Objective

The objective of this study is to characterize and understand the effects of hydrogen on the deformation and fracture behavior of 2090 and 2219, especially at low temperatures. Additionally, 8090 and Weldalite will be included in this program.
HYDROGEN EMBRITTLEMENT OF Al-Li ALLOYS

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Abstract

The objective of this work is to study the effects of dissolved hydrogen on the mechanical properties of 2090 and 2219 alloys. The work done during this semi-annual period consists of the hydrogen charging study and some preliminary mechanical tests. Prior to SIMS analysis, several potentiostatic and galvanostatic experiments were performed for various times (going from 10 minutes to several hours) in the cathodic zone, and for the two aqueous solutions: 0.04N of HCl and 0.1N NaOH both combined with a small amount of As\textsubscript{2}O\textsubscript{3}. A study of the surface damage was conducted in parallel with the charging experiments. Those tests were performed to choose the best charging conditions without surface damage. Disk rupture tests and tensile tests are part of the study designed to investigate the effect of temperature, surface roughness, strain rate, and environment on the fracture behavior. In the present study, the importance of the roughness and environment have been shown using the disk rupture test as well as the importance of the strain rate under hydrogen environment. The tensile tests, without hydrogen effects, have not shown significant differences between low and room temperature.
Hydrogen Embrittlement of Al-Li Alloys

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Overview

• Objectives

• Approach

• Charging Experiments
  - Solutions tested
  - SIMS results

• Mechanical Tests
  - Disk Rupture
  - Tensile tests
Overview (Cont.)

- Aging experiments
  - PA for 2090T3 and W51
  - X-Ray Analysis

- Summary

- Need to Address

- Future work
Objectives

- Characterize effects of temperature, stress state, hydrogen on mechanical behavior.
- Correlate these effects with microstructure.
Approach

- Charpy Impact Test.
- Tensile Test control hydrostatic stress.
- Disk Rupture Test biaxial loading.
- Three Point Bend Test low strain rate.
Charging Experiments

- Methods to Charge Samples
- Electrochemical Solution
- Surface Analysis
- SIMS Results
Charging Experiments

Two principal methods can be used to charge samples:

- Autoclave
- Electrochemical cell
Choice of the Aqueous Solution

- Must contain H⁺
  ⇒ Low pH.

- Must not damage the sample
  ⇒ Choice of the charging voltage or current.
Optical Profilometer

Diagram:
- Video display TV monitor
- Video frame grabber
- Image sensor
- PZT Mirau
- PZT driver
- 12-bit D/A
- 16-bit microcomputer
- IEEE 488 parallel interface
- Desktop computer
- Color display monitor
Choice of the Voltage
Optical Profilometer (Results)

RMS: 0.186µm
RA: 0.139µm
P-V: 3.76µm

INVERTED

INVERTED

Orientation

(Remark: 59.11µm
P-Crv: -2.923µm
P-TT: 2.515µm)
SIMS Results
SIMS Results

SIMS PROFILE
XM = 2.0, YM = 2.0
4,000, 5,000, 1,000, 500
FILE = 010500
H2 O AL 59 SAMPLE

SIMS Profile Chart

Sputter Time (min.)

Counts/sec.
Interim Results

*Hydrogen Charging Parameters*

- 0.04 N HCL + As2O3 at -3V (1)
- 0.1 N NaOH + As2O3 at -3V (2)
- 0.04 N HCl + As2O3 at -500 µA (3)
- 0.04 N HCl + As2O3 at -5000 µA (4)
## Interim Results

### Hydrogen Charging Parameters

<table>
<thead>
<tr>
<th>Solution</th>
<th>Time</th>
<th>Diff. of counts/sec</th>
<th>H content</th>
<th>Surface Roughness RMS (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>5 hrs</td>
<td>0.057</td>
<td></td>
<td>0.0795</td>
</tr>
<tr>
<td>(2)</td>
<td>5 hrs</td>
<td>-</td>
<td></td>
<td>0.185</td>
</tr>
<tr>
<td>(3)</td>
<td>20 hrs</td>
<td>0.059</td>
<td></td>
<td>0.0772</td>
</tr>
<tr>
<td>(4)</td>
<td>20 hrs</td>
<td>0.0185</td>
<td></td>
<td>0.0861</td>
</tr>
<tr>
<td>Uncharged</td>
<td>-</td>
<td>-</td>
<td></td>
<td>0.0752</td>
</tr>
</tbody>
</table>
Interim Results

Hydrogen Charging Parameters

The two selected charging solutions are:

- 0.04 N HCl + As2O3 at -3 V for 5 hrs
- 0.04 N HCl + As2O3 at -500 μA for 20 hrs
Charging Experiments

- SIMS technique has not yet been successful

- Evaluating other surface analytical techniques for hydrogen content and hydrogen profile
Disk Rupture Tests

• vary strain rate
• compare effect of nitrogen vs. effect of hydrogen
• vary surface finish
Disk Rupture tests

Schematic of Disk Pressurizing Assembly

- Clamping Bolt
- Vent
- Clamping Washer
- Disk Sample Under Pressure
- Gas

Original page is of poor quality.
## Interim Results

### Disk Rupture Tests

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Hydrogen</th>
<th>Nitrogen</th>
</tr>
</thead>
<tbody>
<tr>
<td>50psi/20sec</td>
<td>0.16in/.85ksi *</td>
<td>0.22in/1.6ksi</td>
</tr>
<tr>
<td>50psi/200sec</td>
<td>0.2in/1.15ksi *</td>
<td>0.19in/1.65ksi</td>
</tr>
<tr>
<td>50psi/300sec</td>
<td>0.14in/.7ksi</td>
<td>0.18in/1.45ksi</td>
</tr>
<tr>
<td>50psi/20sec (60 grit)</td>
<td>0.15in/.6ksi</td>
<td>= = = = = = = =</td>
</tr>
<tr>
<td>50psi/200sec (60 grit)</td>
<td>0.18in/.8ksi</td>
<td>= = = = = = = =</td>
</tr>
<tr>
<td>50psi/300sec (60 grit)</td>
<td>0.13in/.6ksi</td>
<td>= = = = = = = =</td>
</tr>
</tbody>
</table>

* Leaked instead of rupture
Typical Failures for the Disk Rupture Tests
The strain rate had no effect in nitrogren
Surface roughness decreased failure pressure
Surface roughness results in burst type failure
Intermediate strain rate
Minimized hydrogen embrittlement at

Disk Rupture Tests
Interim Results
Tensile Tests

- charged and uncharged
- vary $\sigma_H$
- vary temperature
- vary gas pressure
Tensile Tests
Interim Results

*Tensile tests*

<table>
<thead>
<tr>
<th>Angle</th>
<th>Envir.</th>
<th>UTS, N/mm²</th>
<th>TD, mm</th>
<th>Et, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 deg</td>
<td>Air</td>
<td>500</td>
<td>1.626</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>LN₂</td>
<td>528</td>
<td>1.321</td>
<td>2.8</td>
</tr>
<tr>
<td>45 deg</td>
<td>Air</td>
<td>456</td>
<td>1.232</td>
<td>2.6</td>
</tr>
<tr>
<td></td>
<td>LN₂</td>
<td>489</td>
<td>1.016</td>
<td>2.1</td>
</tr>
<tr>
<td>90 deg</td>
<td>Air</td>
<td>516</td>
<td>1.626</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>LN₂</td>
<td>546</td>
<td>1.854</td>
<td>3.9</td>
</tr>
</tbody>
</table>
Interim Results

Tensile Tests

Air
LN₂

UTS (N/mm²)

0°  45°  90°

Angle
Tensile Test Specimen at 45° Fractography
Interim Results

Tensile tests

- Greatest UTS for 90, lowest for 45.
- No difference between room temperature and low temperature.
- Fracture initiation close to the hole and rapid propagation.
- Ductile fracture only for 45, and between the holes.
Aging Experiments

- Aging curves for 2090 T3 & W51
- X-Ray analysis
Aging curve of 2090 T3 at 170 C

Times in hours: 8 12 16 20 24 28 32 36

Log (time in minutes)

Maximum
Average
Minimum

Micronhardness Vickers
Aging curve of 2090 W51 at 170 C

Times in hours

Log (time in minutes)

Microhardness Vickers

Maximum - Average * Minimum
Aging Conditions for 2090 T3 & W51

- 16 hrs at 170 C for 2090 T3
- 16 hrs at 170 C for 2090 W51
X-Ray results

INTENSITY FOR 2219 AL-CU ALLOY

INTENSITY FOR 2219 AL-CU ALLOY

LEGEND:  - - AS QUENCHED  ● - ● FIRST PEAK  ▲ - ▲ SECOND PEAK
**X-Ray results**

The shift corresponds to a variation of the lattice parameter of:

- $8.9 \times 10^{-4}$ for the 1st peak
- $6.4 \times 10^{-4}$ for the 1st valley
- $7.9 \times 10^{-4}$ for the 2nd peak
Summary

- *Disk Rupture tests:*
  Rough surface $\Rightarrow$ burst failure.
  Intermediate strain rate $\Rightarrow$ less embrittlement.

- *Tensile tests:*
  45 $\Rightarrow$ lower ductility.
  No apparent difference at low temperature.
Summary
(Cont.)

- **Charpy impact tests:**
  Nearly same impact initiation energy for all orientations.
  Higher propagation energy for L-S and T-S than for T-L and L-T orientations.
  Substantial tearing for T-S and L-S orientations.

- **Charging solutions:**
  Two give embrittlement without surface damage.
Hydrogen Embrittlement
Need to Address

• Orientation of samples for the mechanical tests

• Additional material needed:
  - 2219
  - 2090
  - 8090
  - Weldalite

• 2090 T83 or T84 ??
Inventory

- 2091 T3: - 1/2"x5.9"x13.5"
  - 1/4"x11.8"x31.5"
  - 1/10"x15.7"x39.4"

- 2090 W51: 1/2"x12"x14"

- 2219 T87: 1/4"x12"x36"
Hydrogen Embrittlement
Future work

- Confirmation of SIMS results and quantification of hydrogen content
- Mechanical tests on: 2090 2091 2219
- Fractography