The Influence of Hydrogen on the Stress-Corrosion Cracking of Low-Strength Al-Mg Alloys

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There is growing evidence for hydrogen uptake in aluminum alloys and its contribution to the crack growth of high-strength aluminum alloys, but less evidence for low-strength alloys. This paper summarizes the evidence for hydrogen uptake in a low-strength alloy, AA5083, and its contribution to the stress-corrosion cracking of this alloy. A key factor is the anodic dissolution of grain boundary β *phase* (Al₃Mg₂) and *the associated hydrogen reduction that accompanies this dissolution.*

INTRODUCTION

Evidence for complementary or competing roles of anodic dissolution and hydrogen-induced crack growth is prevalent in aluminum alloys. The dissolution of active intermetallic phases or grain boundary segegants, and the effects of copper enriched around or in particles on localized galvanic corrosion cells are examples of anodic dissolution dominating the crack-growth process. However, hydrogen has been implicated in the stress-corrosion cracking (SCC) of high-strength 7XXX aluminum alloys.¹⁻³ In lower-strength alloys, such as the 5XXX series, there is more evidence that crack growth results from a complementary role of anodic dissolution and hydrogen-induced crack growth.4 The yield strength of 7XXX alloys can be in the 500 MPa to 600 MPa range while AA5083 has a yield strength in the range of 200 MPa to 250 MPa.

There are a number of observations $4-11$ demonstrating that the β phase (Al₃Mg₂) contributes to the SCC of Al-Mg alloys. The development of higher-strength Al-Mg alloys by addition of greater magnesium concentrations has been limited because of observations⁵⁻⁷ that alloys containing more than 3.5% magnesium are susceptible to SCC.

This susceptibility is associated with increased grain boundary precipitation of β phase, with the greatest susceptibility associated with what has been described as "continuous" precipitation of β phase along the grain boundary. However, SCC occurs when the $β$ phase exists as discrete particles along the grain boundary,⁴ so there must be a mechanism by which cracks propagate between particles. Also, many of the observations of a continuous layer of β phase were made with optical metallography of etched surfaces⁷ while transmission electron micrographs of aluminum alloys with <7% magnesium show discrete particles with extensive strain fields.4,8,12,13 These strain fields can cause individual particles to appear continuous if they are closely spaced. Clearly, with increasing magnesium concentration in the alloy and increasing aging time, the β phase particle spacing decreases. Examples of continuous layers of the β phase along grain boundaries as observed by transmission electron microscopy (TEM) are given by Yukawa et al.14 for an Al-9% Mg alloy aged for

100 h at 150°C and by L.I. Kaigorodova¹⁵ for an Al-11% Mg alloy aged for two years at 150°C to 200°C.

HYDROGEN EFFECT IN CRACK GROWTH OF Al-Mg

Hydrogen Permeability

Scully et al.¹⁶ has reviewed the available data on hydrogen solubility and permeability in aluminum and some of its alloys. Their review shows tremendous variability in the available data. However, hydrogen is very insoluble in aluminum at 25°C and 1 atm pressure with values ranging from 10^{-17} to 10^{-11} atom fraction. They also concluded from data for aluminum alloys that lithium and magnesium alloying additions increased the solubility of hydrogen in aluminum because of their chemical affinity for hydrogen. A summary of the hydrogen diffusivity in aluminum also revealed a wide range in values. However, if it is assumed that the presence of aluminum oxide (Al_2O_3) on the surface is likely under all these tests, the fastest diffusivity is expected

to be that closest to bulk diffusivity in aluminum because this likely results from material with a defective or the thinnest oxide film. There are several studies that resulted in diffusion coefficients at 25° C of about 10^{-7} cm²/s for aluminum. The diffusivity of hydrogen was always less in aluminum alloys. However, no data was presented for Al-Mg alloys.

Danielson¹⁷ recently utilized a Devanathan-Stachurski cell to measure the hydrogen permeability through AA 5083 in the solution treated (ST) and quenched (Q) and ST, Q, and aged for 100 h at 175°C condition (Figure 1). This electrochemical permeation experiment is more typical of the conditions of the film-free surface at the tip of a growing stress-corrosion crack compared to the data reported by Scully et al.¹⁶ He found an average hydrogen diffusivity of 3 x 10⁻⁶ cm²/s and a minimum in the permeation rate of hydrogen at the open circuit potential, with increases at both anodic and cathodic potentials. The observation of an increase in the hydrogen permeation rate at anodic potentials is consistent with that reported by Gest and Troiano¹⁸ and shown in Figure 2. The hydrogen permeations results of Danielson¹⁷ and Gest and Troiano¹⁸ are fundamentally similar, but do exhibit some differences in the magnitude of the permeation current. Gest and Troiano¹⁸ reported a ratio of 3:1 between the current at open circuit and -2.0 V (SCE) while Danielson¹⁷ reported only a ratio of 1.5:1 for the same conditions. Also, Gest and Troiano¹⁸ reported a ratio of 4:1 between the current at the open circuit potential and at zero volts (SCE) while Danielson's17 results show a ratio of only 1.7:1. Even though there are quantitative differences between these results, they both demonstrate that hydrogen permeation increases with both positive and negative potentials relative to the open-circuit potential. The reason for this increase at anodic potentials has not been determined; however, in magnesium alloys the rate and amount of hydrogen evolution increases over a small potential range that are noble relative to the open-circuit potential. This effect, termed the negative difference effect by Stampella et al.,¹⁹ could also occur with Al-Mg alloys and contribute to an increased hydrogen permeation rate. Also, a more defective passive film at potentials noble to the open circuit could contribute to an increased hydrogen permeation rate.

Hydrogen Uptake during Corrosion and Stress Corrosion

There have been a number of observations of hydrogen uptake during corrosion and stress-corrosion testing as measured by thermal desorption following exposure. While these observations are less quantifiable than permeation measurements, they do provide direct evidence of hydrogen uptake during specific corrosion conditions. Several methods have been used to monitor hydrogen uptake during corrosion including thermal desorption, TEM of bubbles, and resistivity change. E. Charitidou et al.20 and Haidemenopoulos et al.21 measured the thermal desorption of hydrogen from 2024 aluminum that had been exposed to the exfoliation corrosion solution according to ASTM G 34–90. This solution is an aggressive solution for aluminum alloys because of the low pH (0.3) and high NaCl concentration (4 M). Charitidou et al.²⁰ found that the alloy had absorbed over 1,200 wt ppm after exposure for 40 h following thermal desorption at 600°C but only about 30 wt ppm was released at 100°C. Haidemenopoulus et al.21 measured a hydrogen release corresponding to 90 wt ppm following 216 h exposure to the ASTM G34–90 solution when the hydrogen extraction was done at 100°C. These two results are very similar considering the longer exposure time in the latter measurement. The hydrogen uptake during these tests is significantly greater than that expected in a 3.5% NaCl solution because the G34 solution is extremely aggressive.

The observation of bubbles in alumi-

num and aluminum alloys exposed to water vapor is an indirect method of evaluating hydrogen uptake.22–24 Scamans and Rehal²² found bubbles that they identified as hydrogen bubbles in pure aluminum and aluminum alloys. The authors do not directly measure hydrogen in these bubbles but seem to infer that they are hydrogen filled based on the reaction of aluminum with $H₂O$ to produce hydrogen. In an Al-Mg alloy, they noted bubbles on grain boundaries and dislocations following only 10 min. exposure to water vapor at 70°C. Alani and Swann²⁴ also observed bubbles in Al-Zn-Mg alloys exposed to water vapor at 80°C. They proposed that the bubbles were the result of the precipitation of molecular hydrogen and that the cracks observed to emanate from the bubbles resulted from the pressure in the bubbles. However, they also proposed that it was the atomic hydrogen dissolved along the grain boundaries that was most embrittling. Scully and Young²⁵ evaluated the kinetics of crack growth of a low-copper AA7050 in a 90% relative humidity environment and concluded that crack growth was controlled by hydrogen-environmentassisted cracking over temperatures of 25°C to 90°C.

Hydrogen in solution induces a significant change in the resistivity in aluminum. This property was utilized by Azofeifa et al.²⁶ to measure the in-situ hydrogen-uptake kinetics of aluminum thin foils. The palladiumcoated aluminum foils were exposed to hydrogen gas at a pressure of 4.0 kPa at a temperature of 22°C. The change

Figure 3: The potential drop expressed as crack length for Al-7% Mg alloy without an applied load.

in resistance, reported as $(R-R_0)/R_0$, showed a saturation ranging from 0.2% to 0.9% with a hydrogen gas pressure of about 1.0 kPa, depending on the palladium coating thickness. R_{o} is the resistance without the presence of hydrogen gas and R is the resistance in hydrogen gas. A similar change in resistivity was also observed during a determination of the baseline potential drop on an Al-7% Mg compact tension sample prior to crack-growth testing.²⁷ The technique used for this test was the same as that reported by Jones et al.4 for SCC measurements of AA5083 aluminum but with only a very small load applied to stabilize the sample. The applied stress intensity was 1.5 MPa $m^{1/2}$ and the sample had not been precracked. The potential drop is shown as a function of time in Figure 3 for this alloy in a solution of 3.5% NaCl at its open-circuit potential. The potential drop shows a marked increase to about 100 h, after which the solution that resulted in a high humidity in the cell was drained from the test cell. The removal of the liquid environment that maintained the sample temperature resulted in a sharp decrease in the potential drop that is the result of a temperature change of the sample. However, the potential drop continued to rise slowly, although less so than with the liquid environment, until the cell was completely opened to air, at which time the rise in the potential drop ceased. The sharp drop in potential at 166 h when the cell was opened is thought to be due to the temperature change in the sample as the cell is opened. The potential rises and then continues to decrease for about 10 h, after which time the potential stabilizes. There was no evidence of crack growth or intergranular corrosion to account for the potential drop.

IMPLICATIONS OF HYDROGEN UPTAKE ON STRESS-CORROSION **CRACKING**

Jones et al.⁴ has demonstrated that the intergranular stress corrosion cracking (IGSCC) velocity increases with increasing precipitation of discrete β phase particles on the grain boundary. Many others^{5–11} have also reported that the precipitation of the β phase accelerated SCC growth so there remains no question that the presence of the β phase is a factor in the SCC of AA5083. However, the mechanism by which cracks propagate between the β phase particles and the specific role of the β phase remain open questions.

Jones, et al.⁴ also showed that the β phase corrosion rate is $10⁻² A/cm²$ when polarized to the open-circuit potential of AA 5083 in a solution of 3.5 % NaCl, as shown in Figure 4. The grain boundary particles at the tip of a crack would experience a similar polarization and corrosion rate. However, even with this very high corrosion rate, which could sustain a significant crack-growth rate, the rate-limiting step in the kinetics of cracking is the crack propagation along the grain boundary between the particles. Jones, et al.⁴ also showed that the simultaneous hydrogen-reduction rate on the β phase was $10³$ A/cm². This hydrogen reduction provides a large concentration of hydrogen at the tip of a growing crack. Therefore, the options for crack advance are as follows: anodic dissolution of the β phase and interconnecting grain boundary, anodic dissolution of the β phase and hydrogen-induced crack growth of the interconnecting grain boundary, or hydrogen-induced crack growth around or through the β phase and along the interconnecting grain boundary.

Many4,28,29 have shown that magnesium segregates to the grain boundaries of Al-Mg alloys. The role that this magnesium segregation plays in crack growth along the interconnecting grain boundaries between the β phase is uncertain. Windisch et al.³⁰ found that the corrosion rate of aluminum foils implanted with magnesium showed enhanced corrosion rates relative to high-purity aluminum when tested at a pH of 10 where the passive film on aluminum is unstable. A pH of 3 to 4 is expected at the tip of a stress corrosion crack in aluminum in a solution of neutral pH, 3.5% NaCl. However, the high-pH tests indicate that a magnesiumenriched grain boundary could experience enhanced crack-growth rates if the passive film is ruptured.

There is also the possibility that magnesium segregation to grain boundaries promotes hydrogen-induced crack growth. Scamans²⁸ reported that magnesium segregation promotes hydrogen absorption while Song et al.29 using a quasichemical method concluded that both magnesium and hydrogen lower the work of grain boundary fracture in aluminum. This analysis was supported by SCC results showing a linear increase in the Stage II crack velocity with increasing grain boundary magnesium concentration. Viswanadham et al.31 also concluded that magnesium and hydrogen form a complex that contributes to the hydrogen concentration in the grain boundaries and on hydrogen induced crack growth. This is consistent with the conclusion by Scully et al.16 that magnesium increases the solubility of hydrogen in aluminum because of the chemical affinity between magnesium and hydrogen in aluminum.

Time to failure measurements as a function of applied stress intensity have been reported for AA5083 by Pickens et al.8 The alloy had been aged for 72 h at 150°C to form a semi-continuous layer of β phase along the grain boundaries. There was a clear dependence on loading mode and addition of arsenic to the solution on the time to failure. Arsenic is a hydrogen recombination poison that promotes enhanced hydrogen uptake. Mode I loading resulted in substantially reduced times to failure relative to Mode III loading. The presence of a hydrostatic stress in the Mode I loading

test and absence of a hydrostatic stress in Mode III was used to suggest that hydrogen played a role in the SCC of this alloy. However, cracking did occur in Mode III loading so it is clear that either hydrogen is not necessary or that hydrogen-induced cracking can occur in the absence of a hydrostatic stress to help hydrogen concentrate at the crack tip. Another factor is that shear cracks growing in Mode III have no opening mode so solution access to the crack tip is greatly limited. This factor alone is sufficient to reduce the crack velocity and increase the time to failure.

The addition of 10 ppm of arsenic to the sodium-chloride/chromate solution for the Mode I test reduced the time to failure.8 The authors concluded that, since arsenic functions as a hydrogen recombinant poison and should enhance hydrogen uptake, this observation provides further evidence that hydrogen is a factor in the SCC of AA5083. However, it is unfortunate that the authors did not also add arsenic to the Mode III tests to confirm this conclusion. Also, the authors did not comment on the crack angle in the Mode III tests. On brass samples loaded in Mode III using torsion loading and tested in a tarnishing ammoniacal solution, Qiao and Luo³² noted that the cracks formed at an angle of 45 degrees to the shear stress and sample axis. This direction in a torsion test is the maximum tensile loading direction such that these cracks were actually propagating with Mode I loading.

Crack-growth-rate measurements for AA5083 in the ST and Q condition and ST, Q and aged for 1 h at 175°C have been reported by Jones et al.⁴ These results are shown in Figures 5a and 5b, where there is a clear increase in the crack velocity at potentials positive to the open circuit but not negative. These results are most easily explained as supporting anodic dissolution; however, there is virtually no β phase at the grain boundaries for either thermal treatment, so that anodic dissolution of magnesium- and copper-enriched grain boundaries would be necessary for crack growth. As discussed previously, the results of Windisch et al.30 suggest that both magnesium and copper enriched in the grain boundaries of aluminum would increase the anodicdissolution rate. The permeation data by Gest and Troiano¹⁷ and Danielson¹⁸ supports a hydrogen effect on crack growth at anodic potentials in addition to the anodic dissolution possibility. However, an increase in crack growth would also be expected at cathodic potentials, which was not observed. This could result from the passive film being sufficiently stable that hydrogen uptake is very slow; however, these tests were conducted, within the Stage II crack growth regime, at a stress intensity of 25 MPa m^{1/2}. At this stress intensity, crack-tip deformation is expected to induce film rupture and, therefore, paths for hydrogen uptake without diffusing through the passive film.

Elucidating a definitive mechanism of crack growth is difficult given some of the uncertainties associated with the crack-growth data. Options for a crackgrowth mechanism are: all dissolution;

partial dissolution of the β phase with hydrogen-induced crack growth around or through the particle and along the connecting grain boundary; total dissolution of the β-phase particle and hydrogen-induced cracking between the particles; all hydrogen-induced crack growth. The β phase has an opencircuit potential 0.45 V negative to that of AA5083. Therefore, the β-phase particles cathodically protect the matrix such that a crack intersecting a particle will not progress until the particle dissolves or hydrogen induces cracking around or through it. Crack advance will not occur by anodic dissolution between the particles as long as the β phase remains in contact with the matrix. Efforts to detect the presence or absence of the β phase at SCC tips are in progress. However, this phase is difficult to image on a flat surface such as the side of a SCC sample with secondary or backscattered electrons in a scanning electron microscope. Jones et al.33 examined SCC fracture surfaces but found no evidence for the β phase. At similar imaging conditions, they are observed on fracture surfaces not exposed to the NaCl solution. However, these observations are not convincing since the surfaces were not at the crack tip but from the crack wall following considerable exposure to the solution and, therefore, there was considerable opportunity for the particles to dissolve after the crack advanced. Based on the electrochemistry of the β phase in contact with the aluminum and the many observations of hydrogen uptake in aluminum, the mechanism that appears

to be the most likely crack advance mechanism is total dissolution of the β-phase particle and hydrogen-induced cracking between the particles. Tanguy et al.34 observed intergranular fracture in hydrogen-charged Al-5% Mg without grain boundary β-phase precipitates except at triple points. This effect was reversible following desorption of the hydrogen. These results further support the conclusion that crack growth between the β-phase particles is induced by hydrogen.

CONCLUSION

There is considerable evidence that hydrogen is generated at the tip of a crack growing along grain boundaries of AA5083 that contain precipitated β phase. The β phase is polarized to the open circuit of aluminum, which places it beyond its breakdown potential. About 10% of the total current was shown to be from hydrogen reduction. There is also considerable evidence that hydrogen uptake occurs during corrosion of aluminum alloys including AA5083 as shown by thermal desorption and resistivity change. However, even with the considerable evidence it is not possible to conclude that hydrogeninduced crack growth is the dominant mechanism in AA5083. The following possibilities must be considered viable crack-growth mechanisms:

- Anodic dissolution accounts for crack growth through or around the β-phase particles and along the magnesium- and copper-enriched grain boundary between the particles
- Anodic dissolution accounts for crack growth through or around the β-phase particles but crack growth between the particles is the result of hydrogen-induced crack growth
- Hydrogen-induced crack growth is responsible for crack growth through or around the $β$ phase particles and along the grain boundary between the particles.

Images of SCC tips would help elucidate the operative mechanism since the presence of β-phase particles along the crack wall behind the crack tip would eliminate the first option because the β phase would cathodically protect the grain boundary between the particles. The absence of the β phase behind the crack tip would indicate that this cathodic protection is removed and that anodic dissolution along the grain boundary between the particles was a possibility. Further work is in progress to image the crack tip particle interactions to help answer this question.

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