# Corrosion and Mechanical Performance of AZ91 Exposed to Simulated Inflammatory Conditions

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## INTRODUCTION

Magnesium (Mg) and its alloys are a class of biodegradable metals which have gained increasing interest as orthopaedic implant materials.[1,2] Alloys, including Mg-9%Al-1%Zn (AZ91), possess mechanical properties similar to cortical bone, lessoning concerns of stress shielding in load bearing applications.[1] Previous work has shown that an inflammatory reaction in response to device implantation can influence the corrosion rate of metallic biomaterials.[3]

## OBJECTIVES

- To assess the corrosion rate of AZ91 exposed to simulated inflammatory conditions in a physiologically relevant medium.
- To investigate the effects of a simulated inflammatory response on the mechanical properties AZ91.

## METHODS

### Experimental Groups

| | Test Electrolyte | |
|---|---|---|
| | Normal | Inflammatory |
| Days 0-3 | α-MEM +10% FBS | α-MEM + 10% FBS 150mM $H_2O_2$ pH=5 |
| Days 4-6 | α-MEM +10% FBS | α-MEM +10% FBS |

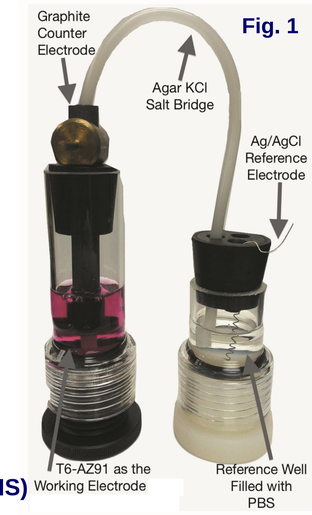



Fig. 1

**Material** T6 treated Mg-9%Al-1%Zn alloy

**Sample Preparation** Wet sanded to 600 grit finish, sonicated, and placed under UV light for 30 minutes for sterilization.

### Electrochemical Tests

**Electrochemical Cell** (Fig. 1)
- Connections were made to a potentiostat
- Cells placed in 37°C, 5%$CO_2$ incubator

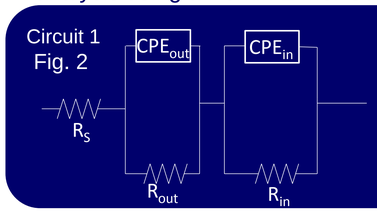
**Electrochemical Impedance Spectroscopy (EIS)**
- Performed at day 3 and day 6
- +/- 20mV (about OCP) from 100KHz-5mHz
- Results fit to one of two circuit models (Fig. 2, 3)

**Scanning Electron Microscopy (SEM)**
- Samples were allowed to air dry and examined with a Hitachi SU70

**Mass Loss**
- Samples were allowed to air dry
- Corrosion product was removed by immersion in 200g/L chromium trioxide and 10g/L silver nitrate

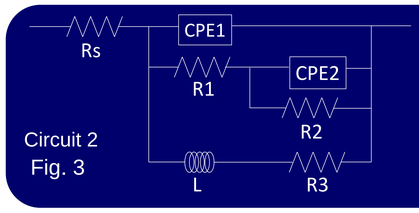
**Inductively Coupled Plasma Mass Spectroscopy**
- Used to identify concentration of ions released into media
- 2mL aliquots were collected and underwent an acid digestion before analysis using a Perkin Elmer Sciex model ELAN DRC-II

Circuit 1
Fig. 2




Rs – Solution Resistance
Rout – Outer Layer Resistance
Rin – Inner Layer Resistance

CPE – Constant Phase Element
Q – Magnitude of the CPE
α – Exponent of the CPE




Circuit 2
Fig. 3

Rs – Solution Resistance
R1, R2, R3 – Circuit Resistors
CPE1, CPE2 – Constant Phase Element
Q – Magnitude of the CPE
α – Exponent of the CPE
L – Inductor

### Mechanical Testing

- AZ91 rods were immersed for 3 or 6 days (Fig. 4)
- A Bose load frame displaced the samples at 2mm/min until failure occurred (Fig. 5)
- 3-point bend tests were used to determine flexural modulus, flexural yield strength, and ultimate flexural strength

Fig. 4

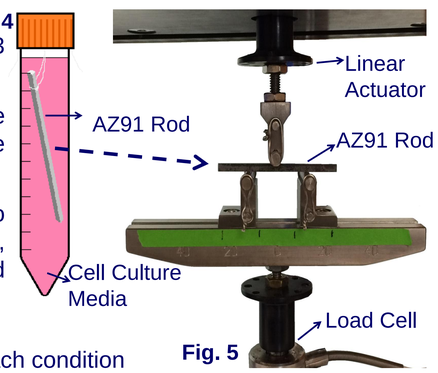



Fig. 5

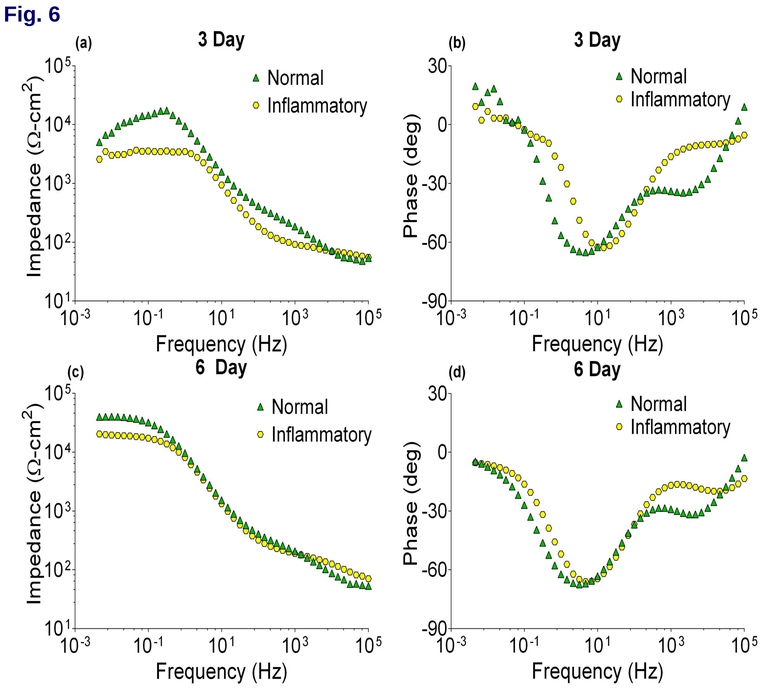
### Statistical Analysis

- 3 Samples were assessed at each condition
- Rp and Corrosion Rate: T-tests ($p<0.05$) were used to compare normal and inflammatory conditions at 3 and 6 days, and across conditions at each time point
- ICPMS and Mechanics: Differences were determined using an ANOVA/Tukey's post-hoc ($p<0.05$)
- Log transform data was used for ICPMS Results

## RESULTS

### Electrochemical Impedance Spectroscopy (EIS) Bode Plots

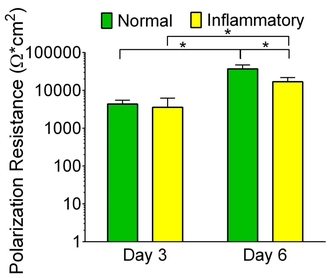
Fig. 6




Bode plots resulting from EIS of normal and inflammatory samples after 3 or 6 days of incubation. Figures 4a and 4c represent the impedance modulus curve at 3 and 6 days respectively, while figures 4b and 4d represent the phase angle curve at 3 and 6 days respectively.

### Polarization Resistance (Rp) Determined from EIS

Fig. 7




Rp Calculations:

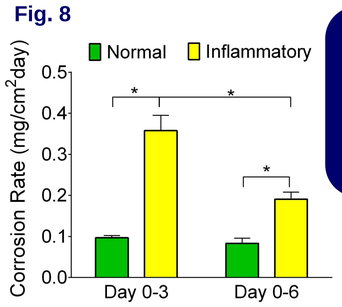
Circuit 1 (Day 6):
$Rp = Rout + Rin$

Circuit 2 (Day 3):
$\frac{1}{Rp} = \frac{1}{R1+R2} + \frac{1}{R3}$

Figure 7 reports the polarization resistance (Rp) calculated for normal and inflammatory samples after 3 or 6 days of incubation. Rp is inversely proportional to the corrosion current.

### Corrosion Rate Determined by Mass Loss Testing

Fig. 8




Corrosion Rate (R) Calculations

Variables:
Wb=Weight before (g)
Wa=Weight after (g)
A=Surface area (cm²)
t=exposure time, days

$$R = \frac{(Wb - Wa) \times 100}{A \times t}$$

Figure 8 reports the corrosion rate for normal and inflammatory samples as determined by mass loss testing. Values reflect the initial (0-3day) period, or entire immersion (0-6day).

### ICPMS Results for Mg, Al, & Zn Ion Concentration

Fig. 9

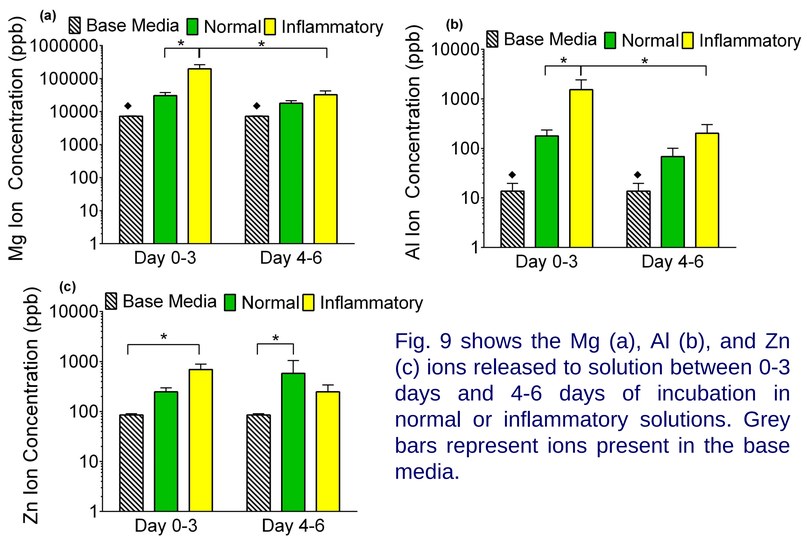



Fig. 9 shows the Mg (a), Al (b), and Zn (c) ions released to solution between 0-3 days and 4-6 days of incubation in normal or inflammatory solutions. Grey bars represent ions present in the base media.

## RESULTS

### Scanning Electron Microscopy (SEM) Results

Fig. 10

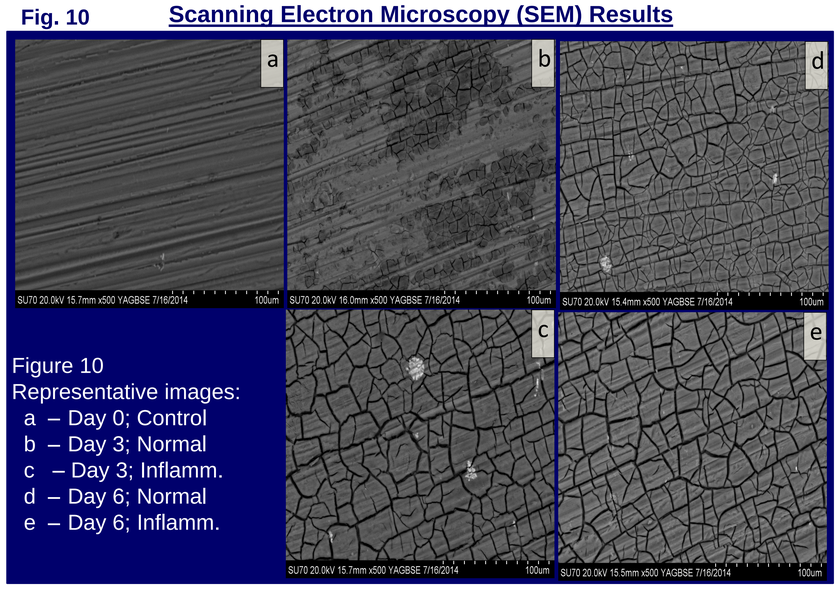
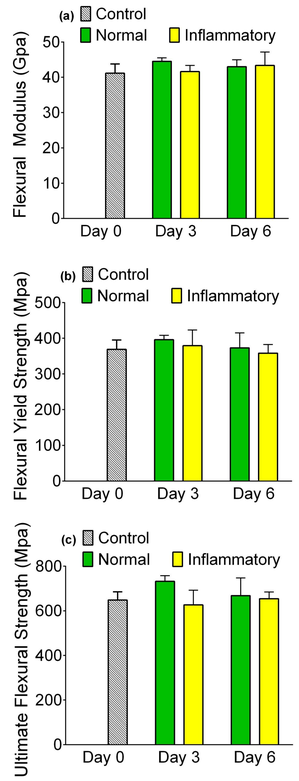



Figure 10
Representative images:
a – Day 0; Control
b – Day 3; Normal
c – Day 3; Inflamm.
d – Day 6; Normal
e – Day 6; Inflamm.

### 3-Point Bend Test Results

Fig. 11



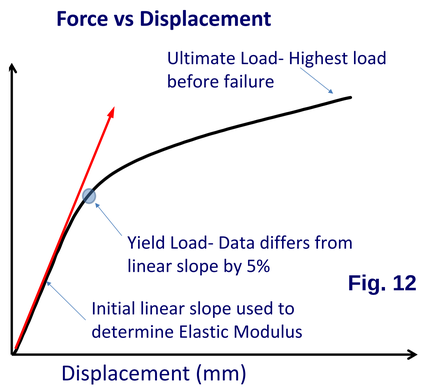




Fig. 12

Fig. 11 reports the values for flexural modulus (a), flexural yield strength (b), and ultimate flexural strength (c) for normal and inflammatory samples. Grey bars represent mechanical properties of a control group which was not immersed in solution.

Fig. 12 displays sample raw data collected and demonstrates how the outcomes were determined from the generated Force vs. Displacement curve.

## SIGNIFICANCE

- AZ91 samples exposed to a simulated acute inflammatory response displayed accelerated corrosion at early time points. This is demonstrated by increased mass loss (Fig. 8) and increased release of Mg and Al ions (Fig. 9a, Fig. 9b) when compared to the control condition during the first three days of exposure.
- The polarization resistance of AZ91 exposed to both normal and inflammatory conditions is increased at 6 days compared to 3 days (Fig. 7), indicating a decrease in corrosion rates over time.
- No significant differences were found in the mechanical properties of AZ91 compared to control samples which were tested as polished (no electrolyte immersion). The lack of changes in mechanical properties is likely due to minimal mass loss over the test period. Longer specimen immersion times will be studied to identify changes in the mechanical properties of AZ91 as the material degrades and corrodes.

## REFERENCES

1. Staiger, M. P., et al. (2006). "Magnesium and its alloys as orthopedic biomaterials: a review." Biomaterials **27**(9): 1728-1734.
2. Barfield, W. R., et al. (2012). "The potential of magnesium alloy use in orthopaedic surgery." Current Orthopaedic Practice **23**(2): 146-150.
3. Fonseca, C., Barbosa, M.A. (2001). "Corrosion behaviour of titanium in biofluids containing H2O2 studied by electrochemical impedance spectroscopy." Corrosion Science **43**: 547-559.