

# On the biodegradation performance of an Mg–Y–RE alloy with various surface conditions in simulated body fluid

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Received 27 March 2008; received in revised form 25 June 2008; accepted 31 July 2008

Available online 14 August 2008

## Abstract

This study documents the influence of different surface conditions produced by various heat treatments on the in vitro degradation performance of an Mg–Y–RE alloy (WE43) investigated by immersion in simulated body fluid. WE43 samples were, respectively (i) annealed at 525 °C (plus artificial aging at 250 °C in one case) and afterwards polished; and (ii) polished, annealed at 500 °C in air and subsequently investigated in the oxidized state. Thermogravimetric analysis (TGA) indicates a mass gain during oxidation in air, following a square-root law over time. X-ray diffraction spectra imply a growing Y<sub>2</sub>O<sub>3</sub> layer upon oxidation, and Auger electron spectroscopy depth profiles show an increased oxide layer thickness which develops according to the behavior observed by TGA. Macroscopically, the degradation performance of the differently heat-treated samples can be divided into two groups. Annealed and polished samples show a fast and homogeneous degradation which slows with time. Their degradation behavior is approximated by a parabolic law. Oxidized samples exhibit a slow initial degradation rate which increases when the protection of the oxide layer is reduced. Overall, they reveal a sigmoidal degradation behavior. Here the differing degradation performances of the annealed–polished and the oxidized samples are related to the different surface conditions and explained on the basis of a depletion hypothesis.

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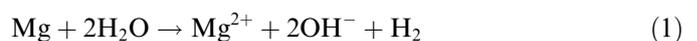
**Keywords:** Magnesium biodegradation; Simulated body fluid; Surface conditions; Oxide layer; Yttrium depletion

## 1. Introduction

In the field of bioabsorbable implant materials, magnesium has become an interesting candidate as a bioactive substance which naturally degrades within the body. In general, bioabsorbable implants offer the advantage that they need not be removed in a second surgical intervention after serving their purpose, which makes them both convenient for the patient and more economical. Magnesium has great potential as a bioabsorbable material: it is an essential element in the human body, it degrades in aqueous solutions and its mechanical properties are more advantageous than those of degradable polymeric materials. However, to be suitable bioabsorbable implant material, Mg alloys must possess certain properties: (i) appropriate

mechanical characteristics at room temperature, in order to offer adequate mechanical support to the injured tissue; (ii) a moderate degradation performance able to assist the healing process effectively and to prevent from early destabilization; and (iii) ions released during degradation need to be biocompatible.

At pH values below 11.5 magnesium corrodes in aqueous solutions [1]. Its degradation can then be described by the following electrochemical equation:



The formation of OH<sup>−</sup> ions and gaseous hydrogen upon the degradation of Mg, however, may harm injured tissue, as the pH value increases locally and gas bubbles build up. Therefore a low initial degradation rate is desirable to avoid further deterioration of the adjacent tissue. Extensive research has been performed on enhancing the corrosion resistance of Mg alloys by modifying their microstructure

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or their surface, including the application of conversion coatings [2,3]. These methods, however, are often rather sophisticated and may include the release of harmful substances. Heat treatments offer a simple method for improving corrosion resistance, by altering the microstructure and/or the surface condition of the alloy without adding undesired substances.

The goals of this study are twofold: (i) to investigate the potential of different heat treatments on the decrease of the initial degradation rate of an Mg–Y–RE alloy (WE43); and (ii) to analyze the influence of surface condition on the degradation performance. We report on the effect of thermal oxidation treatments of WE43 on the formation of Y-containing surface films, and the corresponding change in the in vitro degradation behavior in physiological media. The alloying system was selected because yttrium is known to be beneficial in enhancing the corrosion resistance of Mg-alloys [4–6], and WE43 has been shown in recent studies to meet many requirements for implant applications [7–9].

## 2. Materials and methods

### 2.1. Sample preparation and denotations

Disks of 22 mm in diameter and 3.6 mm thick were cut from an extruded bar. Material specifications are given in Table 1, according to ASTM designations. The whole disk surface was ground and polished (to 0.25  $\mu\text{m}$  diamond polish) either before or after the oxidation heat treatments, and cleaned in isopropanol in an ultrasonic bath. All heat treatments were performed in an air-circulating oven (Heraeus 170/2). Samples of one series were first heat-treated at 525 °C for 6 h (solution heat-treated condition, SHT) and then water-quenched. Some of these samples were additionally artificially aged at 250 °C for 16 h (condition T6). Following the heat treatments all samples of the first series were ground and polished. Samples of the second series were first polished, then annealed at 500 °C and left in the “thermally oxidized” state, being covered by an oxide layer due to the heat treatment (oxidized condition). The oxidation in air was carried out for 1, 8, 24, 48 and 168 h (1 week), respectively, and the samples were air-cooled after annealing. Table 2 gives an overview on the heat treatments performed and sample denotations used in this study. In order to minimize the contact area between spec-

Table 1  
Chemical composition of WE43

Element	Mass fraction (wt.%)
Y	3.7–4.3
RE <sup>a</sup>	2.4–4.4
Zr	>0.4
Mg	Balance
Ni	<5 ppm
Fe	<0 ppm
Cu	<20 ppm

<sup>a</sup> RE consists of Nd and heavy RE (Yb, Er, Dy, Gd).

Table 2  
Heat treatments, sample denotations and sample series

Temperature (°C)	Annealing time (h)	Label	Sample series
525	6	SHT	p <sup>a</sup>
525 + 250	6 + 16	T6	p
500	1	ox1	ox <sup>b</sup>
500	8	ox8	ox
500	24	ox24	ox
500	48	ox48	ox
500	168	ox168	ox

<sup>a</sup> Polished.

<sup>b</sup> Oxidized.

imen and holder, the samples to be oxidized were placed on a tripod of alumina sticks during heat treatments to facilitate homogeneous oxidation of the entire sample surface.

### 2.2. Microstructure and characterization

The microstructures of all samples were investigated using optical microscopy. The oxidized samples were additionally investigated by thermogravimetric analysis (TGA), X-ray diffraction (XRD) and Auger electron spectroscopy (AES). Isothermal TGA measurements (Netzsch STA 449C) were carried out at 500 °C in air for 120 min and the mass gain over time was recorded. The TGA specimens measured 5 mm  $\times$  5 mm  $\times$  9 mm, and were ground with SiC abrasive paper up to a grit of 4000 and degreased in isopropanol directly before measurement. For phase identification of the oxidized samples XRD measurements (PANalytical X'Pert PRO-MPD) were conducted using a Cu  $K_{\alpha 1}$  ( $\lambda = 0.15406$  nm) source operated at 38 kV and 45 mA. AES (Perkin-Elmer PHI 4300 SAM) was performed at 5 keV using an LaB<sub>6</sub> filament. Depth profiles were recorded using an Ar ion gun operated at 4 keV. The sputter rate was 25 nm min<sup>-1</sup> and calibrated for Ta<sub>2</sub>O<sub>5</sub>. Taking into account the thicker surface layer on the samples oxidized for 48 h and 1 week at 500 °C, the sputter rate for these samples was increased to 37.5 and 75 nm min<sup>-1</sup>, respectively.

### 2.3. Immersion testing

The degradation performance was evaluated by immersing the heat-treated samples in simulated body fluid (SBF). According to Eq. (1) every dissolved mole of magnesium produces 1 mol of hydrogen, which makes it possible to derive the corrosion rate from the amount of hydrogen evolved. Song et al. [10] developed the hydrogen evolution method, which is based on the collection of hydrogen gas during degradation of magnesium in aqueous solution. The experimental set-up is simple: a magnesium sample is placed in a beaker containing the immersion liquid. A measuring cylinder filled with the liquid is placed over the sample in order to collect the hydrogen formed during the sample's corrosion. The amount of hydrogen collected in the measuring cylinder over time reveals the degradation

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