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Corrosion stress relaxation and tensile strength effects in an extruded AZ31 magnesium alloy

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ABSTRACT

The corrosion effects on the tensile and stress relaxation behavior of an extruded AZ31 magnesium alloy subjected to immersion and salt-spray environments have been investigated. Specimens were simultaneously corroded and stress relaxed in a 3.5 wt.% NaCl solution and then put under a tensile test to failure to determine the stress–strain response over a 60 h test matrix. The AZ31 magnesium alloy shows an evident relaxation in 3.5 wt.% NaCl at room temperature. According to optical and scanning electron microscopy investigations, the fracture surfaces for the immersion environment show a high sensitivity to stress corrosion cracking.

1. Introduction

Magnesium (Mg) alloys exhibit the attractive combination of low densities and high strength per weight ratios (comparable or greater than that of precipitation strengthened Al alloys), along with good damping capacity, castability, weldability, and machinability [1–6]. These characteristics promote the increase use of Mg alloys in electronics, automobile, and aerospace industries [2–4,7]. Of the various commercial Mg alloys, those developed from the Al–Zn ternary system (i.e. the as-named AZ alloys) have found the largest number of industrial applications [3]. Although Mg alloys are some of the best candidates, their low resistance to corrosion [4,8–12], creep [13,14], and stress relaxation behavior [15] hinder their use. In recent years, efforts to improve the creep and corrosion behavior of Mg alloys have been intensive [14]. While the creep and corrosion properties can be improved considerably through alloy chemistry design [16,17], cost-effective methods for mitigation of corrosion are still lacking. One of the elements commonly added to Mg is aluminum. When added to the alloy at no more than 10%, improved corrosion resistance was observed [17,18]. Evidence can be found by the corrosion quantification studies completed by Walton et al. [19] on AZ31 and Martin et al. [20] on AZ61 and AZ91.

Two techniques are commonly performed for corrosion testing, salt spray (salt fog) and immersion testing. ASTM standards have been developed for both the salt spray environment and the immersion environment, labelled as ASTM B-117 and ASTM G-31, respectively [21,22]. However, the two ASTM standards require different concentrations of salt, signifying a direct comparison between the results cannot be made [21,22]. In addition, the two methods do not translate well to corrosion field tests performed for the automotive industry [23,24]. This issue has led to the industrial development of cyclical tests, which contain a pollution phase and a wet or dry phase, in an effort to expose test alloys to the environmental factors associated with engine cradles, such as de-icing salt, mud, and condensation [24,25].

General corrosion under stress is quite different from stress-free conditions. The combined action of corrosion and stress can cause premature failure compared to the failure rate of the two loading cases separately. Due to the serious negative effects on durability, corrosion stress relaxation of Mg alloys should trigger substantial attention focused on the behavior under automotive service conditions. Knowledge of environmental factors that influence degradation, types of corrosion to which Mg alloys are most susceptible, protection schemes, and design considerations can significantly minimize corrosion and increase the use of this family of lightweight alloys. The conceptual interrelationship of corrosion, stress effects, and hydrogen embrittlement (HE) play a huge role in the understanding all of the key mechanisms. The most serious practical situation is when all three phenomena interact. For example, Scamans et al. [26] showed that AZ63 becomes embrittled if pre-exposed to moist gases prior to tension test. The types of testing are important and the magnitude of applied stress is not the only

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critical loading parameter. Time dependent environmental effects are also of primary importance. The damage mechanism describing Mg alloys under simultaneous corrosion and stress environments is called stress corrosion cracking (SCC) [27–37]. SCC is broken down into two main forms: intergranular stress corrosion cracking (IGSCC) and transgranular stress corrosion cracking (TGSCC) [27,28]. IGSCC is typically caused by a continuous second phase along grain boundaries where TGSCC is caused by an interaction of hydrogen with the microstructure [27]. The understanding of SCC is urgently needed because Mg alloys are being increasingly used in load bearing applications; many common Mg alloys have a threshold stress for SCC of half the yield stress in common environments including high-purity water [27]. Early reviews in the 1960s reported that alloying addition such as Al and Zn to wrought Mg alloys promoted SCC; thus wrought AZ alloys are susceptible to SCC for intermittent exposure to 0.01% NaCl and to the weather [27]. For AZ31, the SCC threshold stress is 40% of the yield stress in a rural atmosphere [27].

A literature review of Mg corrosion stress relaxation found little published material. Unigovski et al. [15] studied the effect of environment, stress, and test temperature on stress relaxation in pure Mg and die-cast Mg alloys (AZ91D, AM50, and AS21) where they found that pure Mg shows a crucial corrosion stress relaxation in 3.5% NaCl solution at room temperature. Trojanová et al. in 2007 [38] studied the stress relaxation phenomena in AJ51 and AX41 Mg alloys. Trojanová et al. [39] followed that work with research on the AZ63 Mg alloy. The work was primarily focused on analyzing the stress relaxation curves as a function of the internal stress at the beginning of the relaxation and the test temperature.

It is generally accepted for many metals that pit evolution under a corrosive solution is the dominant source for crack initiation and propagation. This is reasonable because pits cause local stress concentrations that increase the driving force for cracking. Because this assumption has fundamental significance for modeling, experiments have been conducted to investigate pit initiation and evolution for the AZ31 Mg alloy under 3.5 wt.% NaCl aqueous solution. A cyclical salt spray test and immersion techniques were employed under a simultaneous stress relaxation deformation in an effort to expose the AZ31 Mg alloy to an environment similar to that experienced by automotive engine cradles. The experimental aims for this study were to determine the relaxation behavior of the AZ31 alloy under corrosive environments and to find direct or indirect evidence to support the assumption that pit evolution can lead to the crack initiation and material failure.

2. Materials and methods

2.1. Set-up

Thirty-six AZ31 specimens were machined from an extruded sheet in the longitudinal direction. Table 1 lists the nominal [40] and specimen chemical composition by weight percent. The Fe pickup during the extrusion process is noticed, but is expected to have minimal effect with respect to the corrosion and mechanical properties. In this study, corrosion was localized to one side of the specimen's gauge length. The corrosion surface of the specimens were left untreated, with no surface grinding or polishing, to test the corrosion effects on an as-extruded AZ31 Mg alloy. The other side of the specimens were equipped with strain gauges to monitor an applied strain. After the strain gauges were attached, all surfaces (excluding the corrosion surface) were covered with beeswax to prevent corrosion during the tests. For both environments (salt spray and immersion), three specimens per test environment were pinned in stress relaxation fixtures covered in beeswax. The specimens then were deformed to a true strain corresponding to approximately 80% of tensile yield stress (TYS). The fixtures restrained the specimens at the constant elongation.

2.2. Environmental exposure

Immediately following the applied strain, the specimens were exposed to the two different test environments for various times (t_0 = 0 h, t_1 = 1 h, t_2 = 4 h, t_3 = 12 h, t_4 = 36 h, and t_5 = 60 h), rinsed with distilled water to remove excess salt, and dried in a dessicator. Each relaxation test was continuously loaded. For the cyclic salt spray testing, a Q-Fog CCT (Q-Panel Lab Products, Cleveland, OH) was used to cycle through three stages set at equal times, including a 3.5 wt.% NaCl spray at 35 °C, 100% humidity using distilled water at 35 °C, and a drying purge at 35 °C. This type of testing was used to replicate cyclic automotive exposures. For immersion testing, an aquarium filled with 3.5 wt.% NaCl solution at room temperature was used to replicate the "worst-case" scenario.

2.3. Data analysis and fractography

Due to the inability to monitor load while the specimens were in the immersion and salt spray environments, the stress relaxation data points were gathered at the given interval times following tension testing. This meaning that stress calculations during the relaxation tests were not done, but were interpolated based off of the known strain value on the stress–strain relationship. Since the applied strain for the relaxation tests was in the elastic regime of the AZ31 stress–strain relationship, backing out the stress at the given strain after reloading should theoretically give an accurate stress picture over time. It is important to note that one might expect slightly different results when comparing intermittent loading (capturing stress following unload) with continuous loading (monitoring stress while exposed) due to plastic microdeformation in the relaxation process. A baseline, or control, specimen was run in air for the 60 h time for comparison, but with an Instron electromechanical test machine recording stress over time.

After the combine corrosion and stress relaxation tests, tensile tests were performed using an electromechanical Instron 5882 machine at a constant strain rate of 0.001/s. The machine was equipped with a 5 kN load cell. For each tested time and environment, stress–strain data was garnered for three specimens, for a total of six samples per time. For the cross-sectional area used for true stress calculations, corrosion damage was taken into consideration. A thickness loss and average pit depth measurement allowed for an approximation of the cross-sectional area at the interval times. The stress–strain data gathered was analyzed using the Data Averager (v. 2.5) developed by Mississippi State University to determine average stress–strain plots. The Data Averager was set

Table 1
Nominal elemental composition of AZ31 versus the extruded alloy composition (in wt.%) used in this study.

	Al	Zn	Mn	Si	Cu	Fe	Mg
Nominal AZ31 [40]	2.5–3.5	0.6–1.4	≧0.2	≦0.1	≦0.05	≦0.005	97
AZ31 specimens	2.78	1.02	0.345	0.0063	0.0062	0.024	95.6