Stress corrosion cracking of Al7075 alloy processed by equal channel angular pressing

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Abstract

The aim of the present work was to evaluate the stress corrosion cracking (SCC) behavior of the annealed Al-7075 alloy before and after Equal Channel Angular Pressing (ECAP). The SCC behavior of the Al-7075 alloy before and after ECAP was evaluated using slow strain rate testing (SSRT). Tensile and SCC behavior of the UNECAPed and ECAPed samples were compared. The elongation and ultimate tensile strength (UTS) was decreased 1.25, 1.1 times respectively after SCC in the annealed Al 7075. After ECAP, about 1.6 times decreases in elongation and 1.09 times decrease in UTS is observed. The decrease in ductility is more as compared to UTS. The fracture surface analysis (from the SSRT tests in 3.5% NaCl solution) revealed predominant ductile failure in the before ECAP and mixed (quasi cleavage) mode of failure was observed after ECAP. Though the SCC resistance decreases due to ECAP, this appears a positive sign that the SCC may be improved by modifying process parameters and the condition of the sample.

Keywords: Severe Plastic deformation, equal channel angular pressing, stress corrosion cracking, Al 7075, mechanical properties, TEM.

1. Introduction

High strength aluminum alloys are usually chosen because of their high strength and stiffness, which are derived from precipitation hardening. However, high strength aluminum alloys are poor resistance to stress corrosion cracking (SCC), particularly when they are at near peak strength condition (Speidel, 1975; Bayoumi, 1996; Yamasaki et al., 2001), precipitation hardening is directly responsible for the SCC susceptibility of high strength aluminum alloys. The process of SCC in aluminum alloy is complex and depends on many parameters. Once the crack appears its growth is influenced by a combination of the microstructure of the material, (heat treatment, the amount of impurities, manufacturing background etc.) the level of stresses and the aggressive conditions of the environment. The microstructural state of the material is the most well studied parameter used to find a microstructure resistant to SCC while presenting the high performance mechanical properties of the material (Speidel, 1975; Horita et al., 2001).

The SCC susceptibility of 7xxx aluminum alloys are well known to be strongly affected by their microstructure characteristics. There principal micro structural features have been discussed concurring the influence of Si. They have precipitate free zone (PFZ), Matrix precipitate structure, and grain boundary precipitate (GBP) structure (Adler et al., 1972; Poulse et al. 1974; Thompson and Matthias, 1993; Li et al., 2008). Generally over aging can improve the SCC resistance of high strength aluminum alloys by means of the more homogeneous slip mode and the reduction of slip planarity, due to increase of matrix precipitate size and the associated change from GP zones to semi coherent and incoherent precipitates (Thompson and Matthias, 1993; You et al., 1995) on the other hand the increasing size of GBPs during over aging have been proposed to explain the higher SCC resistance in the 7xxx series aluminum alloys (Poulse et al. 1974). The influence of the PFZ on SCC susceptibility is uncertain, as width of the PFZ may lead to improvement or reduction as may have no effect on SCC susceptibility. However, Limited study had been available about the effect of grain size on SCC. Hovita et al. (2001) had discussed about the grain refinement in SIX different commercial Al alloy using ECAP. Grain refinement can be made by ECAP processing for high strength aluminum alloy and generally results in beneficial effects for mechanical property such as yield strength, ductility, fracture toughness and fatigue life.
Somehow significant effect produced by grain refinement is that on the character of deformation at high temperatures which is super plastic (Shin et al., 1998). Generally, fine grain structure presents smaller size of GBPs (Tsai and Chuang, 1996) and super plastic forming will cause decay of the mechanical properties in the alloy because of the deformation of cavity and grain growth (Bampton and Raj, 1982; Mahoney et al., 1983). It means that the unsuitable fezzes (micro structural) continuous will cause serious SCC damage in the super plastic formed work pieces.

Previous investigations have paid much attention to the SCC resistance of aluminum alloys. However, reports on SCC testing have been very rare (Onoro and Ranninger, 1999) and there is no report on SCC resistance of ECAPed Al 7075 alloy. The purpose of this investigation was to evaluate the influence of the micro structural and mechanical features on SCC susceptibility of Al 7075 alloys at annealed (as received) condition .This is the first attempt made to evaluate the behavior of the SCC of the before and after ECAP pressed billets.

2. Experiment Procedure

2.1 Equal Channel Angular Pressing (ECAP)

The geometry of this die provides that the material is deformed by simple shear at ideal, frictionless, conditions. The cross section of the specimen remains equal before and after a processing step, thus it is possible to subject one specimen several times to ECAP in order to reach highest degrees of plastic deformation. A circular cross section of the channel provides the possibility of a materials processing at four different routes that are distinguished by their different combinations of sample rotation around the channel axes between consecutive processing steps. Fig. 1 shows the photograph of die used in this study, where die angle $\Phi$ is 90° and the outer arc curvature $\psi$ is 20°, and the diameter of the die opening and diameter of the punch were 12 mm, sample also must have the same dimensions of the die opening. Lubricant (MoS$_2$) used for inside the die channels, billet and to the punch. It reduces the friction effect between the punch, billet and the channels during the process. Al7075 alloy with 12mm diameter having the length of 90mm was used for this study. The billet was processed at room temperature for single pass (first pass) only.

![Figure 1](image-url)

**Figure 1 (a)** Experimental Setup of ECAP die with punch. **(b)** Two Dimensional representation of ECAP

The Al-7075 alloy was characterized using X-ray diffraction (XRD) to study the phase analysis, XRD was done using CuK$\alpha$ radiation ($\lambda = 1.540598$ Å). XRD was done from angle 10° to 80°. Tensile tests were carried out using a Tensometer. The samples were cut with dimensions of ASTM–E8 standards with a gauge length of 16 mm and 4 mm gauge diameter. HMV-2000 micro hardness tester was used in this work. The load of 300 grams with the dwell time of 20 seconds indentation time was used for conducting the hardness measurement. The TEM samples were prepared from the cross plane in the centre of the pressed billet. The billet was further polished and manually thinned up to 100microns with the help of disc polishers using 80-1800grade wet polishing papers. Polished samples were further etched and analyzed by Scanning Electron Microscope HITACHI S500 model .Finally the polished sample was thinned to perforation using a twin jet polisher with an electrolyte of 33% nitric acid – methanol at -40°C,and the grain size, precipitate size were analyzed by using TEM images. TEM investigations were carried out with a Philips CM20 microscope operated at 200 kV and a JEOL 3000F TEM working at 300 kV, respectively. The XRD machine was coupled with the computer from which the XRD pattern is obtained. The XRD were performed with a Rigaku Ultima Model-III X-
ray diffractometer. XRD data were analyzed and indexed using standard JCPDS data. Further analysis of the obtained plots to explain the work was carried out using ORIGIN and XRDA software packages.

2.2 Stress Corrosion Cracking (SCC)

The SCC tests were carried out by using a 12 mm rounded samples. The SCC behavior of the alloy was studied using slow strain rate testing (SSRT) method as per ASTM G129-00. SSRT was carried out using a tensile testing machine (Universal Calibration Corporation). The schematic representation of the sample for SSRT as per standard is shown in Fig. 2. Tensile properties were evaluated in the long-transverse direction. The SSR test is carried out in 3.5% NaCl solution and at a strain rate of $10^{-4}$ s$^{-1}$. The SCC susceptibility of the alloy was evaluated by parameters such as elongation, reduction in area and ultimate tensile strength (UTS).

![Figure 2: Schematic diagram of SSRT sample](image)

**Figure 2** Schematic diagram of SSRT sample

Initial billet with the length of 90mm & the diameter of 12mm machined specimen is shown in Fig.3 (a). Rounded tensile samples were prepared as per standard as shown in Fig.3 (b) for the SSR test. The processed billet after first pass of ECAP is shown in Fig.4 (a) and the SSRT tensile specimen prepared from the processed billet with the standard dimension is shown in Fig. 4(b)

![Figure 3: Initial Billet before ECAP (a) Tensile sample for SSRT before ECAP (b)](image)

**Figure 3** (a) Initial Billet before ECAP (b) Tensile sample for SSRT before ECAP

![Figure 4: ECAPed Billet (a) Tensile Sample for SSRT after ECAP (b)](image)

**Figure 4** (a) ECAPed billet (b) Tensile Sample for SSRT after ECAP
3. Results and Discussions

3.1 X-ray diffraction (XRD)
Al-7075 alloy Fig. 5(a) & (b) shows that phase variation in the XRD patterns before and after ECAP. In addition, the peaks MgZn and FeAl precipitates were observed after ECAP. These new phases may be precipitated due to high strains which need further detailed analysis.

![Figure 5](image)

**Figure. 5** Phase analysis of Al-7075 alloy: (a) before ECAP (b) after ECAP

3.2 Micro structural Studies
The optical microstructures of Al-7075 alloy before and after ECAP first pass are shown in Fig. 6 (a), (b). The annealed Al 7075 alloy shows network of very small equiaxed recrystallized grains. The ECAPed alloy shows very small equiaxed grains and spherical particles distributed reasonably homogeneous throughout the matrix. The average grain size of the annealed condition is ~1.5µm. After first pass the fragmented grains are appear and the average grain size was ~ 0.7- 0.9 µm

![Figure 6](image)

**Figure. 6** Optical microstructure of Al-7075 (a) before ECAP (b) after ECAP
Fig. 7(a) & (b) shows the TEM micrographs showing distribution of the precipitates along the grain boundary and the matrix in the annealed Al7075 alloy. Rod shaped precipitates Fig 7. (c) & (d) were observed in the annealed condition. The length was 190-200nm and width was 40-50nm.

Figure. 8 (a), (b) Bright Field TEM micrographs after ECAP.
The TEM bright field image Fig 8(a) & (b) shows that the precipitates distributions in the matrix of the ECAPed alloy. After First pass the precipitates were fragmented and the size of the precipitate cut down into small in size. In addition, the precipitation along the grain boundary also seems to be slightly different.

**Figure 9** (a) EDAX of annealed Al 7075 (b) EDAX of Al 7075 after ECAP

EDAX analysis of the GBPs in both the alloys as presented in Fig.9 (a) & (b) shows that they mainly consist of Zinc, Magnesium and Al. It is also noted that there is no difference in the Chemical composition of the GBPs in the annealed alloy and after ECAP. Comparatively the observed precipitate is around 50% reduction in length (~90μm) and in width of around 25%. While the base alloy shows discontinuity in the arrangement of precipitates in the matrix and along the grain boundary, the alloy after ECAP has continuous presence of precipitates in the matrix and along the grain boundary.

### 3.3 Mechanical properties of Al-7075 alloy before and after ECAP

The mechanical properties of Al-7075 alloy were performed before and after ECAP and the results are given in Table 1. The reported micro hardness value for each sample is the average of six measurements. After ECAP the tensile yield strength, ultimate tensile strength, and micro hardness of the sample are 66%, 28%, and 13% higher than before ECAP. However the % elongation decreases by 45%.

**Table 1** Mechanical properties of Al-7075 alloy

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average Micro hardness (VHN)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Tensile yield strength (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before ECAP</td>
<td>69</td>
<td>245</td>
<td>147</td>
<td>15.33</td>
</tr>
<tr>
<td>After ECAP</td>
<td>78</td>
<td>314</td>
<td>244</td>
<td>8.33</td>
</tr>
</tbody>
</table>

### 3.4 SCC behavior of Al-7075 before and after ECAP

The SCC behavior of the alloy was studied using slow strain rate testing (SSRT) method as per ASTM G129-00. SSRT was carried out using a tensile testing machine (Universal Calibration Corporation). Rounded tensile samples were prepared for the SSR Test. Tensile properties were evaluated in the long-transverse direction. The SSR Test method involved testing specimens in 3.5% NaCl solution. SSRT was carried out at a strain rate of 10^{-4} s^{-1}. The SCC susceptibility of the alloy was evaluated by parameters such as Elongation, Reduction in area and Ultimate Tensile Strength (UTS). The tested tensile samples of before ECAP and After ECAPed billets are shown in Fig.10 (a) & (b)
The SSRT results of the Al-7075 alloy before and after ECAP are given in Table 2 and the load displacement curves are shown in Fig. 11. Before ECAP sample exhibited 12.3% elongation, 26.5% reduction in area and 223 MPa UTS. After ECAP sample exhibited 5.4% elongation, 21.3% reduction in area and 288 MPa UTS. About 1.25 times elongation decreases and 1.1 times UTS is decreased due to SCC for the un ECAPed samples. After ECAP, about 1.6 times decreases in elongation and 1.09 times decrease in UTS is observed due to SCC. The magnitude of decrease in the mechanical properties after SSRT is nearly same for ECAPed and UnECAPed sample. Moreover, the Ultimate Tensile Strength (UTS) and Yield Strength (Y.S) of ECAPed sample after SSRT (288 & 182MPa) is higher than the UTS & Y.S of (245 & 147 MPa) Un ECAPed samples before SSRT. This gives positive hope in continuing the SCC behavior of ECAP studies. Further Studies on SCC with suitable Heat Treatment after ECAP and varying ECAP process parameters will definitely improve SCC behavior of the ECAPed Al 7075.

### Table 2 Mechanical properties after SSRT

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ultimate Tensile strength (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction in area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before ECAP</td>
<td>223</td>
<td>114</td>
<td>12.3</td>
<td>26.5</td>
</tr>
<tr>
<td>After ECAP</td>
<td>288</td>
<td>182</td>
<td>5.3</td>
<td>21.3</td>
</tr>
</tbody>
</table>

3.5 Fractography

Fractographs of the tensile and SSRT specimens before and after ECAP are analyzed by using SEM (Scanning Electron Microscope). Fig. 12(a). SEM image shows that the Dimple like structure is observed before ECAP. In general the dimple like...
structure indicates ductile mode of failure. Less dimple and micro voids are observed in the fracture surface of Al-7075 after ECAP as shown in Fig 12 (b). This is in line with the lesser ductility observed in the ECAP processed samples. Representative fracture surfaces (from the SSRT tests in 3.5% NaCl solution) of the Al-7075 alloy before and after ECAP samples in 3.5% NaCl solution are shown in Fig 12(c) & (d). UnECAPed sample shows a predominantly ductile mode of failure while after ECAP a mixed (quasi cleavage) mode of failure is observed.

![Fractographs](image)

Figure 12 (a) Fractographs of Al-7075 alloy before ECAP (b) Fractographs of Al-7075 alloy after ECAP (c) Fractographs of Al-7075 alloy before ECAP with SSRT (d) Fractographs of Al-7075 after ECAP with SSRT

### 4. Conclusion

Annealed Al 7075 alloy samples were processed by ECAP and the systematic studies were made on SCC by performing SSRT for before and after ECAP. The optical, SEM and TEM image analysis were performed.

1. XRD analysis suggests that the precipitates are not dissolved in the matrix due to ECAP and new phases (Mg$_7$Zn$_3$ and FeAl$_{1.23}$) are precipitated.
2. Decrease in UTS and % of Elongation is observed for both UnECAPed and ECAPed samples due to SCC. The decrease in Mechanical properties for ECAPed sample is higher than UnECAPed samples.
3. The elongation and ultimate tensile strength (UTS) was decreased by 1.25, 1.1 times respectively for the UnECAPed billets. After ECAP, about 1.6 times decreases in elongation and 1.09 times decrease in UTS is observed due to SCC.
4. However, UTS after SCC for ECAPed sample is higher than the UTS of UnECAPed sample subjected to the same SCC medium. This indicates that the SCC resistance after ECAP is better than SCC resistance of UnECAPed sample though slight decrease in % elongation is observed.
5. The fracture surface analysis (from the SSRT tests in 3.5% NaCl solution) revealed predominant ductile failure in the before ECAP and mixed (quasi cleavage) mode of failure in the after first pass in ECAP.
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References


Biographical notes

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