Determination of true stress corrosion cracking susceptibility index of a high strength Al alloy using glycerin as the non-corrosive atmosphere

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Abstract

The purpose of this work is to show that during slow strain rate testing (SSRT), the laboratory air might not be a non-corrosive atmosphere for evaluating the Iscc (stress corrosion cracking susceptibility index) of high strength Al alloys. Instead, it is proposed that glycerin could be effectively used as the non-corrosive atmosphere to find out the correct Iscc. This is demonstrated while examining 7010 Al alloy in the under aged condition.

Keywords: 7010 Al alloy; Stress corrosion cracking index; Slow strain rate testing

1. Introduction

The stress corrosion cracking (SCC) behavior of high strength Al alloys has been studied extensively [1–3]. Considering the laboratory air to be a non-corrosive medium, the SCC susceptibility of Al alloys is usually assessed by comparing the mechanical properties of the alloy obtained in 3.5% NaCl solution to those obtained in laboratory air [4–7]. However, our recent study [8] on the SCC behavior of 7010 Al alloy has shown that the ductility of the alloy in peak aged and over aged conditions decreased as the strain rate was decreased from $10^{-5}$/s to $10^{-6}$/s even when tested in laboratory air. In contrast to our observation, Conde et al [9] studies on 8090 alloy, tested in dry controlled air at three different strain rate $10^{-8}$/s, $10^{-7}$/s and $10^{-6}$/s showed increase in % elongation with decrease in strain rate. Hence, we proposed that the moisture present in the laboratory air could have reduced the ductility of 7010 Al alloy [8]. It is also well known that high strength Al alloys are susceptible to hydrogen embrittlement (HE) in humid atmosphere [10,11].

Literature review shows that the behavior of high strength Al alloy in laboratory air has been discussed by a few authors [12–14]. Hardie et al. [12] work on the HE behavior of 7179-T651 alloy and highlighted the aggressiveness of laboratory air, since moisture present in the atmosphere plays a major role. Scamans et al. [13] reported that Al–6Zn–3Mg alloy tested in laboratory air shows a significant loss in ductility when compared to its behavior in dry air. They have also studied the effect of relative humidity and found that the ductility ratio decreases with increase in relative humidity. Holroyd and Hardie [14] also reported that 7049 alloy exhibits some degree of embrittlement when tested in laboratory air. They showed that, the reduction in area ratio of this alloy decreased with decrease in strain rate when tested in laboratory air and sea water.
In contrast, the reduction in area ratio increased with decrease in strain rate when tested in vacuum/dry air.

In order to quantify the loss in ductility due to moisture, tests have also been carried out in vacuum and compared the data with those obtained in moist atmosphere or laboratory atmosphere [13,15]. Watkinson and Scully [15] used a vacuum system consisting of a rotary pump attached to an oil diffusion pump to study the effect of moisture upon the SCC of an Al–6Zn–3Mg alloy. Scamans et al. [13] also used a vacuum system to quantify the loss in ductility of Al–6Zn–3Mg alloy due to pre-exposure to moist gas. The % elongation of the specimen tested in various gases were divided by the % elongation of similar specimen tested in a vacuum of 10⁻⁵ Torr to quantify the ductility loss. As an alternative to such experimental set-up, glycerin was chosen as a medium for the stress corrosion tests in this study, due to its cheap availability.

7010 alloy in under aged condition was chosen for the study. The SCC results obtained in glycerin were compared with the results obtained in laboratory air and 3.5% NaCl solution. The fracture surfaces of the failed samples were further analyzed to study the mode of failure.

### 2. Experimental procedure

The chemical composition of 7010 alloy used in the present study, in wt.%, is Zn (6.30), Mg (2.35), Cu (1.55), Zr (0.14), Fe (0.09), Si (0.06) and Al (Balance). The material in the form of 5 mm were heat-treated to the under aged condition (i.e. solution treated at 465 °C, water quenched at room temperature and aged at 100 °C/8 h). The materials examined in this investigation were provided by Defence Metallurgical Research Laboratory (DMRL), Hyderabad. SCC susceptibility was studied using SSRT technique at 10⁻⁵/s and 10⁻⁶/s strain rates in laboratory air, 3.5% NaCl solution and glycerin (AR grade). Flat tensile specimens were prepared as per ASTM 557M-94 (gauge dimensions 50 × 12.5 × 5 mm) from 5 mm thick sheets. The specimens were polished to 1000 grade SiC paper and degreased by acetone before loading. Representative fracture surfaces were examined using SEM to identify the mode of fracture. Specimens for transmission electron microscope (TEM) were prepared by electrolyte polishing using 30 pct (by volume) nitric acid and 70 pct methanol at −35 °C. Thin foils were examined on a PHILIPS CM 200 electron microscope.

### 3. Results and discussion

The SSRT results of under aged 7010 alloy tested in laboratory air and 3.5% NaCl at 10⁻⁵/s and 10⁻⁶/s strain rates are shown in Table 1. In the laboratory air, the alloy exhibited 12.0% elongation and 19.5% reduction in area when tested at the strain rate of 10⁻⁵/s. However, for the same strain rate, the ductility of the alloy decreased to 6.0% elongation and 8.9% reduction in area upon exposure to 3.5% NaCl solution. In this solution, a further reduction in strain rate to 10⁻⁶/s resulted in the ductility drop to 3.0% elongation and 5.0% reduction in area. This decrease in ductility was an expected behavior, since high strength Al alloys are known to be highly susceptible to SCC at lower strain rates like 10⁻⁶/s and 10⁻⁷/s. Reconfirming our earlier studies [8], the ductility of the alloy further decreased with a decrease in the strain rates when tested even in the laboratory air. Accordingly, the alloy exhibited 8.6% elongation, 15.5% reduction in area when tested at 10⁻⁷/s strain rate in laboratory air. These values are lower than the corresponding values obtained at 10⁻⁵/s strain rate. Notably, this is in contradiction to the results reported by Conde et al. [9]. Generally, the ductility of Al alloy is expected to at least remain the same when testing strain rate is lowered, because enough time is available for the alloy for void nucleation and dimples formation. The ultimate tensile strength (UTS) of the alloy decreases marginally when tested in 3.5% NaCl at 10⁻⁶/s strain rate compared to the results in the same environment at 10⁻⁵/s strain rate.

In order to examine the inherent behavior of the alloy, tests were conducted in glycerin. The results are summarized in Table 2. The alloy exhibited ductility of 12% elongation and 21.1% reduction in area for 10⁻⁵/s strain rate in glycerin. At 10⁻⁶/s strain rate, the alloy exhibited 13.6% elongation and 21.8% reduction in area. It may be noted that there is no significant difference in the ductility when decreasing the strain rate from 10⁻⁵/s to 10⁻⁶/s, whereas in laboratory air the ductility de-

### Table 1

SSRT results of under aged 7010 alloy at different strain rates

<table>
<thead>
<tr>
<th>Environment</th>
<th>Strain rate</th>
<th>% Elongation</th>
<th>% Reduction in area</th>
<th>Ultimate tensile strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10⁻⁵/s</td>
<td>10⁻⁶/s</td>
<td>10⁻⁵/s</td>
<td>10⁻⁶/s</td>
</tr>
<tr>
<td>Laboratory air</td>
<td>12.0</td>
<td>8.6</td>
<td>19.5</td>
<td>15.5</td>
</tr>
<tr>
<td>3.5% NaCl</td>
<td>6.0</td>
<td>3.0</td>
<td>8.9</td>
<td>5.0</td>
</tr>
</tbody>
</table>
crease. The results indicate a marked deviation in the behavior of the alloy in glycerin as compared to its behavior when exposed to air. These values are higher as compared to those samples exposed in air at both $10^{-6}$/s and $10^{-5}$/s strain rates. It follows that the laboratory air was certainly not an ideal inert atmosphere for the stress corrosion tests and the alloy underwent SCC in the laboratory air. Since the laboratory atmosphere was free from chlorides or other SCC causing species, the specimen is suggested to have undergone HE. This was possible as the relative humidity of the laboratory air was found to be around RH $\approx 50\%$. The moisture present in the air could have caused the HE, as it could give rise to H$_2$ as per following reaction.

$$\text{Al} + 3\text{H}_2\text{O} \rightarrow \text{Al(OH)}_3 + 3/2\text{H}_2$$

Scamans et al. [13] reported that in the laboratory air, the crack growth rate of Al–6Zn–3Mg alloy is controlled by a continuous interaction between the specimen and the water vapor.

To determine the SCC susceptibility indices (Iscc), the ratio of the ductility of the sample in corrosive environment to that obtained in inert environment was obtained for both the parameters of elongation and reduction in area. These values are summarised in Table 3. The Iscc of the sample exposed to 3.5% NaCl was found to be 0.35 and 0.32 with respect to % elongation and % reduction in area respectively. Considering glycerin as an inert atmosphere and the values obtained in this medium were taken as true mechanical properties of the alloy, Iscc was determined. As shown by the Table 3, Iscc had dropped to 0.22 Iscc (for elongation) and 0.23 Iscc (for reduction in area) in this case. These results further indicate that 3.5% NaCl environment is a more severe environment and inflicts more damage to the alloy than that is evaluated using the data obtained in laboratory air. In an attempt to quantify the SCC susceptibility of the alloy in laboratory air, the SCC susceptibility index was also calculated by obtaining ratios between the ductility of the alloy in air and to that in glycerin. The results show 0.63 Iscc (for elongation) and 0.71 Iscc (for reduction in area). Even though the values are not as low as that obtained in 3.5% NaCl, less than unity values show that the alloy is susceptible to SCC in laboratory air.

The reason for the SCC susceptibility of the alloy in laboratory air is primarily due to the relative humidity (RH $\approx 50\%$) present in the atmosphere. Polyanskii pointed out that HE occurs in the presence of water (vapor or liquid) but not in dry hydrogen [11]. It is also widely suggested that diffusion of hydrogen to and along grain boundaries is accelerated by the presence of dislocations adjacent to the grain boundaries and hence increases the SCC susceptibility [16]. In the present study, the TEM micrograph obtained from the under aged 7010 show dislocations in the matrix (Fig. 1(a)); such dislocations appear to be partially embedded in the foil and seen as broken lines along a direction. Array of dislocations are also seen at the sub grain boundary (Fig. 1(b)). It is to be considered that in the under aged

### Table 2

<table>
<thead>
<tr>
<th>Strain rate</th>
<th>% Elongation</th>
<th>% Reduction in area</th>
<th>Ultimate tensile strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$10^{-3}$/s</td>
<td>12.0</td>
<td>21.1</td>
<td>548</td>
</tr>
<tr>
<td>$10^{-5}$/s</td>
<td>13.6</td>
<td>21.8</td>
<td>544</td>
</tr>
</tbody>
</table>

### Table 3

<table>
<thead>
<tr>
<th>SCC susceptibility index</th>
<th>Iscc (elongation)</th>
<th>Iscc (reduction in area)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5% NaCl/laboratory air</td>
<td>0.35</td>
<td>0.32</td>
</tr>
<tr>
<td>3.5% NaCl/glycerin</td>
<td>0.22</td>
<td>0.23</td>
</tr>
<tr>
<td>Laboratory air/glycerin</td>
<td>0.63</td>
<td>0.71</td>
</tr>
</tbody>
</table>

Fig. 1. TEM micrographs of under aged 7010 alloy (a) showing dislocations lines in the matrix and (b) arrays of dislocations at the sub grain boundary.
condition, the microstructure would consist of stable Guiner–Preston zones as well as finer \( \eta' \) precipitates that would be sheared by dislocation during its wake. The resultant strain localization and the dislocation pile-up stress at the grain boundary would further contribute to the SCC of such materials.

The SEM fractographs of the alloy failed in laboratory air, 3.5% NaCl solution and glycerin are shown in Fig. 2. The failed sample in the laboratory air shows cracks initiated at the edge (Fig. 2(a)). Many such cracks were visible along the edges. At a higher magnification, the pancake shaped elongated grains are revealed exhibiting a quasi-intergranular mode of failure (Fig. 2(b)). The literature reports that intergranular failure could result from hydrogen induced grain boundary decohesion in water vapor atmosphere [11]. A close look at the elongated grains shows not only cleavage features but also few small dimples, indicating a certain amount of grain deformation. (Fig. 2(b)). Lynch [17] has reported that even a humidity of 20% RH in air is sufficient to cause brittle fracture of an Al–Zn alloy. The samples tested in 3.5% NaCl solution also show large cracks at the edges (Fig. 2(c)). The mode of fracture is typically intergranular and the small dimples seen in air-fractured samples are not visible (Fig. 2(d)). Interestingly, in glycerin environment, the samples did not show edge cracks (Fig. 2(e)), while this is predominantly seen in laboratory air and environment (3.5% NaCl) tested samples. The fracture mode was predominantly ductile in nature and the dimples were also elongated (Fig. 2(f)). It should be noted that there was a decrease in the dimple size of the fracture samples tested in laboratory air as compared to that of the samples tested in glycerin (Fig. 2(b) and (f)). Ohnishi and Higashi [18] have reported that the decrease in mean dimple size and micro void nucleation in an Al–8%Mg alloy is due to the dissolution of H in the alloy. It would be interesting to examine further whether the ductility ratios of the alloy obtained in air to that obtained in glycerin correspond to HE behavior of the alloy. Further experiments are planned to examine mechanical behavior of this alloy in pure water and with different chloride contents to understand the contribution of HE and SCC towards embrittlement behavior of this alloy.

![Fractography of under aged 7010 alloy tested in: (a) laboratory air shows cracks in the edge, (b) laboratory air shows intergranular attack, cleavage and few small dimples in the centre; (c) 3.5% NaCl solution shows cracks in the edge, (d) 3.5% NaCl solution shows typical intergranular attack in the centre; (e) glycerin shows no cracks seen in the edge, (f) glycerin shows predominant ductile failure with elongated dimples in the centre.](image-url)
4. Summary

- The ductility of under aged 7010 Al alloy in laboratory air decreased when the strain rate was decreased from $10^{-5}/s$ to $10^{-6}/s$. On the contrary, the sample did not show any reduction in ductility with decrease in strain rate, when tested in glycerin. This showed that Iscc values of an environment, determined in relation to the data obtained in laboratory air can be optimistic.
- Under aged 7010 alloy is shown to be susceptible to hydrogen embrittlement.
- Data obtained in glycerin can be safely compared with that obtained in a corrosive environment to calculate the correct Iscc values.

Acknowledgment

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References