CORROSION-STRESS RELAXATION EFFECTS ON TENSILE PROPERTIES OF AN AZ61 MAGNESIUM ALLOY

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Abstract

One way to increase gas mileage 50% by 2050, a goal of the U.S. government, is to reduce the weight of the vehicle, using lightweight alloys such as magnesium. AZXX Mg alloys have been investigated for this purpose, but are still highly susceptible to stress corrosion cracking. The presence of pits and hydrogen embrittled sections concentrate the stress, leading to cracking and failure. In order to determine the interaction between tensile properties and corrosion behavior, two saltwater environments were used to examine the effects of chloride ion exposure on the corrosion of an extruded AZ61 alloy held under constant strain (approximately 80% of tensile yield strength) over 60 hours. The effects of constant strain on the surface corrosive behavior and the tensile strength were determined at various intervals. The stress-strain relationship minimally decreased over time for the salt spray environment, while large changes were seen in the stress-strain relationship for the immersion environment. In addition, there was a minimal decrease in stress over 60 hours of the AZ61 alloy in the salt spray environment but a 20% decrease in stress over 60 hours in the immersion environment. The differences between stress-strain relationships were attributed to a decrease in surface area of the samples due to the continuous presence of water for the immersion environment, which resulted in a decrease in the ability to withstand applied stress. The formation of pits due to chloride ions for both environments concentrated the applied stress, leading to a decrease in the elongation to failure for both environments.

Introduction

The low density, high strength per weight ratio, castability, weldability, and machinability of magnesium alloys make them a desirable material for use in the electronics, automotive, and aerospace industries [1-7]. The addition of aluminum and zinc, to create the Al-Zn ternary system (also known as the AZ alloys), have been used in the largest number of industrial applications [3]. However, the low corrosion resistance of magnesium, as well as the low resistance to creep and stress relaxation behavior, prevents the use of magnesium in areas exposed to the environment [4, 8-15]. Efforts to improve the corrosion and creep performance of magnesium have been focused specifically on improving the alloy chemistry of the alloys [16-17]. The addition of aluminum, up to 10%, improved the corrosion resistance of the magnesium alloy, as long as the β-phase was finely distributed [17-18]. The improvement of corrosion resistance with increasing aluminum can be seen by comparing the quantification studies performed on AZ31 [19] and AZ61 [20]. These results showed that an increase from 3% to 6% aluminum, while holding the zinc steady at 1%, improved the corrosion resistance of the magnesium alloy with respect to general, pitting, and intergranular corrosion in both a salt spray and immersion environment [19-20]. The effect of a poorly distributed β-phase on the corrosion resistance of magnesium can be seen by comparing the quantification studies performed on AZ61 [20] and AZ91 [21]. These results showed that an increase from 6% to 9% aluminum, while holding the zinc steady at 1%, decreased the corrosion resistance of the magnesium alloy with respect to general, pitting, and intergranular corrosion in an immersion environment, while improving the corrosion resistance with respect to general, pitting, and intergranular corrosion in a salt spray environment [20-21]. The difference between the two results was attributed to the continuous presence of water in the immersion environment causing the formation of micro-galvanic cells formed between the α-phase and β-phase that led to an increase in corrosion of the AZ91 alloy [21].

While several corrosion studies have been performed on various magnesium alloys, the corrosion mechanisms in a stress-free environment are different from the corrosion mechanisms under stress. When combining corrosion and stress, the failure rate can greatly increase as compared to the failure rate of just one of the two loads. The main damage mechanism created when stress effects and corrosion interact on magnesium is called stress corrosion cracking (SCC), of which, there are two main forms – intergranular stress corrosion cracking (IGSCC) and transgranular stress corrosion cracking (TGSCC) [22-23]. The formation of IGSCC usually is caused by a continuous β-phase running along grain boundaries, while the formation of TGSCC usually is caused by an interaction of hydrogen within the microstructure [22]. With the increasing usage of magnesium alloys in load bearing applications, an understanding of SCC is urgently needed, especially since many magnesium alloys have a threshold stress for SCC that is half of the yield stress in common environments, including high-purity water [22]. Unlike corrosion, though, the addition of aluminum and zinc did not improve SCC. Instead, SCC was increased, meaning that AZXX alloys are susceptible to SCC even when the exposure to a salt solution or the weather is periodic [22]. While the SCC of magnesium alloys is important, especially for its utilization within the automotive and aerospace industries, only a small amount of published literature is available for corrosion stress relaxation. The study of SCC using various magnesium alloys, including pure magnesium, AZ91D, AM50, and AS21 showed that room temperature and a 3.5% sodium chloride aqueous solution was enough to develop stress relaxation phenomena [15]. Tests run on AJ51 (Mg-5Al-0.6Sr), AX41 (Mg-4Al-1Ca), and AZ63 demonstrated that the stress relaxation phenomena was also a function of both the internal stress at the beginning of the relaxation and the test temperature [24-25].

The available research showed that the time dependent environmental effects are important, not just the magnitude of the applied stress. In order to develop an understanding of how the environment affects the corrosion stress relaxation, two techniques commonly performed for corrosion testing can be used. These techniques were developed for salt spray (salt fog) and immersion testing and come from the ASTM standards of ASTM B-117 and G-31, respectively [26-27]. Unfortunately, though, utilizing the ASTM standards exactly as written would produce results that cannot be completely compared, as the salt concentrations for the two standards are different (5.0% vs. 3.5%, respectively) [26-27]. In addition, the continuous nature of both the salt spray and the immersion environments do not produce results that are translatable to the automotive industry, which requires a pollution phase, a wet phase, and a drying phase [28-29]. In order to produce data that was easily comparable between the two environments, and that was translatable to the automotive industry, a cyclical salt spray testing utilizing a salt spray (pollution) phase, 100% humidity (wet) phase, and a dry air (drying) phase was developed [30]. This allowed for a direct comparison between the worst case scenario (immersion) and a more industrially relevant scenario (cyclical salt spray) to expose the test alloys to the cyclical nature of environmental factors, such as de-icing salt, mud, and condensation [28, 31].

Because metals used in automotive service conditions experience both stress relaxation effects and corrosion, magnesium alloys should be thoroughly examined to understand the negative effects of the combined corrosion and stress relaxation loads on durability. The interaction between stress effects and corrosion, including hydrogen embrittlement, significantly influences the understanding of key failure mechanisms. In addition, the presence of pitting, caused by chloride ions, is the dominant source for crack initiation and propagation, due to the formation of local stress concentrations that increase the driving force for cracking. The interaction between pit nucleation, growth, and coalescence and stress relaxation deformation was examined in a 3.5% sodium chloride aqueous solution utilizing a cyclical salt spray environment and an immersion environment on an extruded AZ61 magnesium alloy, to determine the overall effect to the stress-strain relationship.

Materials and Methods

Sample Cutting

Thirty-six AZ61 specimens were cut from an extruded crash rail provided by Ford using a CNC Mill (Haas, Oxnard, CA). A schematic of the specimen geometry is illustrated in Figure 1.
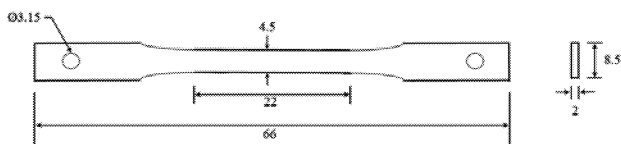

Figure 1. Schematic of specimen used in this study. All dimensions are in millimeters.

Application of Stress

Prior to situating the specimens within the cradle (shown in Figure 2), the gage length of the specimens was lightly sanded on one side and then cleaned with isopropyl alcohol. A resin was

mixed and a strain gauge was attached to the surface, situated to measure the strain from the top to the bottom.

Two weeks after the strain gauge was attached, to allow for the resin to cure, the samples were pinned in the stress relaxation fixtures (Figure 2). All of the available surfaces of both the stress relaxation fixture and the magnesium specimens, with the exception of the surface on which corrosion was to occur, were coated in melted beeswax to prevent preferential corrosion of magnesium, as the fixture was made of steel. In this study, corrosion was localized to one side of the specimen's gauge length. The corrosion surface of the specimens was left untreated, with no surface grinding or polishing, to test the corrosion effects on an as-extruded AZ61 Mg alloy. For both environments (salt spray and immersion), three specimens per test environment were pinned in stress relaxation fixtures covered in beeswax (Figure 2).

After the samples were pinned and coated with beeswax, the strain gauges were attached to a computer and monitored as the nuts on the left side of Figure 2 were tightened. The nuts were tightened until the specimens were deformed to a true strain of approximately 80% of tensile yield stress. Following specimen deformation, additional beeswax was used to fully coat any areas where the previously applied beeswax may have cracked due to the moving of the pinned specimen supports.
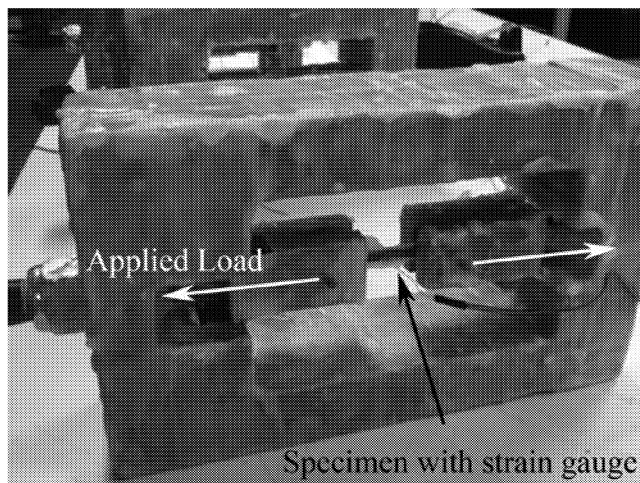

Figure 2. Experimental corrosion stress relaxation fixture setup showing load direction and specimen equipped with a strain gauge to monitor applied strain.

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 desiccator. 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. For immersion testing, an aquarium filled with 3.5 wt.% NaCl solution at room temperature was used.

Optical Microscopy Testing Set-Up

For optical microscopy, one set of immersion and salt spray specimens (six total specimens) were exposed to the test environment for 1 h, removed, rinsed with distilled water to

remove excess salt, dried, and then examined under an optical microscope. Following the optical analysis, the coupons were then placed back into the test environment for an additional 3 h, an additional 8 h, an additional 24 h, and another 24 h; the samples were removed, cleaned, and examined after each time period. These times allowed for a longitudinal study to follow pit growth and surface changes over time. Optical microscopy with an inverted light was used to take multiple images of the resulting corrosion surface at 10x magnification (Axiovert 200M Mat, Carl Zeiss Imaging Solutions, Thornwood, NY). The 10x magnification was used to pictorially show the changes over the six cycles.

Chemomechanical Testing

Because the strain gauges could not be hooked up to the computer to monitor the change in load, due to stress relaxation, during testing, the stress relaxation data points were interpolated at the given interval times following tension testing. As with the optical microscopy set-up, specimens were pinned in the cradles, coated with beeswax, pulled to approximately 80% yield stress, and recoated with beeswax where needed. The specimens were placed in either the immersion environment or the salt spray environment for 1 h, 4 h, 12 h, 36 h, or 60 h. These specimens were not the same specimens as those used for optical microscopy. After the assigned time, the specimens were removed from the environment and from the fixture, cleaned with deionized water to remove excess salt, and dried in a desiccator. 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 collected for three specimens, for a total of six samples per time. 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 to interpolate over 2000 strain values to get the most accurate curve. Since the applied strain for the relaxation tests was in the elastic regime of the AZ61 stress-strain relationship, backing out the stress at the given strain after reloading should theoretically give an accurate stress picture at the time.

Results

Corrosion stress relaxation tests were carried out for 60 hours on the AZ61 Mg alloy specimens. Figure 3 shows the normalized time-dependent stress ($\sigma(t)/\sigma_0$) decrease for the 60 hour testing matrix. $\sigma(t)$ is the current relaxation stress and σ_0 (t=0) is the initial value of stress. This represents the stress relaxation relationship for the salt spray environment and immersion environment specimens. The salt spray environment shows an approximate reduction of static strength of less than 1% over the 60 hours. In the salt spray environment, the reduction in stress is not as significant as in the immersion environment. The immersion environment has a stronger deleterious effect immediately. After one hour, notice that there is approximately an 8% reduction of static strength, which is more than the entire salt spray decrease. After the 60 hours, the specimens exposed to the immersion environment had a 20% reduction of static strength.
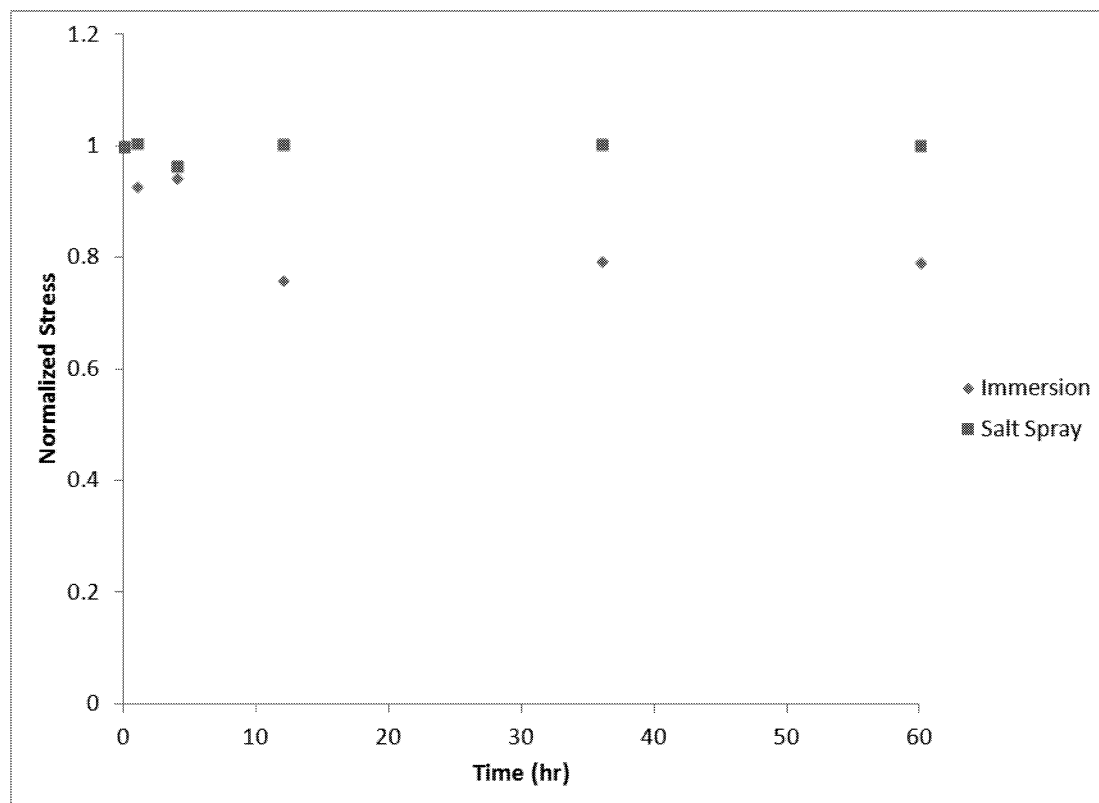

Figure 3. Normalized stress relaxation of AZ61 Mg alloy specimens at room temperature in a 3.5 wt.% NaCl salt spray fog environment and a 3.5 wt.% NaCl immersion environment. The constant strain value was approximately 80% of the tensile yield strength.

Following the corrosion-stress relaxation environmental testing, monotonic tensile tests were performed. Figure 4 illustrates the average stress-strain relationship curves for the salt spray (Figure 4a) and immersion (Figure 4b) environments of AZ61. Notice that the salt spray environment had a minimal effect on the tensile yield strength and elongation to failure. The difference between the curves was less than 5% for all averaged curves. On the other hand, a noticeable reduction of both tensile yield strength and elongation to failure is illustrated in the immersion environment stress-strain plots.

pits. Because the oxide layer was continuously removed, the samples would lose some thickness, leading to a decrease in surface area. This decrease in surface area leads to a decrease in the stress that the samples can withstand. When this reduction in surface area is combined with stress concentration points caused by the pits, the immersion samples would gradually see a reduction in the static stress that they could withstand. The salt spray environment, on the other hand, only has chloride ions during one phase of the cycle, which means pitting can only initiate during the salt spray cycle. Water is present in two of the
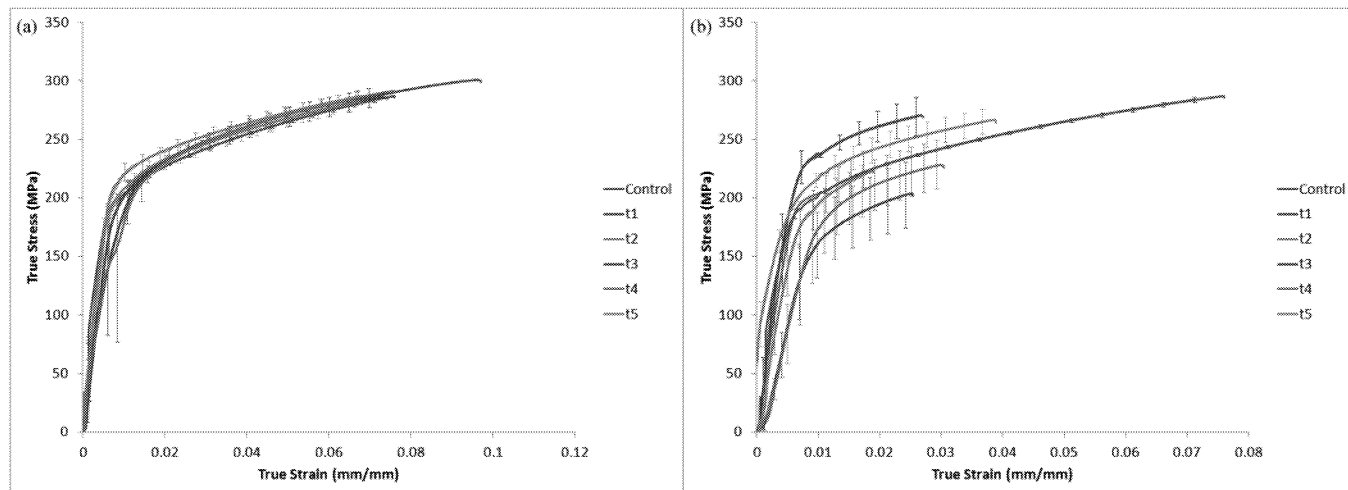

Figure 4. Monotonic tensile true stress-true strain response of the AZ61 Mg alloy following corrosion stress relaxation testing in the (a) salt spray and (b) immersion environments at t_1= 1 h, t_2= 4 h, t_3= 12 h, t_4= 36 h, and t_5= 60 h. All tests were run at room temperature and strain rate of 0.001/s.

Discussion

Stress relaxation of a material is typically defined as the decrease in stress under constant strain and constant temperature. For Mg, this is primarily due to slip in the basal plane [32]. These slips lead to an increase in microstructure dislocation density, which ultimately leads to a decrease in the stress needed to hold a material at a given strain. These dislocations within the material relieve inner stresses caused by plastic microdeformations [15]. A corrosive environment, whether that is the salt spray or immersion environment, increases the relaxation rate, as the water and chloride ions react with the metal. The introduction of pits, caused by chloride ions, and the general removal of metal, caused by water, ultimately decrease the stress the material is able to withstand. Figure 3 shows the normalized stress relaxation of the specimens versus time for the salt spray and immersion environments. In the immersion environment, a reduction of static stress over time was observed, while the static stress remained approximately the same in the salt spray environment. After 60 hours, the applied stress on the immersion samples was approximately 80% of the initial stress, while the applied stress on the salt spray samples was approximately 99% of the initial stress. This indicates that the immersion environment had a much more deleterious effect as compared to the salt spray environment, resulting in larger stress relaxation effects.

The difference between the changes in stress relaxation that the samples are able to withstand is entirely a function of the environment. The immersion environment is continuously exposed to water and chloride ions, leading to more general corrosion, caused by the removal of the $Mg(OH)_2$ oxide layer, and more pitting corrosion, leading to stress concentration around

the three cycles, meaning general corrosion could occur during the salt spray cycle and the humidity cycle. However, with the drying cycle, general corrosion is stopped and the $Mg(OH)_2$ oxide layer would not dissolve away from the surface as readily. In addition, the $Mg(OH)_2$ oxide layer could convert to a more protective oxide layer during the drying phase. The much slower general corrosion rate, combined with the lower rate of pitting corrosion, leads to a material that does not lose as much material, nor possess as many stress concentration points. Without the decrease in surface area to decrease the stress the material could withstand, and without the stress concentration points, the salt spray samples would not experience as large a decrease in static stress, and therefore would not be as greatly affected by stress relaxation. Since these tests were performed at room temperature, without changes to the material, stress relaxation would not have a major effect on the stress the alloy could withstand.

Figure 4 shows the average stress-strain response for the salt spray (Figure 4a) and immersion (Figure 4b) environments compared to the stress-strain response in air. As was the case with Figure 3, the salt spray environment shows almost no change, while the immersion environment shows a decrease in the stress-strain curves over time. Both environments experienced a change in the elongation to failure, indicating the formation of pits that would concentrate the stress, leading to failure at a point sooner than the air samples. The pits caused by the chloride ions would lead to points of stress concentration. These stress concentration points would allow for multiple cracks to begin forming that would then lead to the material failing faster as the cracks coalesce. For the salt spray environment, the stress required to break the samples would still be the same, as the material itself is unchanged, but the pits caused the material to fail

at a shorter point. For the immersion environment, the stress also changed, as the longer the samples were exposed to water, the more surface area was removed, leading to a decrease in stress. There are large error bars for all of the immersion samples as well, indicating that the immersion environment affected the samples differently, as corrosion itself is unpredictable. While the samples were cut from the same extruded rail, different defects with the extrusion process, as well as slightly different chemical compositions within the rail could all affect the corrosion processes differently.

Because of the applied load, there is also a chance that some of the pits were not actually pits, but instead were surface defects that were opened by the applied load of 80% of the yield stress. These small openings would then be a location for the concentration of chloride ions for pitting and water collection for general corrosion. Because the specimens exposed to the immersion environment were continuously exposed to both the chloride ions and water, these small openings could gradually get larger, due to both the basal plane slip relieving inner stresses and the constant corrosive attack. As these openings become larger, the amount of surface area lost increases, as does the stress concentration. Both of these increases lead to a decrease in the tolerance of applied stress. These same openings are likely present on the salt spray specimens, and both chloride ions and water can collect within the openings during the salt spray and humidity cycles. However, there is not a continuous corrosive attack removing metal from the surface in the salt spray environment, so the inner stresses are not relieved and the basal plane slip does not have the same effect as it does for the immersion environment. The presence of these openings, as with the presences of pits, would allow for shorter elongation to failure rates than specimens without the openings or pits. These openings and pits both concentrate stress. With this stress concentration, the failure at these locations occurs faster than the failure within the metal without these stress concentration points, leading to a reduction in elongation to failure.

For both environments, there is also a chance that hydrogen embrittlement occurred, as hydrogen ions are produced during the corrosion reactions; these ions could then diffuse into the metal, prior to becoming hydrogen gas, leading to a reduction in stress [33]. Stress corrosion cracking (SCC) is described as an anodic dissolution assisted cracking which is accompanied by a hydrogen reduction reaction [33]. Since AZXX magnesium alloys are known to be prone to SCC [22], the development of brittle hydrides within the matrix is not unexpected and would result in a decrease in the elongation to failure. The development of SCC, especially from pit nucleation and growth, would not be unexpected, as magnesium alloys are known to be susceptible to environmentally-assisted failure, where pitting corrosion and hydrogen ions induced cleavage cracks lead to failure [34-35].

Conclusions

Corrosion with stress-relaxation studies were performed on AZ61 in a cyclical salt spray environment and an immersion environment over 60 hours. The immersion environment was more detrimental, with a reduction seen in the normalized stress, elongation to failure, and the stress-strain curves. The deleterious effect of the immersion environment was caused by the continuous presence of water and chloride ions. The continuous presence of water allowed for general corrosion, which reduced the surface area of the specimens, reducing the ability of the material to withstand stress. The presence of water also allowed

for the development of hydrogen ions, which could diffuse into the magnesium alloy and lead to hydrogen embrittlement, thereby reducing both the stress and the elongation to failure. The continuous presence of chloride ions induced pitting that would lead to stress concentration points. The applied load would provide a location for additional openings in the surface that would allow for a higher degree of attack by the chloride ions and water. In addition, as the corrosive attack continued and the applied load remained, basal plane slip could occur, with the inner stresses relieved at these openings. The combination of the reduction in surface area, stress concentration points caused by pitting and open defects, and the reduction of stress within the metal as the basal plane slips, would reduce the stress the material was able to withstand. In addition, because of the continuous attack of the chloride ions, more pits are present, so more cracks develop, which lead to a reduction in the elongation to failure.

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