Research Article

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An investigation on the degradation behaviors of Mg wires/PLA composite for bone fixation implants: influence of wire content and load mode

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Abstract: Poly-lactic acid based biocomposite strengthened with magnesium alloy wires (Mg wires/PLA composite) is prepared for bone fixation implantation. The influence of wire content and load mode on the degradation performances of the composite and its components is studied. The result suggests the degradation of Mg wires could slow down the pH decrease originated from the degradation of PLA, while a relatively high wire content contributes to descend the degradation rate of Mg wire in the composite. Dynamic load significantly promotes the mechanical loss of the specimens. After 30 days immersion, the S_b retention is about 65%, 52% and 55%, respectively for pure PLA, the composite at 10 vol% and 20 vol% under dynamic load, comparing to 75%, 70% and 72% under no load. Moreover, dynamic load could further mitigate the degradation of Mg wires by increasing convective transport of acidic products out of the composite.

Keywords: biodegradable; biomaterials; composites; degradation behaviors

1 Introduction

Biodegradable polymers, including poly-lactic acid (PLA) and poly-glycolic acid (PGA), are considered as one of the important and applicable biomaterials used for implants [1].

The main hurdles of biodegradable polymers in service for bone fixation implants are the lack of bioactivity, inflammation induced by a significant pH decrease and the relatively low mechanical properties [2]. For instance, the acidic degradation products of PLA are noticed to induce a mild inflammation response, resulting in a continuous fibrous capsule with a dense population of fibroblast-like cells around the PLLA screw implant [3]. Many studies try to overcome these drawbacks by adding components such as HA [4] or bioglass [5] into the polymeric matrix. Very recently, the biodegradable polymer based composite reinforced with magnesium additives attracts huge interests [6–9]. It is reported the mechanical properties and the acidic degradation of polymers could be significantly improved with the corporation of Mg additives. Moreover, the presence of Mg could profoundly promote the fracture healing by enhancing mineralization and osteogenesis [10]. Besides those, the Mg additives would also benefit from the protection of surround polymer matrix to avoid rapid degradation [7] and lactate could further elicit ER-Mitochondrial Mg^{2+} dynamics to integrate cellular metabolism [11]. The composite shows potential applications for the implants.

In service, the implants are desired to have tailored degradation rate to match the healing process of the fracture tissues. Therefore, the degradation performances, specially the degradation behaviors of the composite are of importance. Apparently, the degradation performances of the composite strongly depend on the degradation of its components, involving the hydrolysis of the polymeric matrix and the biodegradation of Mg additives. It is well known that the hydrolysis rate of the polymeric matrix is strongly related to pH values, where acidic or alkaline environment could both accelerate the hydrolysis [12]. Since the degradation of Mg would release excess OH^- ions to raise the pH values, its content would be of importance for the degradation of the composite. Meanwhile, as the main supports of the composite, the degradation behaviors of Mg additives directly influence the mechanical property retention of the composite. Therefore, the degradation behaviors of each

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Figure 1: Schematic illustration of the apparatus used in the test and the right-upper picture is the image of the specimen.

component in the composite should be taken into account for further applications.

Moreover, it should be mentioned that the implant would suffer different physiologically mechanical loads *in vivo* which would complicate the degradation process of the composite. Commonly, it is reported that these loads would play an important role on tissue healing [13–15]. For example, dynamic loading is reported to profoundly promote bone fracture repair by enhancing the bone formation [16]. Numerous investigations show the load would obviously affect the degradation of pure biodegradable polymers [17, 18] or Mg alloys [19, 20]. However, the relative studies on the composite, specially the degradation behaviors of its components are scarce. In this work, magnesium alloy wires are conducted to unidirectionally strengthen PLA matrix (Mg wires/PLA). The effects of wire content and load mode on the degradation behaviors of the composite and its components are studied.

2 Materials and methods

2.1 Materials

Commercial PLA particles were purchased from Natureworks LLC. The diameter of AZ31B magnesium alloy wire is 0.3 mm. The wires were self-fabricated by cold-drawing process. WHD-30 type micro arc oxidation (MAO) equipment was conducted on the Mg wires to improve the interface adhesion between the wires and the PLA matrix [21]. The composite with a volume fraction of Mg wires at 10 vol% and 20 vol% were prepared by a laminating method described in our previous work [22], while pure-PLA was prepared under the same condition.

2.2 Immersion tests

The immersion tests of the specimen were conducted under different load modes, including no load, static compression load at 1 MPa (static load), and dynamic compression load at 1 MPa and 1 Hz (dynamic load). The dimension of the specimen for the tests was 50 mm×12 mm×2 mm. Kirkland's bio-corrosion media (KBM) [23] with a pH 7.4 and temperature 37°C was applied as the immersion solution. The ratio of solution's volume to sample's surface area was larger than 50 ml/cm². During the immersion, the solution was changed every day and the pH value was recorded before each changing. The home-made apparatus for applying the loading was shown in Figure 1. The switch-on clock and the pressure regulating valve was used to control the loading frequency and magnitude, respectively. The immersion durations were predetermined to be 7 days, 14 days, 21 days and 30 days. At least three specimens were performed for each parameter and the averages were presented in the assessment.

2.3 Mechanical and thermal tests

Tensile tests were performed to determine the tensile strength (S_t) and elastic modulus (E) at the longitudinal direction. Three-point bending tests based on the standard ASTM D790-2010 were conducted to determine the bending strength (S_b) of the specimens along the transverse direction. The tests were performed by using a CMT4503 electronic universal testing machine at room temperature. During the bending test, the crosshead speed is 2 mm/min with a bending span of 32 mm. The S_b retention was obtained by dividing the initial S_b with the S_b of the immersed specimen.

The thermal behaviors of PLA matrix were determined on a differential scanning calorimeter equipment

(DSC404F3). The test was performed under a nitrogen atmosphere, and the heating rate is 10°C/min from 30°C to 210°C. The relevant thermal transitions respectively reflect the glass transition (T_g), cold crystallization (T_{cc}), and melting (T_m). After the test, T_g was got by using the midpoint of the transformation, while the crystallinity X_c of PLA matrix was measured according to the following equation:

$$X_c = (\Delta H_m - \Delta H_{cc})/\Delta H_0; \quad (1)$$

where ΔH_m reflects the enthalpy of melting, ΔH_{cc} reflects the enthalpy of cold crystallization, and ΔH_0 is the enthalpy of 100% crystalline PLLA (about 93.1 J/g) [7].

2.4 Electrochemical tests

Electrochemical impedance spectroscopy (EIS) was performed on a CHI604 potentiostat workstation to investigate the degradation performance of Mg wires in the composite. A classical three-electrode system was used, where a Mg wire at the middle of the composite acts as the working electrode, a platinum plate is used as the counter electrode, and a saturated calomel electrode (SCE) is conducted as the reference electrode, as shown in Figure 1. The EIS was measured from 100,000 to 0.1 Hz with 10 mV perturbation.

2.5 Microstructural characterization

After the immersion, the composite is dissolved in chloroform to collect the Mg wires in the composite. Scanning electron microscopy (SEM, the Philips XL30 FEG) was performed to investigate the surface morphologies of the wires during the degradation.

XR-CT analysis using a CT system (Y.CT PRECISION, voltage 195 kV, current 0.28 mA) was conducted to represent the morphologies of the composite. The internal defects

were calculated by using VGStudio Max 3.0 analysis software. Detailed analysis procedure is described in another work [9].

3 Results

3.1 Evolution of degradation properties for the composite

The pH evolution in the solutions of pure PLA and the composite during immersion are represented in Figure 2. pH changes slightly under no load, but decreases significantly under the loading conditions for both pure-PLA and the composite. In the case of the composite, an apparent pH decrease is noticed under dynamic load during the first 10 days, as the arrow shown in Figure 2(b) and 2(c). For the composite at 20 vol%, the pH drops to about 5.8 after 10 days immersion under dynamic load while it is about 7.4 and 6.5 respectively under no load and static load. Thereafter, pH values increase gradually, indicating the mitigation on pH decrease by the degradation of Mg wires.

The initial mechanical properties of pure PLA and the composite are shown in Table 1. It could be noticed that increasing the Mg wire content could significantly enhance the mechanical properties of the composite. Figure 3 shows

Table 1: Initial mechanical properties of pure PLA and the composite.

	S_t (MPa)	E (GPa)	S_b (MPa)
Pure PLA	45(±4.66)	4.0(±0.37)	76(±2.52)
The composite at 10 vol%	67(±2.32)	6.2(±1.61)	119(±2.61)
The composite at 20 vol%	83(±4.51)	8.1(±1.32)	159(±4.01)

Figure 2: pH changes in the fluid for the different specimens: (a) pure PLA; (b) the composite at 10 vol%; (c) the composite at 20 vol%.

Figure 3: S_b retention changes of the different specimens: (a) pure PLA; (b) the composite at 10 vol%; (c) the composite at 20 vol%.

the changes of S_b retention for pure PLA and the composite during the immersion. The initial S_b of pure PLA is about 75 MPa, while it increases to about 119 MPa and 159 MPa, respectively at 10 vol% and 20 vol% for the composite. Mg wires could apparently raise the mechanical properties of PLA. After immersion for 30 days, the S_b of pure PLA reduces to about 59 MPa, 53 MPa and 49 MPa, respectively under no load, static and dynamic load. Similarly, dynamic load would also promote the S_b reduction of the composite. As the immersion proceeds to 30 days, the S_b retention is about 65%, 52% and 55%, respectively for pure PLA, the composite at 10 vol% and 20 vol% under dynamic load, comparing to 75%, 70% and 72% under no load. Moreover, it could be noticed that the strength of composite decreased faster than that of PLA at the same condition, though the

former still represents the larger S_b values than that of the later after 30 days immersion.

3.2 Evolution of PLA matrix performances in the composite

DSC curves of PLA matrix for pure PLA and the composite are shown in Figure 4. The curves represent firstly a glass transition. At about 100°C, an exothermic phase transition appears, which is induced by cold crystallization. The final peak (at about 170°C) is an endothermic transition originated from the polymer melting. The T_m and X_c of PLA matrix are depicted in Table 2. As the immersion proceeds, the X_c increases significantly, but the T_m changes slightly after immersion. Moreover, it seems the addition of Mg wires and

Figure 4: DSC curves of pure PLA and PLA matrix in the composite at initial and immersion for 30 days under different load modes: (a) pure PLA; (b) PLA matrix in the composite at 10 vol%; (c) PLA matrix in the composite at 20 vol%.

Table 2: The thermal properties of pure PLA and the PLA matrix in the composite under different load modes.

	Pure PLA		PLA matrix at 10 vol%		PLA matrix at 20 vol%	
	T_m (°C)	X_c (%)	T_m (°C)	X_c (%)	T_m (°C)	X_c (%)
initial	169.87	34	169.84	33	169.56	30
no load (30 days)	168.82	43	169.37	42	168.41	43
static load (30 days)	170.06	43	166.88	39	167.33	44
dynamic load (30 days)	169.00	45	171.01	40	169.32	41

the external loads have slight influence on the crystalline fraction of PLA matrix.

3.3 Evolution of Mg wires performances in the composite

Figure 5(a) and 5(b) depicts the Nyquist plots of the wires in the composite under different load modes after 14 days immersion. The spectra are fitted with the equivalent circuit model in right-upper image of Figure 5(a) and 5(b). A constant phase element (CPE) instead of an ideal capacitor is conducted. The CPE represents the surface inhomogeneity [24] and R_{sol} means the resistance of the solution. As the immersion proceeds, the changing of polarization resistance (R_p) of the wire in the composite is shown in Figure 5(c) and 5(d). After 1 day, the R_p is about $6000 \Omega \text{ cm}^2$. As the immersion proceeds to 7 days, the R_p raises and the

largest R_p appears under the dynamic load. Thereafter, R_p at no load increases initially and then descends gradually. As a comparison, R_p decreases continually under the static load. For the composite under dynamic load, R_p represents a dramatically increase after 30 days immersion. Moreover, the R_p at 20 vol% seems to have a larger value than that at 10 vol% after 30 days immersion under different load modes.

Figure 6(a-f) depicts the surface morphologies of the wires in the composite at 10 vol% by dissolving the PLA matrix in chloroform. The surface coating is almost integral while only micro cracks could be observed after 7 days immersion. However, as the immersion time increases to 30 days, the coating exhibits more critical breakage under static and dynamic load comparing to that under no load, indicating the external load would enhance the breakage of the coating. Figure 6(g-l) depicts the surface morphologies of the wires in the composite at 20 vol%. Similarly, dynamic

Figure 5: Electrochemical behavior of the wire inside the composite during immersion: (a) Nyquist plots and the fitting curves of the wire inside the composite at 10 vol% after immersion for 14 days under different load modes and the right-upper image is the applied equivalent circuit model; (b) Nyquist plots and the fitting curves of the wire inside the composite at 20 vol% after immersion for 14 days under different load modes and the right-upper image is the applied equivalent circuit model; (c) R_p evolution of the wire inside the composite at 10 vol% versus immersion time; (d) R_p evolution of the wire inside the composite at 20 vol% versus immersion time.

Figure 6: Surface morphologies of Mg wires in the composite: (a) at 10 vol% under no load after 7 days immersion; (b) at 10 vol% under static load after 7 days immersion; (c) at 10 vol% under dynamic load after 7 days immersion; (d) at 10 vol% under no load after 30 days immersion; (e) at 10 vol% under static load after 30 days immersion; (f) at 10 vol% under dynamic load after 30 days immersion; (g) at 20 vol% under no load after 7 days immersion; (h) at 20 vol% under static load after 7 days immersion; (i) at 20 vol% under dynamic load after 7 days immersion; (j) at 20 vol% under no load after 30 days immersion; (k) at 20 vol% under static load after 30 days immersion; (l) at 20 vol% under dynamic load after 30 days immersion.

load could promote the coating breakage. However, it seems that the coating breakage of the wire at 20 vol% is slighter than that at 10 vol%, indicating a high wire content would mitigate the breakage of the coating, meeting well with the R_p values.

Figure 7(a) and 7(c) shows the defects inside the composite at 20 vol% under after immersion for 21 days and 30 days under no load. The largest defect volume after 21 days immersion is about 0.37 m^3 , while it increases to 0.41 m^3

after 30 days immersion. Additionally, it could be noticed that most of the defects mainly appear along the interface between the Mg wires and the matrix, as seen in Figure 7(b) and 7(d), denoting the important role of the interface for the diffusion of the small molecules and the weakening of the interface as the immersion proceeds. Moreover, the largest defects seem to emerge randomly at the end side of the composite or the part near the surface.

Figure 7: The distribution of the defects in the composite at 20 vol% under no load: (a) the profile without the wires after 21 days immersion; (b) the profile including the wires after 21 days immersion; (c) the profile without the wires after 30 days immersion; (d) the profile including the wires after 30 days immersion.

4 Discussions

4.1 Effect of Mg wire content

It is widely accepted that the hydrolysis of PLA is strongly related to the pH values of the surround environment. In this work, it could be observed that the pH values of the solution drop significantly at a high Mg wire content under loading conditions during the initial immersion. This is probably attributed to the large interface area between the wires and the matrix at a high Mg wire content, which would be one of the main diffusion pathways for the small molecules. However, as the immersion proceeds, the pH values would gradually increase at a high Mg wire content, suggesting the degradation of Mg wires could mitigate the pH decrease originated from the degradation of PLA.

According to this study, it seems that Mg wires in the composite at 20 vol% experiences lower degradation rate than that at 10 vol% after 30 days immersion, which may contribute to retain the strength. The low degradation rate of the wire at a high wire content is probably attributed to the high pH value inside the composite. However, during the immersion, small defects preferred to appear along

the interface between the wire and the matrix, which may further decrease the interface properties of the composite. This counter reaction leads to the approximate strength retention for the composite at 10 vol% and 20 vol% under no load. Additionally, it could be noticed that the largest defect preferred to appear at the peripheral outer surface rather than the two ends of the composite. This result may suggest the end sealing treatment would not be essential for the composite at a high wire content or with other composite structure [25], though this may increase the diffusion pathways of water from the two ends of the composite.

4.2 Effect of load mode

According to the study, external load significantly influences the degradation behaviors of the composite and its components. In this study, the specimens subjected to static and dynamic load undergo a significant reduction in pH values and bending strength compared to those under no load. For the PLA matrix, the lower pH value may indicate a higher degradation rate of the PLA matrix under external load conditions, specially under dynamic load. This is probably attributed to the decreased activation energy [26, 27]

and promoted motion of chain segments induced by external load [28]. Interestingly, the R_p of Mg wire in the composite firstly increases after immersion for 7 days. This is probably attributed to the formation of inter- or intramolecular crosslinks induced by the interaction between Mg^{2+} ions from Mg fillers and the carboxylic acid groups from the degradation of PLA matrix [29]. Under the condition of dynamic loading, this interaction would be more significant, which would further decrease the solubility of PLA matrix, leading the largest R_p after immersion for 7 days. As the immersion proceeds, Mg wires would degrade faster under static and dynamic load.

However, the degradation rate of Mg wires under dynamic load is dramatically larger than that under static load after 30 days immersion. This result is consistent with the pH values, which represents a larger value under dynamic load. As noted above, dynamic load could increase convective transport of degradation products, specially the acid degradation products out of the composite. Therefore, the results may indicate the pH value inside the composite under dynamic load would be higher than that under static load, leading to a relatively low degradation rate of Mg wire.

5 Conclusions

Degradable Mg wires /PLA composite are prepared for bone fixation implantation. A high wire content could apparently enhance the mechanical properties of the composite and mitigate the degradation of Mg wires in the composite. The external load is found to significantly affect the *in vitro* performances of the composite and its components. Dynamic load could enhance the bending strength loss of the composite and the transport of acidic degradation products out of the composite, which would further retard the degradation of the inside Mg alloy wire components.

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