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Environmentally Assisted Cracking of Magnesium Alloys

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Abstract. The propensity of the magnesium alloys AM30 and AZ91 to environmentally assisted cracking, and in particular to hydrogen embrittlement, was assessed in constant extension rate tensile tests on smooth and pre-cracked specimens which were subjected to monotonic loading in corrosive environment. The experimental findings can be rationalized by model approaches: A meso-scale fibre bundle model was employed to simulate the results obtained in tests on smooth AZ91 tensile specimens, assuming a combination of pitting and subsequent hydrogen embrittlement as the underlying failure mechanism. The experiment data as well as the model results revealed the effect of hydrogen embrittlement on crack growth resistance. The model calculations generated fracture surfaces which were in remarkable correspondence with those observed in the experiments, and stress-strain curves similar to the experimental ones, both reflecting the influence of the applied strain rate on hydrogen induced failure. The effect of hydrogen embrittlement on cracking in AM30 was assessed using a fracture mechanics based approach. A cohesive model which accounts for hydrogen enhanced crack extension and which earlier has been successfully applied to HE of steels is currently readjusted to EAC of magnesium.

Introduction

The increased use of magnesium alloys in structural applications, like automotive components, requires a sound knowledge of the susceptibility of these materials to environmental degradation if these alloys are to be used in aqueous environments. While uniform and general corrosion are relatively well controllable, environmentally assisted cracking (EAC), i.e., stress corrosion cracking (SCC) and corrosion fatigue (CF), are potentially dangerous and can result in catastrophic failures [1,2]. In structural applications, problems of EAC and in particular those of hydrogen embrittlement, HE, are likely to be encountered [3]. Hydrogen evolving from the corrosion reaction inside pits can be adsorbed/absorbed by the material and can lead to HE as a specific form of SCC [4,5]. The ongoing research seeks to provide a fundamental understanding of the factors that cause environmental failure of magnesium alloys, and to expand this understanding through experimental evidence. Fracture mechanics based techniques have turned out to be very useful for studying SCC and HE and to rationalize the underlying mechanisms of material degradation caused by the uptake of atomic hydrogen [6]. In this study, constant extension rate tensile (CERT) experiments on smooth and on pre-cracked specimens were used to characterize the susceptibility of Mg alloys to hydrogen embrittlement.

When subjecting magnesium specimens to CERT tests in an aqueous environment, a sequence of film rupture events at the specimen surface leads to the exposure of bare metal to the environment. This gives rise to an electrochemical reaction with the liquid in which magnesium hydroxide and hydrogen are produced according to the following reactions (Eq. 1-3) [7]:

$$2\,Mg \Leftrightarrow 2\,Mg^{+} + 2\,e^{-} \qquad \text{(anodic partial reaction)} \qquad (1)$$

$$2\,H_2O + 2\,e^{-} \Leftrightarrow H_2 + 2\,(OH)^{-} \qquad \text{(cathodic partial reaction)} \qquad (2)$$

with the overall reaction

$$Mg_{(solid)} + 2\,H_2O_{(liquid)} \Leftrightarrow Mg(OH)_{2(solid)} + H_{2(gaseous)} \qquad (3).$$

Most of the hydrogen atoms generated in this reaction recombine at the specimen surface and form hydrogen gas which bubbles up in the test solution. Yet, some atoms are adsorbed at the surface and enter into the bulk of the specimen. Subsequently, these hydrogen atoms diffuse inside the material to the highly stressed regions and raise the hydrogen concentration at these sites.

Although there exists a considerable body of research outlining the phenomenology of EAC of Mg alloys, little consensus can be found in the literature regarding the underlying mechanisms [3,8]. Yet, the development of durable Mg alloys for automobile components requires a profound understanding of these mechanisms. One approach towards gaining this understanding is modelling of SCC and in particular of hydrogen induced cracking of magnesium alloys.

Experimental Details and Modelling Approaches

Constant extension rate tensile tests were conducted at a number of extension rates on unnotched cylindrical tensile specimens of the magnesium cast alloy AZ91 and on pre-cracked compact tension specimens (C(T)) of the alloy AM30. The tests were performed in double distilled water; reference tests were performed in laboratory air [5,6].

In the CERT tests on smooth tensile specimens the waisted gauge sections of these specimens were constantly immersed in the environment and the extensions were measured by a pair of linear variable displacement transducers (LVDTs), which were attached sidewise to the specimen fixtures.

The fracture mechanics based SCC tests on C(T) specimens were performed such that a constant increase in crack mouth opening displacement (CMOD) with time was achieved. Here, the aim was to determine the threshold value of the fracture toughness, K_{ISCC}, and to study crack morphology [8]. The use of an electrical potential drop equipment enabled the identification of crack initiation and growth. To this end, a pulsed DC current of alternating polarity was sent through the specimens, and an increase in the potential drop signal was taken as indication of an increase in crack length [9,10].

To rationalise the results of the tests on smooth specimens, a meso-scale model was applied in which the material under test was represented by a bundle of parallel bars or fibres. Details of this model can be found elsewhere [5]. Previous experience on steel had also shown that a modified cohesive model which takes into account the uptake of atomic hydrogen from the corrosive environment is suited for rationalising the results of fracture mechanics SCC tests [11]. However, this model has so far not become available for HE investigations of magnesium.

Results

Fig. 1a shows stress vs. apparent strain curves measured on AZ91 tensile specimens in distilled water plus one curve which was obtained in laboratory air. The "apparent" strain rates were calculated from the increase of extension measured at the specimen fixtures, and are labelled at the curves. The figure reflects the increasing influence of hydrogen on the specimen failure, i.e., at low strain rates sufficient time exists for the hydrogen to diffuse into the bulk of the material. In Fig. 1b the appearance of an experimentally obtained fracture surface is compared with the result of simulating hydrogen induced failure of a tensile specimen tested under similar conditions. Here, the pictures shows the cross section of the specimen generated by the fibre bundle model and the progress of damage due to the combination of hydrogen embrittlement and increasing strain. Hydrogen emanates from the corrosion reaction at small pits at the edge of the specimen and migrates into the bulk. The pits form following the rupture of the magnesium hydroxide layer on the surface of the specimen due to the applied deformation, giving rise to localised corrosive attack of the magnesium.

With increase in time, the number of pits increases, and hydrogen diffuses from the pits towards the centre of the specimen leading to failure of individual fibres, the strain to failure of which is assumed to be reduced by the hydrogen concentration. The black areas in the lower portion of Fig. 1b characterize pits or broken fibres, while the centre area represents un-cracked ligament, and the areas adjacent to the cracked portions indicate different concentrations of hydrogen in the diffusion front. The same model parameters had also been used to mimic the stress-strain curves which are additionally shown in Fig. 1a.

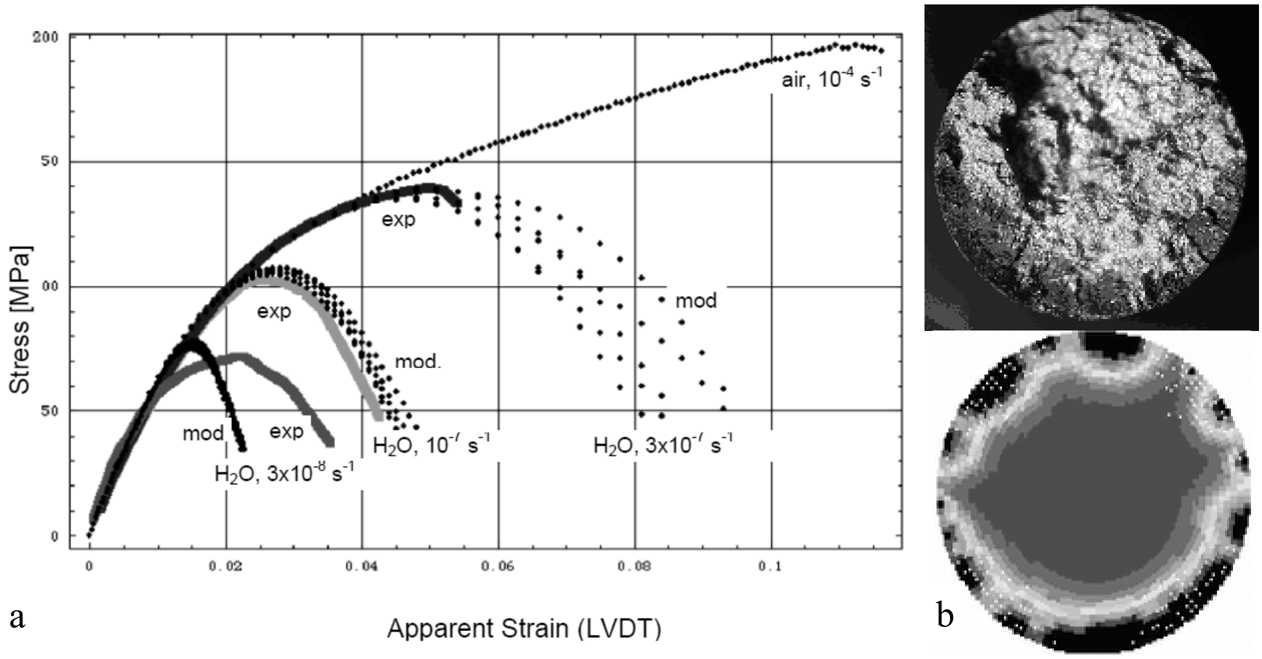

Fig. 1: Stress-strain curves of AZ91 in distilled water:

a: experimental stress vs. "apparent strain" curves measured in CERT tests for three different strain rates ("exp") and sets of curves calculated from the fibre bundle model for the same strain rates ("mod").

b: fracture surface of an AZ91 tensile specimen tested in distilled water, showing areas of hydrogen induced cracking;
top: experimental (OM),
bottom: hydrogen distribution predicted by the fibre bundle model.

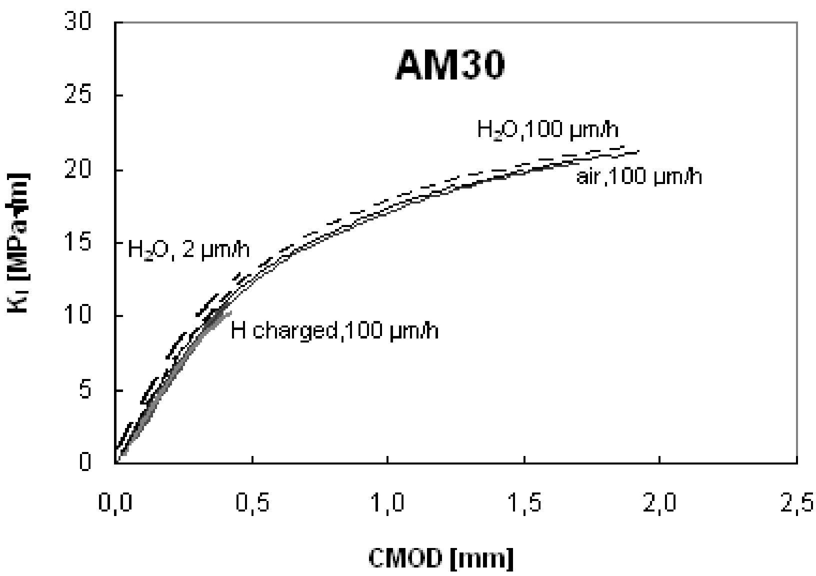

Fig. 2: Curves of the stress intensity factor, K_I, vs. crack mouth opening displacement, CMOD, up to crack initiation, measured at two extension rates (2 µm/h, 100 µm/h) in laboratory air, distilled water and under conditions of electrolytic hydrogen charging (-1.7 V in 0.1 M NaOH solution).

In Fig. 2 results of various fracture mechanics tests on pre-cracked AM30 C(T) specimens are displayed. The tests were conducted at extension rates of 2 µm/h and 100 µm/h, respectively, with the extension being measured as crack mouth opening displacement, CMOD. Values of the stress intensity factor, K_I, are plotted versus the related CMOD values up to the point at which the DCPD signal had indicated crack initiation, i.e., K_{ISCC} was reached. The curves measured at 100 µm/h are similar in air and in distilled water, whereas in the test at 2 µm/h in distilled water cracking had occurred much earlier - after only 0.5 mm increase in CMOD compared to about 2 mm in air - due to hydrogen embrittlement. Similar curves were observed when the material was cathodically charged with hydrogen in-situ during CERT testing at a potential of -1.7 V in 0.1 M NaOH solution.

Summary

The EAC of the magnesium alloys AZ91 and AM30 was studied in CERT tests at low extension rates on smooth and pre-cracked samples in distilled water promoting HE. A modelling approach based on the meso-scale fibre bundle model could reproduce the experimental data obtained on smooth AZ91 tensile specimens. The data obtained in FM based tests on AM30 C(T) specimens clearly demonstrate the embrittling effect of hydrogen on this alloy, too. Although previous experience has shown that in principle such results can be simulated by a modified cohesive model, such a model has for magnesium not become available yet.

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