





# Effect of hydrogen on stress corrosion cracking of magnesium alloy in 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution

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#### **Abstract**

The effect of hydrogen on stress corrosion cracking (SCC) of an as-cast MgAl9Zn1 magnesium alloy was investigated using slow strain rate test (SSRT). The results showed that hydrogen diffused to interior of matrix, enriched and formed hydride at  $\beta$  phase that caused the cracking of  $\beta$  phase. Micro-cracks initiated at  $\beta$  phase and combined into main crack. The main crack propagated by coalescence of the existing micro-cracks ahead. It was indicated that hydrogen embrittlement was the main mechanism for the SCC of MgAl9Zn1 alloy. © 2007 Elsevier B.V. All rights reserved.

Keywords: Magnesium alloys; Stress corrosion cracking; Hydrogen embrittlement

#### 1. Introduction

The stress corrosion cracking (SCC) of magnesium and its alloys was characteristic of transgranular stress corrosion cracking (TGSCC), which might involve hydrogen generated by corrosion reaction diffusing into the interior of magnesium and its alloys. Recently, many critical reviews [1–3] and works [4–7] about the SCC of magnesium and its alloys suggested that delayed hydride cracking (DHC) [6–10] or hydrogen enhanced localized plasticity (HELP) [11] might be responsible for the TGSCC. However, the mechanism for the TGSCC of magnesium and its alloys is still ambiguous.

In our previous work [12], after immersion or cathodic charging, cracks were found to initiate at  $\beta$  phase  $(Mg_{17}Al_{12})$  due to the synergistic effect of hydrogen pressure of the formation of hydrogen gas and expansion stress of the formation of magnesium hydride. Moreover, the process of crack initiation at  $\beta$  phase was also in situ observed using environmental scanning electron microscope (ESEM) on the pre-charged MgAl9Zn1 (AZ91) alloy [13]. It means pure chemical effects caused by hydrogen invasion could result in the crack initiation at the surface layer and interior of matrix. Whether the effects were more evident under load was the major concern.

In present work, the SCC behaviors of AZ91 alloy in various environments were studied. Special attention was paid to the effects of hydrogen produced by corrosion reaction and cathodic charging on the SCC behaviors of AZ91 alloy.

#### 2. Experimental procedure

The material used was an as-cast AZ91 magnesium alloy. Its chemical composition (wt.%) was 8.89 Al, 0.78 Zn, 0.21 Mn, <0.001 Ni, 0.002 Fe, 0.002 Cu and balance Mg. The sample surface was polished using 1000, 2000 grit silicon carbide papers, cleaned with distilled water and acetone, and dried in cool air. Some mechanically polished samples were galvanostatic charged at 27.8 mA/cm<sup>2</sup> for 3 h in 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution (pro-analysis grade, pH 6.1) before slow strain rate test (SSRT). After cathodic charging, the solution was changed.

SSRT was used to investigate the SCC behaviors of AZ91 alloy in deionized water and 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution in contact with air at about 30 °C. The samples was plane plate with the gauge geometry of 25 mm  $\times$  8 mm  $\times$  2.5 mm. The strain rate was  $1\times10^{-6}$  s<sup>-1</sup>. The data for stress, time to fracture and elongation was recorded using computer. Assumed that the deformation over the entire gauge section of samples was uniform, the average specimens elongation was measured by two linear variable displacement transducers (LVDs) attached at two sides of samples. Therefore, the actual strain and actual strain rate, caused by mechanical rupture or SCC, could not be measured using this

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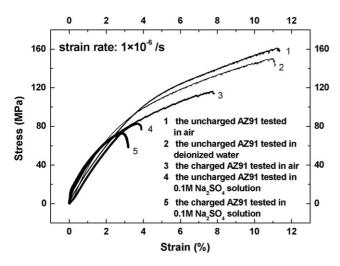


Fig. 1. The apparent stress-strain curves of as-cast AZ91 alloy in different environments.

equipment. The elongation measurement was in agreement with that of Kannan et al. [14].

#### 3. Results

## 3.1. Effect of pre-charging and media on mechanical properties

The apparent stress–strain curves of the uncharged and charged AZ91 alloy in various corrosive mediums are illustrated in Fig. 1. For the specimens tested in air (curves "1" and "3") and Na<sub>2</sub>SO<sub>4</sub> solution (curves "4" and "5"), respectively, the maximum stress and elongation of the charged specimens decreased, and the decrease was more obvious for the charged samples

tested in air. In the meantime, the addition of aggressive anions such as  $SO_4^{2-}$  to the solution also resulted in the deterioration of mechanical properties. The changes and decrease of mechanical properties of AZ91 alloy in different environments are depicted in detail in Tables 1 and 2, respectively.

#### 3.1.1. Effect of pre-charging

For the specimens tested in air, the reduction of area (ROA) and time to failure (TOF) of the charged samples decreased 59% and 43% of those of the uncharged respectively; for the samples tested in  $0.1\,M$  Na<sub>2</sub>SO<sub>4</sub> solution, ROA and TOF of the charged samples reduced 27% and 2% compared with those of the uncharged (the anodic dissolution of matrix might result in the measured ROA deviating from the actual value).

#### 3.1.2. Effect of deionized water

For the uncharged sample tested in deionized water, the maximum stress reduced 7%, but the plasticity such as ROA and TOF obviously decreased 32% and 26% compared with those for samples tested in air (in Table 2).

### 3.1.3. Effect of $SO_4^{2-}$ anions

When  $SO_4^{2-}$  anions were added into deionized water, maximum stress for the uncharged samples decreased 45%, and both ROA and TOF deteriorated 72% and 65% of the specimens tested in deionized water, respectively.

#### 3.2. Fractography and cross-section observation

The fracture surfaces of the SSRT specimens in different environments are shown in Fig. 2. For the samples tested in air, it was characteristic of cleavage fracture with dimples locating at the

Table 1
The mechanical properties of as-cast AZ91 alloy in various conditions<sup>a</sup>

	Maximum stress (MPa)	Elongation (%)	Reduction of area (%)	Time to failure (h)
The uncharged AZ91 tested in air	161	11.4	5.8	32.9
The uncharged AZ91 tested in deionized water	150	11.1	3.9	24.4
The charged AZ91 tested in air	116	7.8	2.4	18.8
The uncharged AZ91 tested in solution	82	3.9	1.1	8.5
The charged AZ91 tested in solution	72	3.2	0.8	8.3

<sup>&</sup>lt;sup>a</sup> The test in each corrosive medium was repeated three times, and the relative error was below 5%. So a series of data was selected to illustrate the effect of hydrogen on the SCC of AZ91 alloy.

Table 2
Decrease of mechanical properties of as-cast AZ91 alloy in different environments

		Decrease of maximum stress (%)	Decrease of elongation (%)	Decrease of ROA (%)	Decrease of TOF (%)
Compared to the uncharged sample tested in	n air				
Uncharged sample	In deionized water	7	2	32	26
	in Na <sub>2</sub> SO <sub>4</sub> solution	49	66	81	74
Charged sample	In Na <sub>2</sub> SO <sub>4</sub> solution	55	72	86	75
Compared to those of the uncharged sample					
The charged sample tested in air		28	31	59	43
The charged sample tested in Na <sub>2</sub> SO <sub>4</sub>		12	18	27	2
Compared to those of the uncharged sample	tested in deionized war	ter			
The uncharged sample tested in Na <sub>2</sub> SO <sub>4</sub>		45	65	72	65

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