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A study of long-term static load on degradation and mechanical integrity of Mg alloys-based biodegradable metals

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ABSTRACT

Predicting degradation behavior of biodegradable metals *in vivo* is crucial for the clinical success of medical devices. This paper reports on the effect of long-term static stress on degradation of magnesium alloys and further changes in mechanical integrity. AZ31B (H24) and ZE41A (T5) alloys were tested to evaluate stress corrosion cracking (SCC) in a physiological solution for 30 days and 90 days (ASTM G39 testing standard). Scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX) and micro-computed tomography (micro-CT) were used to characterize surface morphology and micro-structure of degraded alloys. The results show the different mechanisms of stress corrosion cracking for AZ31B (transgranular stress corrosion cracking, TGSCC) and ZE41A (intergranular stress corrosion cracking, IGSCC). AZ31B was more susceptible to stress corrosion cracking under a long term static load than ZE41A. In conclusion, we observed that long-term static loading accelerated crack propagation, leading to the loss of mechanical integrity.

1. Introduction

Biodegradable metals are expected to degrade gradually *in vivo,* with an appropriate host response while it provides mechanical strength at the initial stage of implantation and slowly be absorbed to the human body. Three metal alloys including magnesium based alloys [1–3], iron based alloys [4,5] and zinc based alloys [6,7] are recently explored as biodegradable metals which can dissolve completely upon fulfilling the mission to assist with tissue healing without leaving any trace. However, complex physiological stresses make biodegradable metals degradation unpredictable, which is one of the challenges for industrial success. Implantable devices in general are subjected to acute dynamic loadings during normal physical activities [8], which can cause stress corrosion cracking (SCC) [9–15] and corrosion fatigue (CF) [13,16,17]. A majority of permanent implant failures are caused by SCC and CF [18–20] after exposure to physiologically stressed environments. However, in case of magnesium alloy based biodegradable metals, higher stress over intrinsic maximum stresses of alloys or threshold stress of SCC can be built by degradation process, which can cause earlier failure of medical device [9–14,17]. A key step is to identify well-defined fracture mechanisms of magnesium alloys under the stress conditions that can be caused from various physiological environment using simplified and standardized *in vitro* test method that can get similar results from *in vivo* until tissues are recovered with enough strength (8–12 weeks). Clear understanding of the relationship between corrosion and mechanical stress can help to design better alloy and medical devices to resist to cracking/fracture.

The magnesium-based biomaterials are under complicated stress conditions like tensile, compressive, bending, shear stresses and distortion during the physical activity after surgery. Implanted surgical pins, screws, and plates can be put under static and dynamic stresses by fixation on irregular bone shape and body weight, and can be three to five times more increased by dynamic stresses during ordinary activities like walking and running [21]. The question is how these complex stresses affect magnesium degradation in terms of degradation rate, uniform degradation, mechanical integrity, localized degradation, product formation, and time-lapse degradation. It is necessary to evaluate the SCC susceptibility of magnesium alloys in simulated physiological conditions with the appropriate testing method. SCC can be caused by concentration of stress at a localized corrosion area, cyclic load and hydrogen embrittlement (HE) during the *in vivo* degradation

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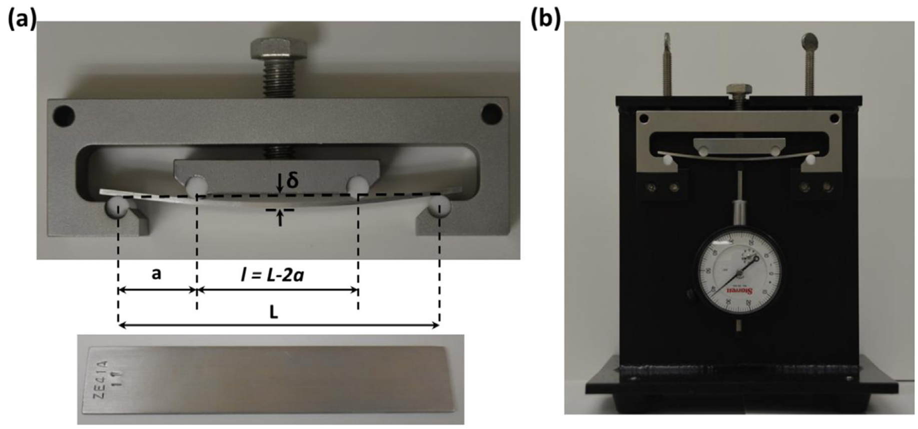

Fig. 1. Four-point bending test apparatus and specimen for stress corrosion cracking test in Hank's Balanced Salt solution. (a) ASTM G39 test apparatus and ZE41A specimen, and (b) Stressing holder with a deflection gauge. a = 22.25 mm, L = 89 mm, l = 44.5 mm.

Table 1
Experimental summary for stress corrosion cracking test.

Classification	Description
Alloys	AZ31B (H24) and ZE41A (T5)
Maximum deflection (y)	AZ31B (3.11 ± 0.03 mm), ZE41A (2.80 ± 0.04 mm)
Solution and volume ratio	Hank's Balanced Salt Solution, 45 ml/cm^2
Test duration	30 and 90 days
Evaluation	Weight loss, SEM, CT, Mechanical properties (E, YS)

Table 2
Summary of tensile and flexural mechanical test with AZ31B and ZE41A (n = 3).

Alloy	Tensile		
	Elastic modulus (GPa)	Yield strength (MPa, 0.2% offset)	Ultimate tensile stress (MPa)
AZ31B	43 ± 0.3	211 ± 1.7	286 ± 3.6
ZE41A	42 ± 1.3	148 ± 1.9	249 ± 2.5
Alloy	Flexural		
	Flexural modulus (GPa)	Yield strength (MPa, 0.05% offset)	Ultimate flexural stress (MPa)
AZ31B	45 ± 0.1	214 ± 17.3	286 ± 7.7
ZE41A	45 ± 0.3	154 ± 4.9	251 ± 9.5

process [22], which can possibly induce sudden fracture under theoretically expected fracture stress limit in shorter life time. Most of SCC studies were done using a slow strain-rate technique over a short term period [10,14,23–25], long-term study with better *in vivo* mimicking environment is critical to understand effect of stress on mechanical and degradation integrity.

This paper investigates the effect of long-term loading on degradation magnesium alloys in terms of corrosion rate, product formation, pitting, crack propagation, and mechanical integrity. The SCC test is conducted with ASTM G39 standard (four-point static load) in Hank's Balanced Salt solution with representative magnesium alloys, AZ31B (H24) and ZE41A (T5). We explore mechanism of SCC through well characterization of surface morphology and micro-structure.

2. Materials and methods

2.1. Materials and stress corrosion cracking test

Certified magnesium alloy coupons, AZ31B (2.5–3.5% Al, 0.7–1.3% Zn, 0.2% Mn, H24) and ZE41A (3.5–5.0% Zn, 0.1–1.7% RE, 0.4–1.9% Zr, T5) were purchased from Metal Samples (division of Alabama Specialty products Inc., Munford, AL). SCC test was conducted with two magnesium alloys (AZ31B (H24), and ZE41A (T5)) in Hank's Balanced Salt solution and it was followed by ASTM G39 testing standard (four-point bending load). In terms

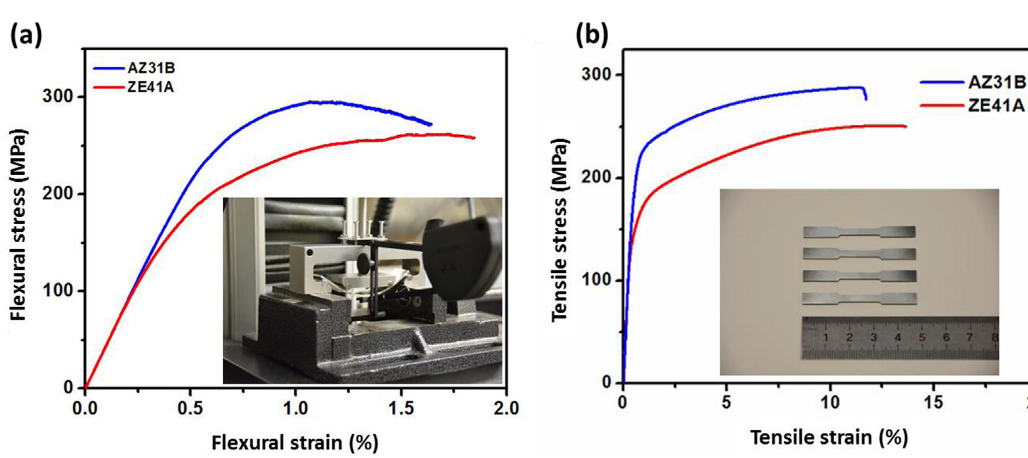

Fig. 2. (a) Flexural stress–strain from four-point bending test of AZ31B and ZE41A (inset shows four-point bending test), and (b) Tensile stress-strain curves from tensile test (inset shows tensile specimens).