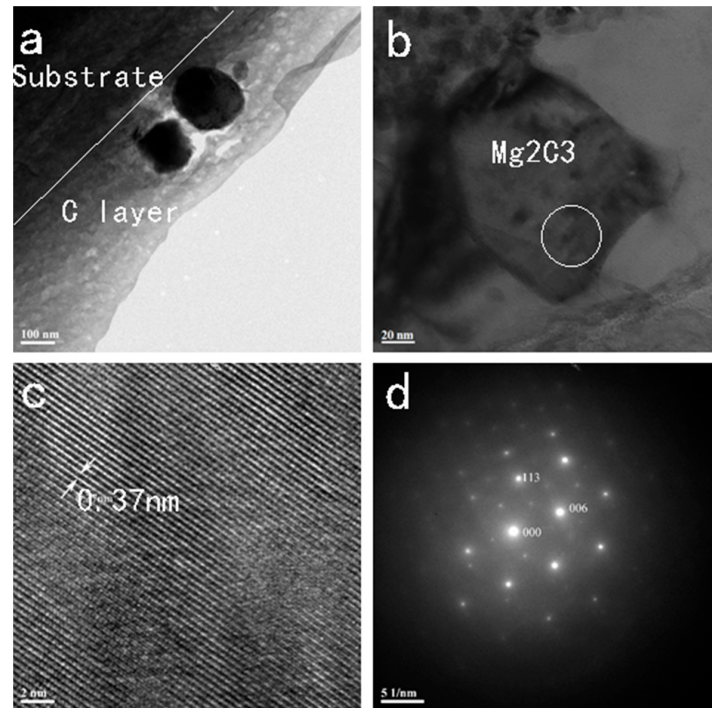
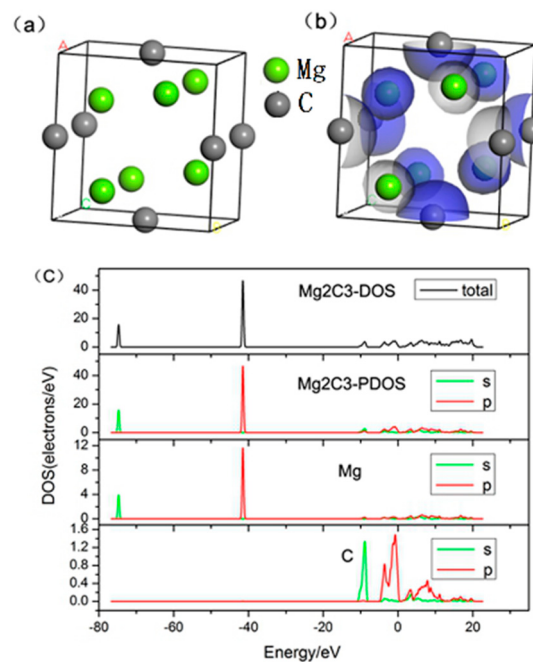
**Figure 5.** Cross-sectional TEM images of Sample C3: (a) surface microstructure, (b) compound microstructure, (c) high resolution TEM images, and (d) diffraction patterns of the compound.**Figure 6.** First-principle study of Mg_2C_3 : (a) unit cell model, (b) electron density map, and (c) state density maps.

Figure 7 presents the potentiodynamic polarization curves of the different samples in the 3.5% NaCl solution. The corrosion potential (E_{corr}) and corrosion current density (I_{corr}) are determined by Tafel extrapolation. If a tangent is made to the cathodic polarization curve and the anodic polarization curve, the ordinate of the intersection of the two lines and the E_{corr} potential line is the I_{corr} . Compared to that of the un-implanted AM60 control sample, the E_{corr} of Sample C3 shifts from -1.48 to -1.35 V, indicative of better corrosion resistance in the NaCl solution. The I_{corr} of sample C3 is $20.1 \mu\text{A}/\text{cm}^2$, which is much lower than that of the AM60 control sample ($50.6 \mu\text{A}/\text{cm}^2$). The more positive E_{corr} and smaller I_{corr} demonstrate that C ion implantation improves the corrosion resistance of the Mg alloy by forming the modified structure as described previously [37,38].

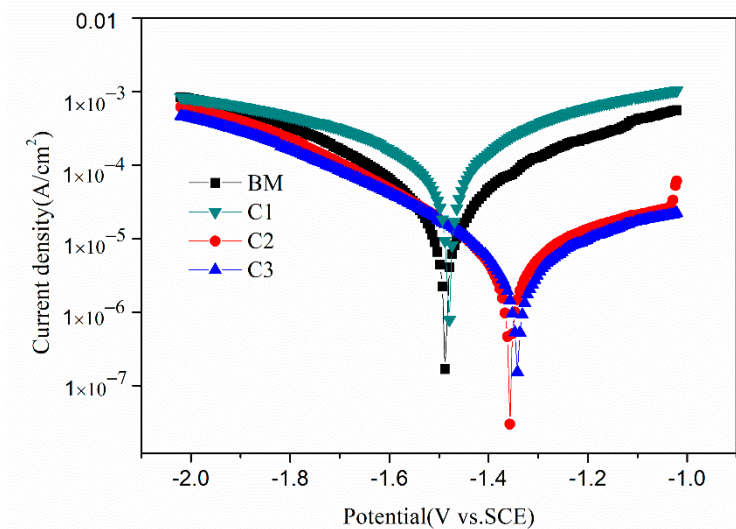
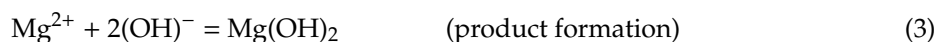
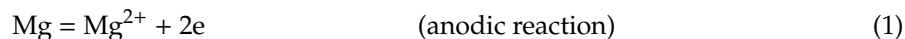


Figure 7. Polarization curves of different samples.

The morphology of the different samples after immersion in 3.5% NaCl is shown in Figure 8. Lots of corrosion defects are observed from the surfaces of the control samples (Figure 8a-2). The SEM images were obtained from the marked part in the black box of the digital picture. It can be seen that the corrosion area of sample BM is larger than that of the other samples. The EDS performed on Point 1 as shown in Figure 8d-2 indicates that there are Mg, O, and C elements on the surface after C ion implantation.

Figure 9 is the schematic presentation of the corrosion of the different samples. The inner layer is composed of AM60 alloy, and a thin layer of MgO is attached to the outer layer. The MgO layer on the surface is hydrated during exposure to an aqueous environment, which converts the MgO into a larger stable $\text{Mg}(\text{OH})_2$ layer [24], and H_2 is produced in the corrosion process as shown in Figure 9a. The reaction may be expressed as the sum of the following reactions:



The anodic reaction (Equation (1)) probably involves intermediate steps, which may produce the monovalent magnesium ion (Mg^+), which has a short lifetime [39]. The reduction process for hydrogen ions and the hydrogen overvoltage of the cathodic phase play an important role in the corrosion of magnesium [40]. After C ion implantation, there is a C layer on the surface. The sample from C ion implantation gives rise to better corrosion resistance. Mg_2C_3 scattered in the C layer and C layer can decrease the corrosion and prevent corrosion from entering the substrate as shown in Figure 9b.

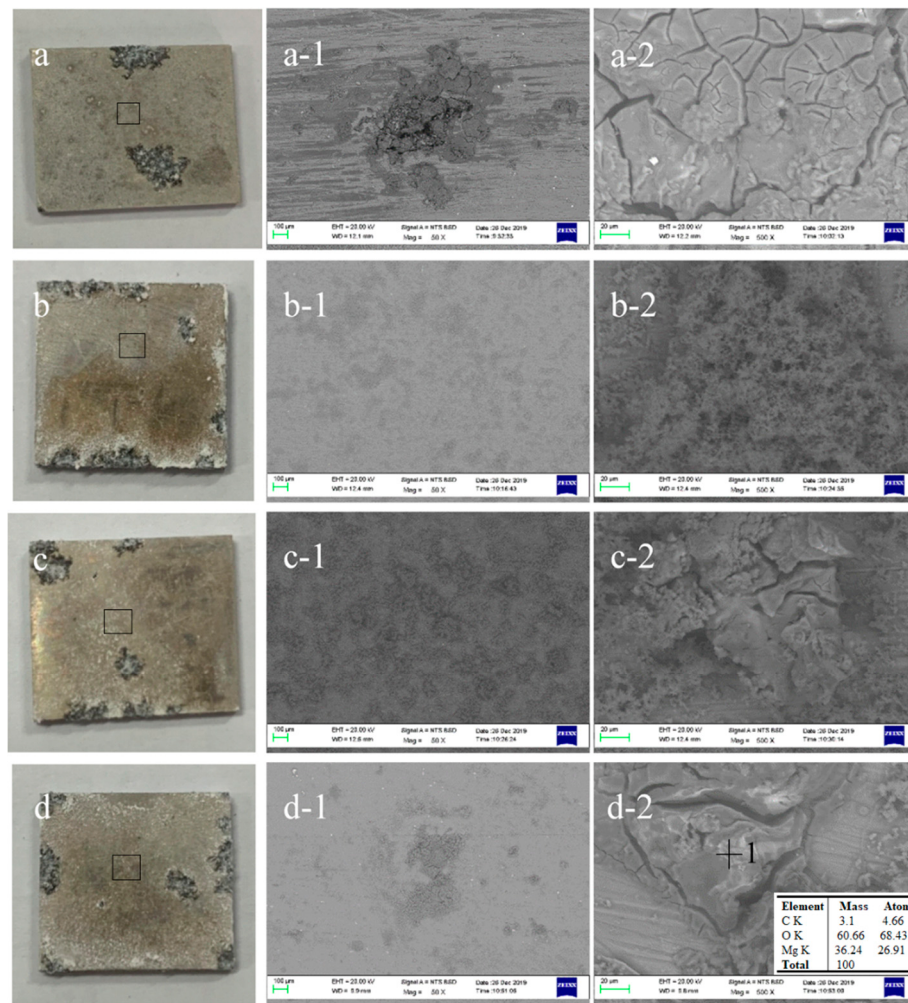


Figure 8. Morphology of the corroded samples: (a) BM, (b) C1, (c) C2, and (d) C3; (i-1) low-magnification SEM pictures, and (i-2) high-magnification SEM pictures. EDS results acquired from Point 1 ($i = a, b, c$, and d).

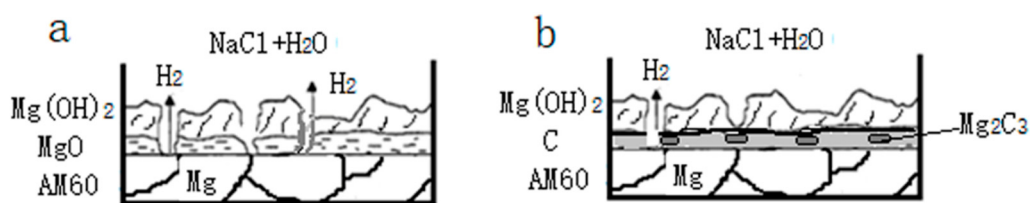


Figure 9. A schematic presentation of the corrosion of different samples: (a) corrosion of AM60 Mg alloy, and (b) corrosion of AM60 with C nanolayer.

4. Conclusions

- (1) A 250 nm-thick C-rich coating is formed on the surface of AM60 after C implantation with an ion implantation dose of 6×10^{16} ions/cm², and a Mg₂C₃ phase is observed from the ion-implanted region.
- (2) The large peak in the density of states (DOS) shows the two atomic p orbitals of Mg₂C₃. The main electron energy is concentrated between −50 and −40 eV, and the electron energy mainly comes from Mg (*p*) and Mg (*s*).
- (3) The more positive E_{corr} and smaller I_{corr} demonstrate that C ion implantation gives rise to better corrosion resistance in AM60 magnesium alloys.

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