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HAL Id: jpa-00226631
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Submitted on 1 Jan 1987

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CORROSION BEHAVIOUR OF Al-Li-Cu-Mg ALLOY 8090-T651

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Summary: Using accelerated laboratory tests the Al-Li-Cu-Mg alloy 8090-T651 was found to be susceptible to intergranular and exfoliation corrosion. In the long transverse direction the material was proved to be resistant against stress corrosion cracking, whereas in the short transverse direction a susceptibility to stress corrosion cracking was ascertained. In constant load and constant strain tests a threshold stress below 50 MPa was determined. The stress corrosion crack path was intergranular. On the SCC fracture surfaces a brittle fracture mode and attacked grain and subgrain boundaries were observed.

1. Introduction

Metallurgical investigations performed to replace the alloys 2024 and 7075 which predominate in aircraft structural applications, have led to the development of a new kind of alloy 8090 with lithium as main alloying element and a new lithium containing 2000 series alloy 2091. Because of the extremely base electrochemical behaviour of the element lithium, the corrosion performance of the lithium containing aluminium alloys has thoroughly to be investigated. The published data of the susceptibility of these alloys to various forms of corrosion are limited. The aims of the present investigations are to obtain information about the corrosion behaviour of the Al-Li-Cu-Mg alloy 8090-T651 (British Alcan Aluminium Ltd. former specification: DTDXXXA-T651). The localized corrosion phenomena like pitting, exfoliation and intergranular corrosion and stress corrosion cracking (SCC) are in the centre of interest.

2. Experimental methods

The 8090 aluminium alloy tested in the present investigation was supplied by British Alcan Aluminium Ltd. as 25 mm thick plate in the T651 heat treatment. The unrecrystallized alloy had the composition (wt.-%) Al-2.42Li-1.24Cu-0.60Mg-0.12Zr-0.12Fe-0.05Si-0.05Ti.

The following corrosion phenomena using the testing techniques and standards specified, have been investigated:

- Localized corrosion (pitting, exfoliation, intergranular corrosion)
  - Neutral continuous salt spray test (ASTM B117); the electrolyte is an aqueous 5% NaCl solution.
  - Acidified continuous salt spray test (ASTM B287); the electrolyte is an aqueous 5% NaCl solution at pH = 3.2 (±0.1) acidified using acetic acid.
  - Intermittent acidified salt spray test (MASTMAASIS) [1]; same electrolyte as used in ASTM B287.
  - Continuous immersion method of Military Specification MIL-H-6088F; the electrolyte is an aqueous solution of 57 g/l NaCl and 10 ml/l H₂O₂.
  - EXCO test (ASTM G34-79); the electrolyte is an aqueous solution of NaCl, KNO₃ and HNO₃, with an apparent pH of 0.4.

- Stress corrosion cracking
  - Constant deformation tensile tests (LT-direction); alternate immersion in an aqueous 3.5% NaCl solution (ASTM G44-75).
  - Constant load tensile tests (LT- and ST-direction); continuous immersion in an aqueous solution of 2% NaCl and 0.5% Na₂Cr₂O₇ at pH = 3 according to LN 85585.
  - Constant deformation C-ring tests (ST-direction) (ASTM G38-73); alternate immersion in an aqueous 3.5% NaCl solution (ASTM G44-75).
  - Slow strain rate tests (LT-direction); continuous immersion in an aqueous 3.5% NaCl solution.
The localized corrosion behaviour was studied using flat rectangular coupons with a size of 25 by 30 mm aligned parallel to the rolling plane (the EXCO-test specimens had a size of 40 by 60 mm). Two locations of the plate were examined: U/2 and U/8 plane (t = thickness of the plate). Previous to the corrosion test, one surface of each specimen was ground using a 1000 grit emery paper. The remaining surfaces were masked by means of an easily removable organic coating. After the salt spray tests the corrosion products were removed from the specimen surfaces according to the cleaning procedure suggested in ASTM G1. Then the weight loss was determined. To qualify the severity of the corrosion attack, five metallographic cross sections of each test piece were examined. The depth and the length (perpendicular and parallel attack regarding the surface) of the corrosion attack were measured and the maximum and the average values of both parameters were determined.

To study the stress corrosion behaviour under constant deformation, tensile specimens in the LT-direction with a gauge length of 22 mm and a diameter of 3.5 mm were mounted in a special rigid frame for the application of a constant strain. Then they were stressed up to 75% of their 0.2% proof stress and 30 days alternately immersed in 3.5% NaCl solution according to ASTM G44-75. Tensile specimens of the same dimensions and orientation were also tested in dead weight loaded tensile test machines under constant load according to the German specification LN 65666. The applied initial stress was again 75% of the 0.2% proof stress. The maximum test period was 1000 h. Specimens not broken during the test were metallographically examined whether stress induced cracks did appear or not.

Slow strain rate tests in the LT-direction were also carried out. The strain rates were in the range between \(10^{-3}\) and \(10^{-4}\) s\(^{-1}\). Details about this testing technique are described elsewhere [2]. The specimens were tensile tested in an inert environment, e.g. laboratory atmosphere dried by embedding the specimens in \(Mg(ClO_4)\_2\) -powder, as reference test, and in aqueous, aerated 3.5% NaCl solution. To characterize the susceptibility to stress corrosion cracking, the relative fracture energy was chosen. This parameter is defined as the ratio of the plastic fracture energy of a specimen tested in the corrosive environment to the plastic fracture energy of a specimen tested in the inert environment at the same strain rate. The fracture energy is the area below the stress-elongation curve.

The stress corrosion behaviour in the ST-direction was studied using C-ring specimens with an outer diameter of 24 mm and a wall thickness of 1.5 mm. The C-rings were stressed according to the method described in ASTM G38. Due to the experimental stress procedure the lowest value of applied stress which yet could be determined accurately, was 50 MPa. The stressed C-rings were alternately immersed in 3.5% NaCl solution. During the initial period of testing the specimens were frequently inspected using a stereoscopic light microscope at a 20fold magnification. If a first crack was visible at this magnification, the specimen was considered as failed. Constant load tests in the ST-direction were also carried out using tensile specimens with a reduced gauge length of 9 mm due to the thickness of the alloy plate. The etching solution, which is required in the German specification LN 65666 to remove the oxide film of the specimens before exposure, seems not to be suitable for Al-Li alloys, because the grain boundaries were heavily attacked. Therefore an aqueous solution of 20 g/l \(Cr_2O_3\) and 50 ml/l \(H_2PO_4\) was used (ASTM G1). In spite of this above mentioned attack of the LN 65666 solution no influence of the etching procedure on the stress corrosion behaviour was observed.

3. Results

3.1 Localized Corrosion Behaviour

In the neutral salt spray fog the type of corrosion attack was predominantly pitting, whereas in the acetic acid salt spray fog exfoliation corrosion prevailed at longer exposure time periods. In the latter case the exposed surfaces showed noticeable leafing; furthermore the bottom of the pits had a red brown colour indicating an enrichment in copper. In the metallographic cross sections of the specimens exfoliation corrosion was observed often surrounded by intergranular corrosion. But also undermining pitting was found. In the intermittent acidified salt spray fog the principal type of corrosion attack was shallow pitting accompanied by less pronounced exfoliation.

In Figure 1 the weight loss of the specimens exposed to the different salt spray tests is shown for various exposure time periods. The weight loss is negligible in the neutral salt spray test and most pronounced in the acetic acid salt spray test. In the latter, the centre of the plate was more attacked compared with the edge. The reverse result was found in the intermittent salt spray fog. Here the edge region of the plate was more corroded.

In Figure 2 the average and maximum values of the depth and length of corrosion attack after various exposure time periods are shown for the different salt spray tests. From these figures the same ranking is found as indicated from the weight loss results, e.g. the corrosion attack is most severe in the acetic acid
Figure 1. Weight loss of specimens in different salt spray tests as function of exposure time periods and locations in the plate.

Salt spray test, intermediate in the intermittent acidified salt spray test and less in the neutral salt spray fog. (This is not valid for the maximum value of the depth near the edge of the plate).

Figure 2. Depth and length of corrosion attack on specimens tested in different salt spray fogs for various exposure time periods and different locations in the plate.

After the MIL-H-6088F test the metallographic sections of the specimens reveal pitting and intergranular attack. In the centre of the plate intergranular corrosion extends more in the rolling plane in comparison with the edge region, where pitting is more pronounced. The maximum depth of corrosion attack measures 80 and 160 μm in the centre and the edge region of the plate, respectively.

In the EXCO test very severe exfoliation was generated. The visual assessment of exfoliation according to ASTM G34-79 is presented in Table 1 for various immersion time. After 48 hours the highest degree of exfoliation (ED) was observed. No difference between the two locations of the plate was found.
Table 1. Visual assessment of exfoliation according to ASTM G34-79.

<table>
<thead>
<tr>
<th>exposure time</th>
<th>visual rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 h</td>
<td>P</td>
</tr>
<tr>
<td>6 h</td>
<td>P/EA</td>
</tr>
<tr>
<td>24 h</td>
<td>EA</td>
</tr>
<tr>
<td>48 h</td>
<td>ED</td>
</tr>
</tbody>
</table>

Table 2. Results of the slow strain rate tests of 8090-T651 in LT-direction in inert environment and 3.5% NaCl solution.

<table>
<thead>
<tr>
<th> </th>
<th>Inert environment</th>
<th>3.5% NaCl solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>edge</td>
<td>centre</td>
</tr>
<tr>
<td>0.2% Pb (MPa)</td>
<td>394</td>
<td>474</td>
</tr>
<tr>
<td>UTS (MPa)</td>
<td>485</td>
<td>530</td>
</tr>
<tr>
<td>EL (%)</td>
<td>9.4</td>
<td>8.1</td>
</tr>
<tr>
<td>RA (%)</td>
<td>28</td>
<td>21</td>
</tr>
<tr>
<td>FE (J/cm²)</td>
<td>43</td>
<td>42</td>
</tr>
</tbody>
</table>

3.2 Stress Corrosion Cracking

Constant strain and constant load tests characterized the investigated plate material in the LT-direction being not susceptible to stress corrosion cracking. In both test methods no macroscopic fracture of the specimens was observed during the test period. The metallographic cross sections of the specimens revealed no cracks propagated normal to the stress axis but intergranular corrosion was found up to a depth of 700 ㎛ and extending up to 1000 ㎛ in stress direction.

In the ST-direction there is a considerable susceptibility to stress corrosion cracking. In Figure 3 the time to failure of the C-rings versus the initial stress is shown. The results of analogous C-ring tests of 2024-T351 and 2014-T351 plate material are also plotted. The threshold stresses of the three alloys in the given heat treatment were determined to be less than 50 MPa. At the same stress levels the time to failure of the 8090-T651 alloy was always longer in comparison with the 2000 series alloys. The metallographic cross section of a 8090-T651 C-ring stressed to 50 MPa clearly reveals a stress corrosion induced intergranular crack, whereas the cross section of an unstressed C-ring 1000 h alternately immersed exhibits only intergranular corrosion.

![Figure 3](image-url)  
Initial stress-time to failure relationship of C-ring specimens of 8090-T651, 2014-T351 and 2024-T351 alternately immersed in 3.5% NaCl solution (ST-direction).

The results of the constant load tests in the ST-direction are plotted in Figure 4. At stress levels of 50 and 30 MPa the specimens were not broken during the maximum time periods of 1000 h, but the metallographic analysis of these specimens indicated intergranular deep cracks (up to 1.5 mm) normal to the stress axis. Therefore these specimens were diagnosed as having failed. The threshold stress in the ST-direction determined from the constant load tests is lower than 30 MPa.
Figure 4. Initial stress-time to failure relationship of 8090-T651 tensile specimens tested in ST-direction under constant load in 2% NaCl + 0.5% Na₂CrO₄ at pH = 3.

The results of the slow strain rate tests in the LT-direction are given in Table 2. In inert environment and in 3.5% NaCl solution the strain rate has no influence on the mechanical properties in the investigated range.

A thickness profile of the plate was found, e.g. the strength is higher in the centre of the plate than in the edge region, whereas the ductility is lower in the centre. The aggressive environment has no influence on the mechanical properties of 8090-T651 in the LT-direction.

3.3 Fractography

To ascertain the influence of the electrolyte on fracture mode, at first the fracture surface of specimens, which were tensile tested in an inert environment, had thoroughly to be investigated. The main features of the fracture in the ST-direction without an influence of the environment are:

1. The macroscopic fracture plane is normal to the load axis.
2. The fracture path is predominantly intergranular.
3. The fracture planes normal to the load axis reveal a fine shallow dimple structure.
4. These planes are parallel to the rolling plane and linked by vertical featureless steps.

More details and discussion of the fracture behaviour of this plate material are described elsewhere [4].

Figure 5 shows the fracture surface of a slow strain rate tested specimen exposed to 3.5% NaCl solution. In the corroded region a brittle fracture mode and grooves caused by dissolution of grain and subgrain boundaries were observed (Figure 5a and 5b). The grooves are typical on all fracture surfaces of SCC induced failed specimens. Figure 6a shows the fracture surface of a specimen failed in the 2% NaCl + 0.5% Na₂CrO₄ electrolyte under the constant load corresponding to an initial stress of 60 MPa (ST-direction). Between the corrosion grooves and the ductile dimple structure a featureless brittle region is observed (Figures 6a and 6b). On the fracture surfaces slip steps were occasionally found. In the region of fast fracture (without any influence of the environment) these slip steps were associated with a fine dimple structure (Figure 6c), whereas on SCC induced fracture surface the dimples were not observed (Figure 6d).

4. Discussion

Due to the results of the EXCO test and the acetic acid and intermittent acidified salt spray tests, the 8090-T651 is susceptible to exfoliation corrosion. The worst behaviour to exfoliation corrosion was observed in the EXCO test. As found by Lane et al. [4], this test and the intermittent acidified salt spray test deduce an exfoliation corrosion behaviour not correlated to that observed at marine exposure. Therefore it is assumed that at least the EXCO test is too severe for this Al-Li alloy and doesn't describe exactly the service behaviour. Because of the lack of longtime exposure data, it can not be decided, whether these salt spray tests are realistic or not. The different corrosion behaviour between the centre and the edge region of the plate corresponds to the thickness profile of the mechanical properties. This profile is caused by crystallographic texture [5]. Metallographic examinations and transmission electron microscope investigations at this plate material don't show any variation of the microscopic structure over the thickness [3].

The test procedure according to MIL-H-6088F causes the occurrence of intergranular corrosion in this material. This type of corrosion attack which was also observed in other corrosion tests, occurs preferentially along grain and subgrain boundaries in the rolling plane and may be caused by the dissolution of grain...
Figure 5. Fracture surface of a 8090-T651 tensile specimen tested in ST-direction at a strain rate of $2.6 \times 10^{-7}$ s$^{-1}$ in 3.5% NaCl solution (in Figure 5b the specimen is tilted 55°).

Figure 6. Fracture surface of a 8090-T651 tensile specimen tested in the ST-direction under constant load of 60 MPa initial stress in 2% NaCl + 0.5% Na$_2$CrO$_4$ at pH = 3 (Figure 6c shows a region of the fast fracture).
boundary precipitates, possibly the equilibrium phase $\delta$ (AILi). Transmission electron microscope observations confirmed that in this plate material the $\delta$ phase is precipitated on grain and subgrain boundaries [3]. As shown by Niskanen et al., the $\delta$ phase which is anodic to the matrix has a deleterious effect on the corrosion behaviour of Al-Li, Al-Li-Mn and Al-Li-Zr alloys [6].

The results of the C-ring tests and the constant load tests indicate a stress corrosion threshold stress below 50 MPa in the ST-direction. Thus the stress corrosion behaviour of 8090-T651 is similar to that of 2000 and 7000 series alloys in the T6 heat treatment, whose threshold stresses were estimated to be less than 55 MPa in the ST-direction [7].

The grooves observed on the SCC fracture surfaces correspond to attacked grain and subgrain boundaries which are nearly vertical to the primary fracture plane, and may be formed by the dissolution of the active $\delta$ phase. The attack on grain boundaries normal to the stress axis should be enhanced by the applied strain. Therefore the brittle fracture surfaces of specimens slow strain rate tested in 3.5 % NaCl solution may result from an anodic dissolution process caused by a particular crack tip chemistry of lithium containing aluminium alloys. Whereas in chloride solution a pH of 3.2 was found at the crack tip of conventional alloys [8], in Al-Li alloys the pH can increases due to the hydrolysis of the Li$^+$ ions [9]. LiOH is a strong base and mostly dissociated. The OH$^-$ ion is more aggressive against aluminium alloys than the halide ions [10]. But an embrittlement mechanism of hydrogen evolved during the hydrolysis can not be excluded.

If the specimens were failed in the 2% NaCl + 0.5 % Na$_2$C$_2$O$_4$ solution under constant load, large featureless regions were observed on the fracture surfaces (Figures 6a and 6b). In an acid electrolyte at pH=3 stable surface films cannot occur. The dissolution of metal is hindered by chromate inhibitors. Nevertheless metal dissolution is not entirely prevented indicated by the network of grooves which is not so large than in the neutral 3.5% NaCl solution (Figure 5a) and more confined to narrow zones which can reach to the centre of the fracture surfaces. In contrast to the anodic dissolution the cathodic reaction of hydrogen discharge should not be influenced by the chromate inhibitors in the electrolyte. Therefore, in this electrolyte the brittle fracture mode is obviously caused by hydrogen embrittlement.

Only from fractographic studies it is difficult to establish that the cracks propagate as a result of hydrogen embrittlement or of an active path mechanism. Maybe both mechanisms are operative. Due to the pronounced susceptibility to intergranular corrosion it is also possible that a strain assisted intergranular corrosion causes the low SCC resistance in the ST-direction. Further experiments in electrolytes with cathodic poisons and under potentiostatic control must be performed to answer these questions.

5. Conclusions

From the results of the present investigations the following conclusions concerning the corrosion behaviour of 8090-T651 can be drawn:

1. The plate material is susceptible to exfoliation corrosion due to the elongated grain structure (pronounced pancake structure).
2. Corrosive environments promote intergranular attack.
3. In the LT-direction the material is resistant against stress corrosion cracking.
4. The stress corrosion threshold stress in the ST-direction is lower than 50 MPa. As compared with 2014-T351 and 2024-T351 the SCC behaviour of the alloy 8090-T651 is at least equivalent or better.

6. References