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

F. C. Rivet, Dr. R. E. Swanson

Department of Materials Engineering
Virginia Polytechnic Institute and State University

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 \( \text{As}_2\text{O}_3 \). 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

F.C. Rivet, M.S. Student
Dr. R.E. Swanson, Principal Investigator

Virginia Polytechnic Institute & State University
Dept of Materials Engineering
Blacksburg, Va 24061
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.
Instrumental Scheme

Printer

Computer

Potentiostat/Galvanostat

Electrometer

R.E.  W.E.  C.E.
Optical Profilometer

Diagram:

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

Anodic charging

Cathodic charging
Optical Profilometer (Results)

RMS: 0.186um
RA: 0.139um
P-V: 3.76um

Surface

Inverted
SIMS Results
**SIMS Results**

SIMS PROFILE
XR = 2.0, YR = 2.0
4.0 V, 5.0 V, CN = 500
FILM: 012500
H2 TAE 81 AND SAMPLE

SPUTTER TIME (MIN.)

CO-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>1 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
# 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.

Rough surface decreased failure pressure.

Rough surface 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

Schematic of Two-Hole Flat Tensile Specimen

\[ \sigma_{ij} = f(\theta) \]

\[ \varepsilon_{ij} = g(\theta) \]
# Interim Results

## Tensile tests

<table>
<thead>
<tr>
<th>Angle</th>
<th>Envir.</th>
<th>UTS, N/mm²</th>
<th>TD, mm</th>
<th>Ef, %</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

![Bar Chart](chart.png)

- **UTS (N/mm²)**
- **Angle (°): 0°, 45°, 90°**
- **Legend:**
  - Air
  - LN₂
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): 25, 27, 28, 29, 31, 32, 33, 34, 35

Original page is of poor quality
Aging curve of 2090 W51 at 170 C

Times in hours 8 12 16 20 24 28 32 36

Log (time in minutes)

Microhardness Vickers

* Maximum  —— Average  * Minimum

Vickers

180

170

160

150

140

2.5 2.6 2.7 2.8 2.9 3 3.1 3.2 3.3 3.4 3.5
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

ORIGINAL PAGE IS OF POOR QUALITY
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
- Fractography