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# Magnesium stress corrosion cracking

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**Abstract:** The significant positive green environment influence of magnesium alloy usage in transport could be compromised by catastrophic fast fracture caused by stress corrosion cracking (SCC). Transgranular stress corrosion cracking (TGSCC) of AZ91 was evaluated using the linearly increasing stress test (LIST) and the constant extension rate test (CERT). The TGSCC threshold stress was 55–75 MPa in distilled water and in 5 g/L NaCl. The TGSCC velocity was  $7 \times 10^{-10} - 5 \times 10^{-9}$  m/s. A delayed hydride-cracking (DHC) model for TGSCC was implemented using a finite element script in MATLAB and the model predictions were compared with experiment. A key outcome is that, during steady state TGSCC propagation, a high dynamic hydrogen concentration is expected to build up behind the crack tip. A number of recommendations are given for preventing SCC of Mg alloys in service. One of the most important recommendations might be that the total stress in service (i.e. the stress from the service loading + the fabrication stress + the residual stress) should be below a threshold level, which, in the absence of other data, could be (conservatively) estimated to be about 50% of the tensile yield strength.

Key words: stress corrosion cracking; linearly increasing stress test; LIST; CERT; hydrogen

## **1** Introduction

Magnesium offers a high potential as a lightmass structural material for use in transport, and consequently can have significant green environment influence. Consequently, magnesium usage is growing rapidly. The rapid increase is due to the lightmass of magnesium alloys, which provides considerable mass saving potential in automobile and transport industries, although corrosion is an issue[1-17]. Its good processing capability, particularly its ability to be die cast into large thin sections, can also lead to considerable savings. However, there is a significant risk of SCC causing catastrophic fracture of critical Mg components that are exposed to road spray. The total risk increases with the current rapid increase in Mg usage in auto applications. Mg SCC can occur in any application when a stressed Mg component is subject to wet conditions. Catastrophic failure is expected from SCC when the environment (e.g. road splash) decreases the SCC initiation stress to below the operating stress.

An overview of Mg SCC is emerging from our critical literature review[18] and current research[19–25] for existing Mg alloys and alloys being developed. Our recent critical review of SCC[18] indicated that many common Mg alloys show SCC in common environments such as distilled water and dilute chloride solutions and that the threshold stress for SCC is commonly about half the yield stress. There can be, nevertheless, significant SCC differences between alloys[18,21–22]. Intergranular stress corrosion cracking (IGSCC) is not the subject of our research because Mg IGSCC is well understood. IGSCC in Mg alloys is typically caused[23] by corrosion associated with a continuous second phase along the grain boundaries, a microstructure typical of cast creep-resistant Mg alloys.

Transgranular stress corrosion cracking (TGSCC) occurs through the Mg matrix. TGSCC is the intrinsic form of SCC and TGSCC can occur in alloys resistant to IGSCC. TGSCC is the focus of our research. The main features of Mg TGSCC have been established[18]. PUGH and co-workers[26–30] provided convincing evidence for a brittle cleavage mechanism involving

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hydrogen. Particularly noteworthy were the stepped and facetted interlocking fracture surfaces. The slow strain rate tests[1-2] indicated a mechanism involving strain induced breaking of a crack tip film leading to corrosion and hydrogen production, with crack advance due to hydrogen. MAKAR et al[1] confirmed the fractography, the importance of strain rate and hydrogen, but they proposed a brittle hydride model. Thus, there is agreement that hydrogen is a key part of Mg TGSCC crack propagation but considerable disagreement concerning the details. Concerning the environment, the mechanistic hypothesis[18] is that TGSCC occurs for conditions leading to the local breakdown of a partially protective surface film. Film breakdown can be caused by pitting by chloride ions or by the applied stress. The surface film also inhibits hydrogen ingress into the metallic Mg alloy, the hydrogen being produced as part of the Mg corrosion reaction.

In summary, mechanistic understanding indicates that hydrogen plays a key role[18] in Mg TGSCC. It is most likely[18] that TGSCC involves repeated cycles of (1) hydrogen diffusion to the stressed region ahead of the crack tip and (2) stress corrosion crack advance through hydrogen influenced process zone. а Research needs[18–19, 21 - 22] are for а fundamental understanding to overcome Mg TGSCC, which is the inherent SCC response.

# 2 Characterization of TGSCC

SCC tests[19–21] were performed using high purity AZ91 (>90.11%Mg, 8.99%Al, 0.78%Zn, 0.21%Mn, mass fraction) using the linearly increasing stress test (LIST) or the constant extension rate test (CERT). In the LIST apparatus[34-42] (Fig.1) the specimen is attached to one end of a lever arm. To the opposite end of the arm a known mass is attached such that the tensile load applied to the specimen increases linearly as the distance between the fulcrum and the mass is increased by means of a screw thread and synchronous motor. The CERT apparatus (Fig.2) maintains a constant extension rate by means of an open-loop control system; the average specimen elongation is measured by two high-resolution LVDTs in parallel with the specimen whilst a geared synchronous motor increases the elongation. SCC susceptibility was characterised according to the threshold stress, which was determined using the DC potential drop (DCPD) method[43-47]. The stress corrosion crack velocity,  $v_c$ , was calculated for both LIST and CERT according to  $v_c = a/l$ , where *a* is the length of the stress corrosion crack at the end of the test as estimated from the fracture surface and t is the time for stress corrosion crack growth from crack initiation (identified using the DCPD method) to the end of the

test.



Fig.1 Schematic illustration of LIST apparatus



Fig.2 Schematic illustration of CERT apparatus

Figs.3 and 4[19–21] show typical stress—strain behaviour and DCPD measurements for CERT. There was a considerable reduction in the UTS relative to air for all of the samples exposed to distilled water. Moreover, the UTS decreased with decreasing strain rate. The threshold stress corresponded to the point where the DCPD curve became non-linear (Fig.4). The SCC threshold stress was 55–75 MPa in distilled water and 5



Fig.3 Stress—apparent strain curves for AZ91 in distilled water and air[19–21]



Fig.4 DCPD results for AZ91 in distilled water[19-21]

g/L NaCl. The measured velocity was in the range of  $7 \times 10^{-10}$ -5×10<sup>-9</sup> m/s. These values are compared with literature values in Table 1.

# 3 Delayed hydride cracking model

A DHC model[24] was proposed based on transient hydrogen diffusion towards and, when the H solvus concentration is exceeded, hydride precipitates in the region ahead of the crack tip. The model evaluates  $v_c$ based on the time-to-reach the critical hydride dimension during stage-2 TGSCC crack growth. A finite element script was developed in MATLAB to solve for the transient H distribution and hydride precipitation. A

Table 1 Measured stress corrosion crack velocity values

typical result for the DHC model is presented in Fig.5. It shows contours of equal values of  $\varphi$ . The region bounded by  $\varphi=1$  is comprised completely of hydride, whereas at the next contour,  $\varphi=0.95$ , there is 95 % hydride and 5% Mg metal. In this case, DHC was simulated for  $K_1=K_{1SCC}\approx 6$  MPa·m<sup>1/2</sup>, neglecting the influence of the plastic zone, for a period of time corresponding to  $\varphi=1$  at a distance of about 0.8 m ahead of the crack tip.



**Fig.5** Distribution of hydride volume fraction  $\varphi$  for  $K_1 = 6 \text{ MPa} \cdot \text{m}^{1/2}$  and t = 6.2 s [24]

For most of the simulation (about 4.5 s)  $\varphi < 0$  for the entire domain; the greatest period of time (*t* about 4.5 s) is that which is required for the hydrogen concentration to reach the solvus concentration at the point of maximum stress, then hydride first forms and thereafter the hydride grows rapidly. Fig.6 shows the hydride distribution for various times between (1) complete hydride throughout the first element (at the

| Material   | Environment                           | Load condition        | Crack velocity/ $(m \cdot s^{-1})$     | Ref. |
|------------|---------------------------------------|-----------------------|--|------|
| Mg-7.5Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | -                     | 3×10 <sup>-5</sup>                     | [26] |
| Mg-7.6Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | Constant load         | $6 \times 10^{-6} - 4 \times 10^{-5}$  | [30] |
| Mg-7Al     | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | SSRT                  | $2 \times 10^{-6} - 5 \times 10^{-6}$  | [48] |
| ZK50A-T5   | Distilled H <sub>2</sub> 0            | Constant load         | 3×10 <sup>-9</sup> -8×10 <sup>-9</sup> | [50] |
| ZK50A-T5   | 1.4 m Na <sub>2</sub> SO <sub>4</sub> | Constant load         | 6×10 <sup>-4</sup>                     | [50] |
| ZK50A-T5   | 5 m NaBr                              | Constant load         | $10^{-5}$                              | [50] |
| Mg-7.6Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | Constant load         | $10^{-5}$                              | [50] |
| Mg-8.8Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | SSRT                  | $10^{-7} - 8 \times 10^{-6}$           | [31] |
| Mg-8.8Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | Constant load         | $2 \times 10^{-6} - 3 \times 10^{-5}$  | [31] |
| Mg-8.8Al   | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | Constant strain       | $8 \times 10^{-7} - 2 \times 10^{-5}$  | [31] |
| RSP Mg-1Al | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | SSRT                  | $2 \times 10^{-7} - 10^{-5}$           | [33] |
| RSP Mg-9A1 | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | SSRT                  | $8 \times 10^{-8} - 3 \times 10^{-6}$  | [33] |
| Pure Mg    | NaCl+K <sub>2</sub> CrO <sub>4</sub>  | Const deflection rate | $10^{-8} - 5 \times 10^{-2}$           | [49] |
| AZ91       | Distilled water                       | SSRT                  | $7 \times 10^{-10} - 5 \times 10^{-9}$ | [21] |
| AZ91       | Distilled water                       | LIST                  | 5×10 <sup>-9</sup>                     | [21] |



**Fig.6**  $\varphi$ =1 zone after 6.19, 6.2, 6.21 and 6.22 s for  $K_1$  = 6 MPa·m<sup>1/2</sup>

point of maximum stress) and (2) when  $\varphi=1$  front is 0.8  $\mu$ m ahead of the crack tip (assuming  $K_1 = 6 \text{ MPa/m}^{1/2}$ ).

Assuming a critical hydride size of 0.8 µm for all values of  $K_1$ , the values predicted by the present DHC model for  $v_c$  range from  $4.6 \times 10^{-8}$  m/s to  $3.3 \times 10^{-7}$  m/s. These DHC model predictions are consistent with the lower measured crack velocities reported by previous workers and thus could explain the stress corrosion crack velocity for Mg alloys in distilled water. However, the DHC model as presently formulated does not predict stress corrosion crack velocity values of  $10^{-5}$  m/s as measured e.g. by SPEIDEL et al[50] for the stress corrosion cracking of ZK50 in dilute NaBr solution or by PUGH and co-workers[26–30] for Mg-Al alloys in NaCl + K<sub>2</sub>CrO<sub>4</sub>.

A key outcome of the modeling [24] is that the standard initial condition for DHC models is unlikely to be correct. It was assumed that the region near the crack tip was free of H and that H diffuses from the crack surface only. This assumption, although made in previous DHC models, is valid only for the initial crack propagation step in a "virgin" H-free material and is not valid for steady state TGSCC propagation. In the case of the initial crack propagation in a "virgin" H-free material it is appropriate to choose a low, uniform H concentration as an initial condition. For steady state TGSCC propagation the appropriate hydrogen concentration around the crack tip can be deduced from Fig.5 by moving the point of origin to the right by a distance corresponding to the prior crack advance (about  $0.8 \mu m$ ), such that the new crack tip is in a region with a

hydride volume fracture  $\varphi$ =0.95 rather than a hydrogen concentration of about 0. Fig.6 shows that in a "virgin" material the growth of the region corresponding to  $\varphi$ =1 grew to about 0.8 µm in 0.02 s. Given an initial condition of  $\varphi$ =0.95 at the crack tip, the predicted stress corrosion crack velocity is then

 $v_{\rm c} = 0.8 \ \mu {\rm m}/0.02 \ {\rm s} = 4 \times 10^{-5} \ {\rm m/s}$ 

This estimation shows that the DHC model may be developed to predict a TGSCC crack velocity sufficiently high to explain the stress corrosion crack velocities measured by SPEIDEL et al [50], PUGH et al [26–30], EBTEHAJ et al [31] and MAKAR et al [33].

### 4 Recommendations to avoid SCC

The general principle for SCC prevention is to avoid loading a susceptible alloy above a critical stress during exposure to a SCC producing environment. SCC prevention is a serious task for Mg alloys, since SCC can be produced in distilled water for applied stresses above 50% YS [18]. There is inadequate data, partly because the use of Mg alloys as a structural material is relatively recent and reported service failures are not numerous. Fig.7 schematically illustrates how threshold values can be included in an assessment of structural integrity. In an inert environment, the mechanical limits can be defined by the yield stress,  $\sigma_y$ , and the fracture toughness,  $K_{1C}$ . In a SCC environment, the mechanical limits are reduced to the threshold stress intensity factor,  $K_{1SCC}$ , and the threshold stress,  $\sigma_{SCC}$ , determined on smooth tensile specimens of the same material and in the same environment. These two parameters define an "acceptable region" with respect to immunity against SCC. Depending on the susceptibility of the material/environment combination under investigation, this region can be significantly smaller than the original "acceptable region" derived from tests in air.

There is a general trend in SCC prevention to start with a design that avoids concentration of stresses at the start, or during use, or through increasing susceptibility to different forms of degradation in service, especially localised corrosion, galvanic corrosion and (corrosion) fatigue. SCC is expected to not be an issue for dry atmospheres provided the relative humidity is less than 95% and provided that there are no crevices. For crevices, capillary condensation can cause the formation of a liquid in the crevices at lower values of relative humidity[51]. It would be prudent to fill crevices with a corrosion-inhibited putty. Furthermore, crevices are part of the microstructure of diecast alloys, which might be part of the reason that MILLER[52] measured threshold valued of 40%-50% of YS for AZ91, AM60 and AS41 in distilled water. The total stress in service (stress from the service loading + the fabrication stress + the residual stress) should be below the threshold level which, in the absence of other data, could be estimated to be about 40%–50% of the tensile yield strength.



Fig.7 Mechanical limits of typical system involving an inert and SCC environment[53–54]

# **5** Summary

1) The TGSCC threshold stress is 55-75 MPa for AZ91 in distilled water and 5 g/L NaCl. The measured TGSCC velocity is in the range of  $7 \times 10^{-10}-5 \times 10^{-9}$  m/s.

2) Stress corrosion crack velocities about  $10^{-4}$  m/s, which are typical for Mg alloys in aqueous solutions, cannot be predicted by the DHC model based on the time to reach a critical hydride size for material with a low initial H concentration throughout. Such TGSCC

velocities might be predicted by a DHC model based on the time to reach the critical hydride size in steady state, when a significant hydrogen concentration would have built up at the crack tip.

3) During steady state stress corrosion crack propagation of Mg in aqueous solutions, a high dynamic hydrogen concentration would be expected to build up just behind the crack tip. This may be a feature of all cases of SCC where the crack propagation mechanism is Hydrogen Environment Assisted Cracking (HEAC).

4) The total stress in service (stress from the service loading + the fabrication stress + the residual stress) should be below the threshold level which, in the absence of other data could be estimated to be about 40%-50% of the tensile yield strength.

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