Engineering Fracture Mechanics 137 (2015) 2–11

Contents lists available at ScienceDirect

Engineering Fracture Mechanics

journal homepage: www.elsevier.com/locate/engfracmech

Corrosion fatigue of a magnesium alloy in modified simulated body fluid

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ARTICLE INFO

Article history:
Received 18 June 2014
Accepted 13 July 2014
Available online 21 July 2014

Keywords:
Implant materials
Magnesium alloys
Environmental cracking
High cycle fatigue
Hydrogen embrittlement

ABSTRACT

For magnesium (Mg) alloys to be used as temporary biodegradable implants it is essential to establish their resistance to body fluid-assisted cracking. In this paper the fatigue behaviour of a common magnesium alloy, AZ91D, is studied in air and in modified simulated body fluid (m-SBF), and the effect of different electrochemical conditions on corrosion fatigue life is investigated. The alloy was found to be susceptible to corrosion fatigue. Results suggest inclusions and corrosion pits to be the crack initiation sites, and hydrogen embrittlement to play a dominant role in cracking of AZ91D Mg alloy in m-SBF.

1. Introduction

Metallic biomaterials such as titanium alloys, stainless steels and cobalt-chromium alloys are widely used implant materials due to their corrosion resistance and strength [1,2]. However, when these materials are used as temporary implants, their retention in the body becomes unnecessary after they have fulfilled their function, and a removal surgery is required which increases the health care cost as well as inconvenience to the patient [3]. Moreover, the mechanical properties of these alloys are considerably different from those of human bone which results in the problem of 'stress shielding' and consequent reduction in bone density [4].

Magnesium (Mg) alloys are suitable as potential temporary biomedical implants because they are biodegradable and can completely dissolve in the body [5–8], which eliminates the need for secondary surgery to remove an implant. Magnesium is also biocompatible, is essential to human metabolism, and in addition any excess Mg is harmlessly excreted [4,9]. Furthermore magnesium has mechanical properties much closer to bone and this mitigates stress shielding [8]. These properties make magnesium and its alloys suitable as temporary orthopaedic implants (e.g. bone plates and screws) and cardiovascular implants (e.g. stents). In spite of these advantages Mg alloys have found very little actual use in implants, primarily because they tend to corrode too quickly in chloride solutions including physiological environments and therefore lose their mechanical integrity before accomplishing their purpose [10–13].

Orthopaedic and cardiovascular implants generally experience cyclic loading [14,15], which along with the corrosive physiological environment can cause corrosion assisted cracking. Depending on the nature of loading, corrosion assisted cracking includes stress corrosion cracking (tensile loading) and corrosion fatigue (cyclic loading). Stress corrosion cracking (SCC) of Mg alloys has been widely investigated including in physiological environments [16–20] but corrosion fatigue (CF)

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http://dx.doi.org/10.1016/j.engfracmech.2014.07.007
0013-7944/

Nomenclature

Symbols

σ_{LH}	fatigue strength in air
σ_{CE}	fatigue strength in corrosive environment

Abbreviations

Al	aluminium
BEI	back scattered electron imaging
CF	corrosion fatigue
Cl^-	chloride
EDX	energy-dispersive X-ray spectroscopy
HEPES	2-(4-(2-hydroxyethyl)-1-piperazinyl) ethanesulfonic acid
ICP-AES	inductively coupled plasma atomic emission spectroscopy
Mg	magnesium
m-SBF	modified simulated body fluid
NDE	negative difference effect
OCP	open circuit potential
SCC	stress corrosion cracking
SEM	scanning electron microscopy

of Mg alloys in human body fluid has received little attention even though CF fractures cause catastrophic failures of biomedical implants [14,21–24]. There are two main mechanisms for SCC of Mg alloys: hydrogen induced cracking (hydrogen embrittlement), and dissolution assisted cracking [25,26]. But there is limited mechanistic understanding of the role of electrochemical polarization on corrosion fatigue cracking of Mg alloys.

Aluminium (Al) containing Mg alloys are widely used due to the beneficial roles of Al in corrosion resistance and mechanical properties [27,28]. The AZ91D alloy exhibits good biocompatibility and causes no harm to the surrounding tissues [6,29,30]; in vivo and in vitro studies on this alloy have shown no adverse toxicity due to Al. However, there are also reports to suggest that Al causes Alzheimer's disease, muscle fibre damage, as well as Al^{3+} ions combining with inorganic phosphate, causing phosphate deficiencies in the body [27,31–33]. Consequently, it is unlikely that AZ91D can be used for implants. However, AZ91D is the most investigated alloy for corrosion assisted cracking in chloride solutions, and the present study on CF of this alloy in the physiological environment provides an improved mechanistic understanding, and baseline data for magnesium alloys that can be actually used as biodegradable implants.

2. Experimental procedure

2.1. Materials

Magnesium alloy AZ91D alloy was received in sand-cast form, the chemical composition of which was analysed by inductively coupled plasma atomic emission spectroscopy (Table 1). The tensile properties of the AZ91D are listed in Table 2.

2.2. Fatigue and corrosion fatigue tests

Fatigue samples with 6 mm gauge diameter and 15 mm gauge length (ASTM E466 [34]) were tested (Fig. 1a). The specimen gauge was abraded in the loading direction with 1200 and 2500 grit emery papers, and then polished with 1 μm diamond paste followed by cleaning with ethanol and de-ionised water. In order to obtain an appropriate alignment of the specimen and also save the amount of material at two shoulders, a sample holder (made of high strength steel) with collet chuck was used for gripping (Fig. 1b).

A corrosion chamber made of acrylic was attached to the sample (Fig. 2). Any leakage of solution from the chamber was prevented by using two O-rings fitted to the shoulders of the fatigue specimens. Modified simulated body fluid (m-SBF) used for corrosion fatigue experiments has a chloride ion concentration close to that of blood plasma and was buffered with 2-(4-(2-hydroxyethyl)-1-piperazinyl) ethanesulfonic acid (HEPES) at a pH of 7.4 (Table 3) [35].

Table 1
Chemical composition of AZ91D magnesium alloy.

Element	Mg	AL	Zn	Mn	Cu	Fe	Ni	Si	Be
Wt.%	Bal	8.89	0.78	0.20	0.002	0.002	<0.001	<0.01	<0.001