

**Program 6    Deformation and Fracture of Aluminum-Lithium Alloys:    The Effect  
of Dissolved Hydrogen**

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**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  $As_2O_3$ . 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

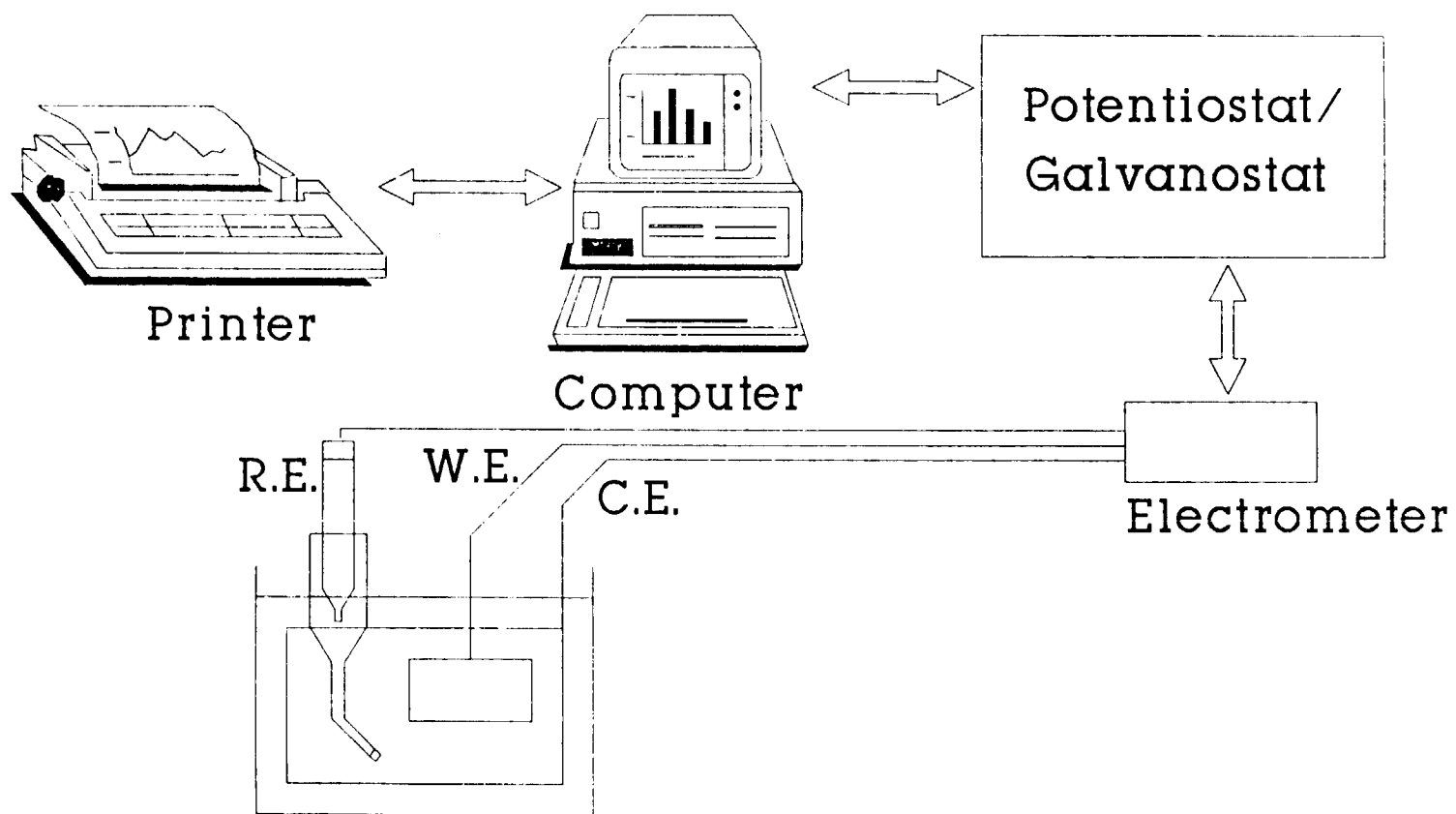
Two principal methods can be used to charge samples:

- Autoclave
- Electrochemical cell

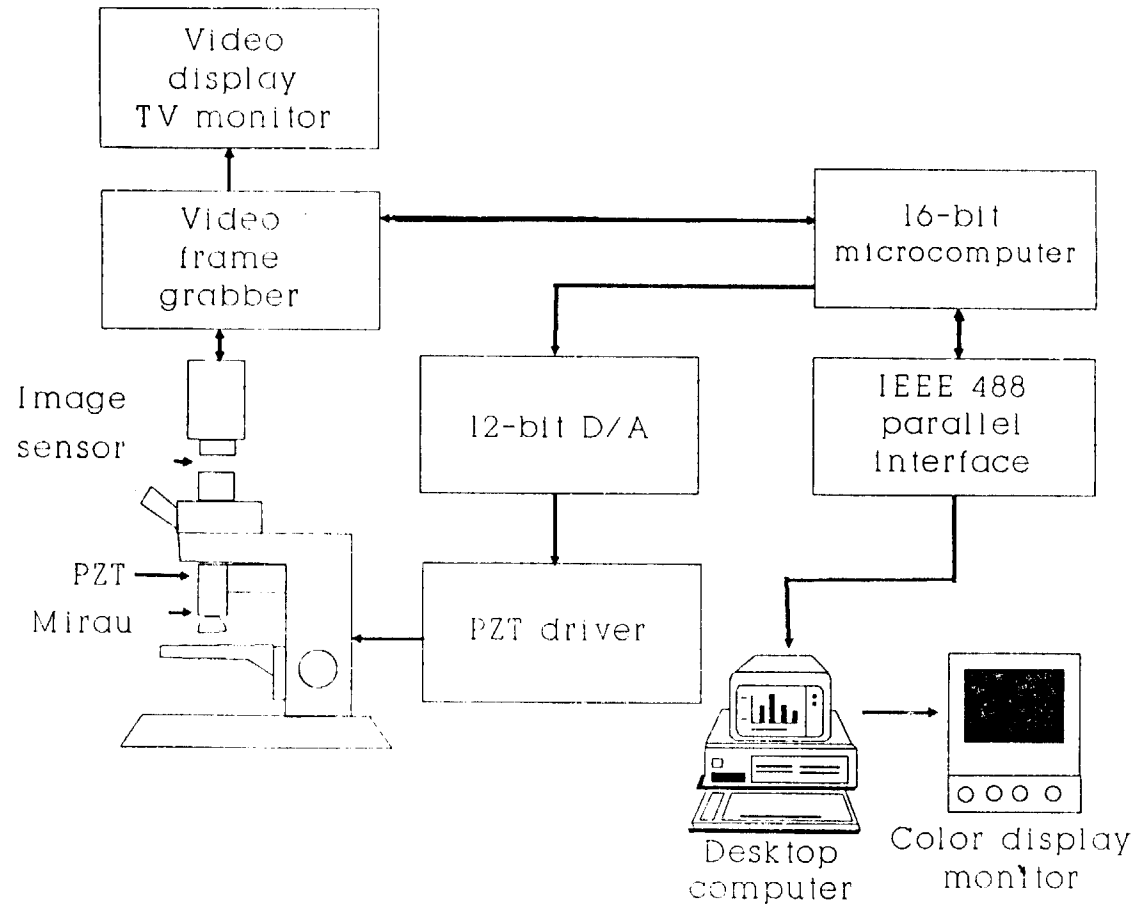
## *Choice of the Aqueous Solution*

- Must contain H<sup>+</sup>  
=> Low pH.
- Must not damage the sample  
=> Choice of the charging voltage  
or current.

# Instrumental Scheme

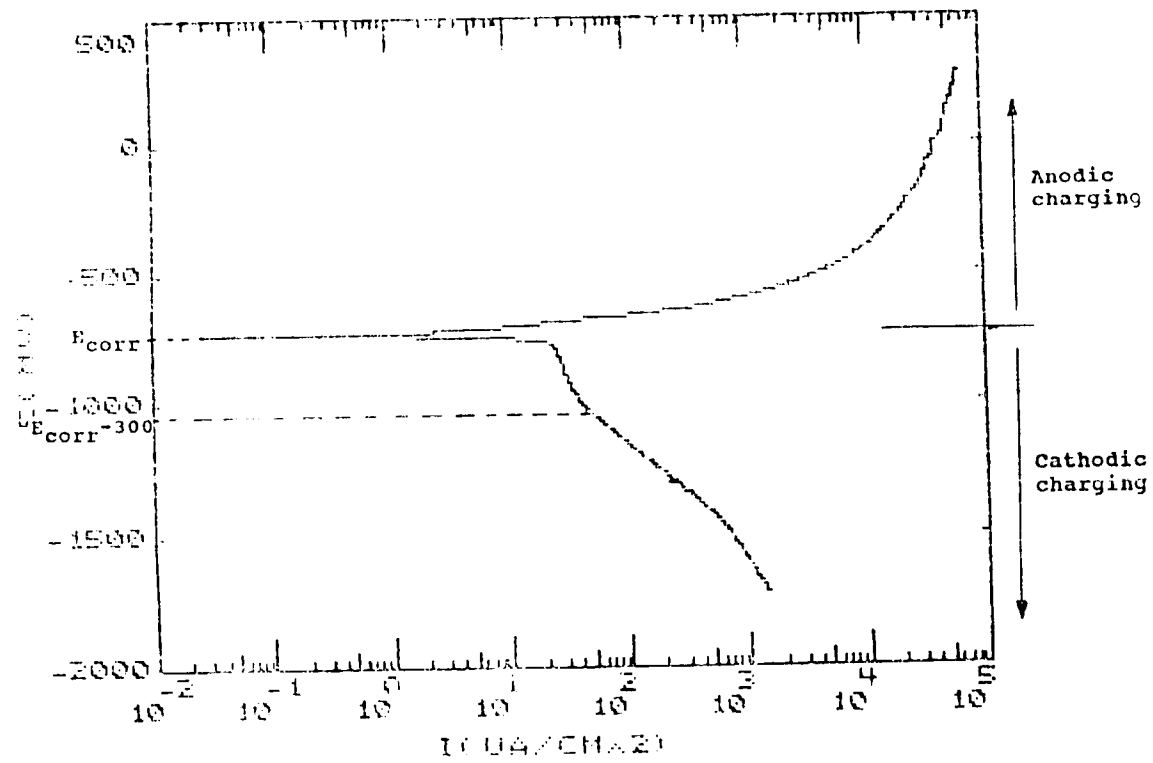


# Optical Profilometer



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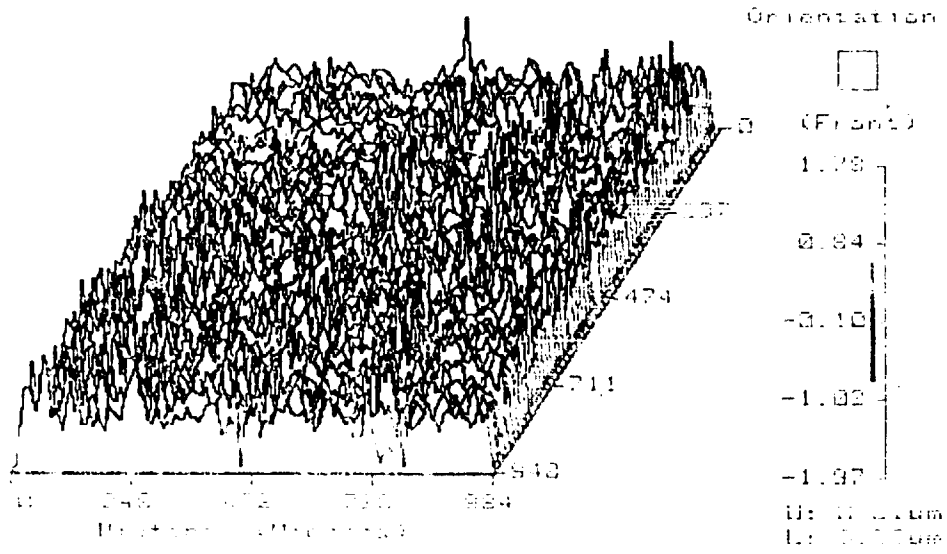
# Choice of the Voltage



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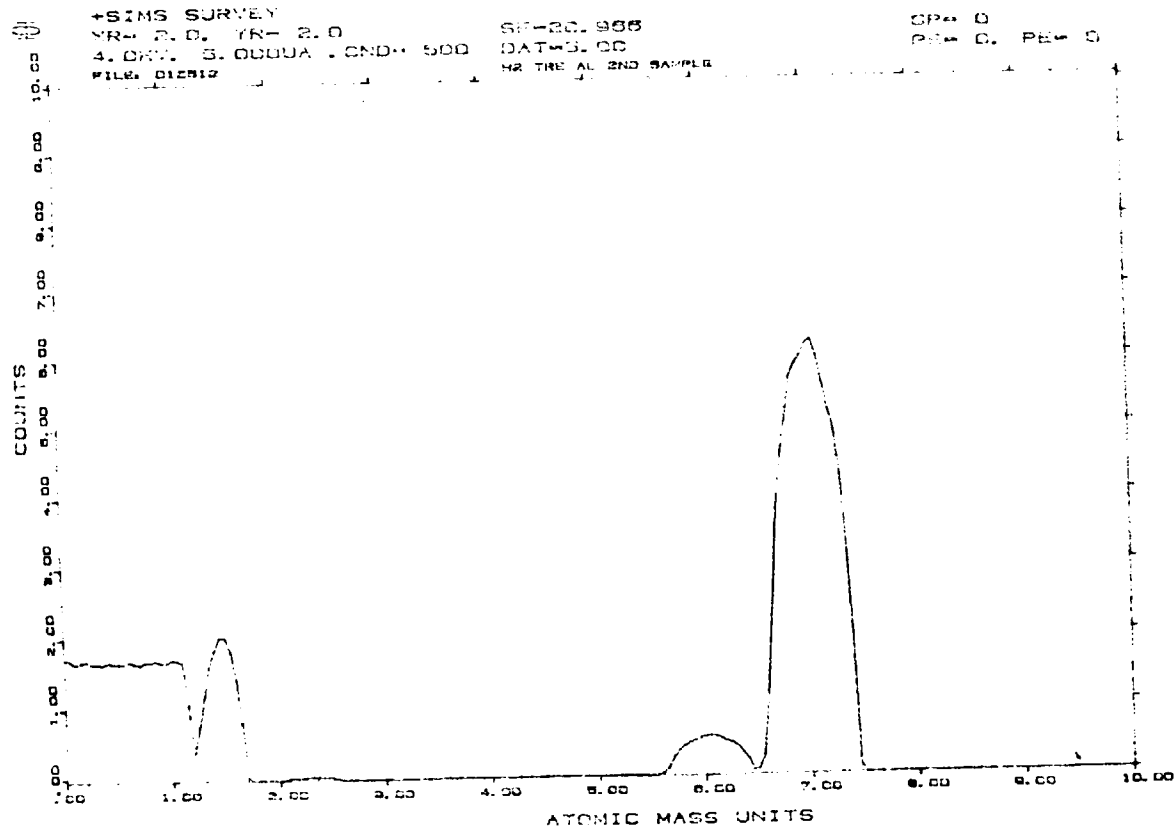
# Optical Profilometer (Results)

10.11  
RMS: 0.186um      SURFACE      AVEEN: 59.18um  
RA: 0.139um      INVERTED      P. Dev: -2.529m  
P-V: 3.76um      P. CV: 3.312m  
76.10°



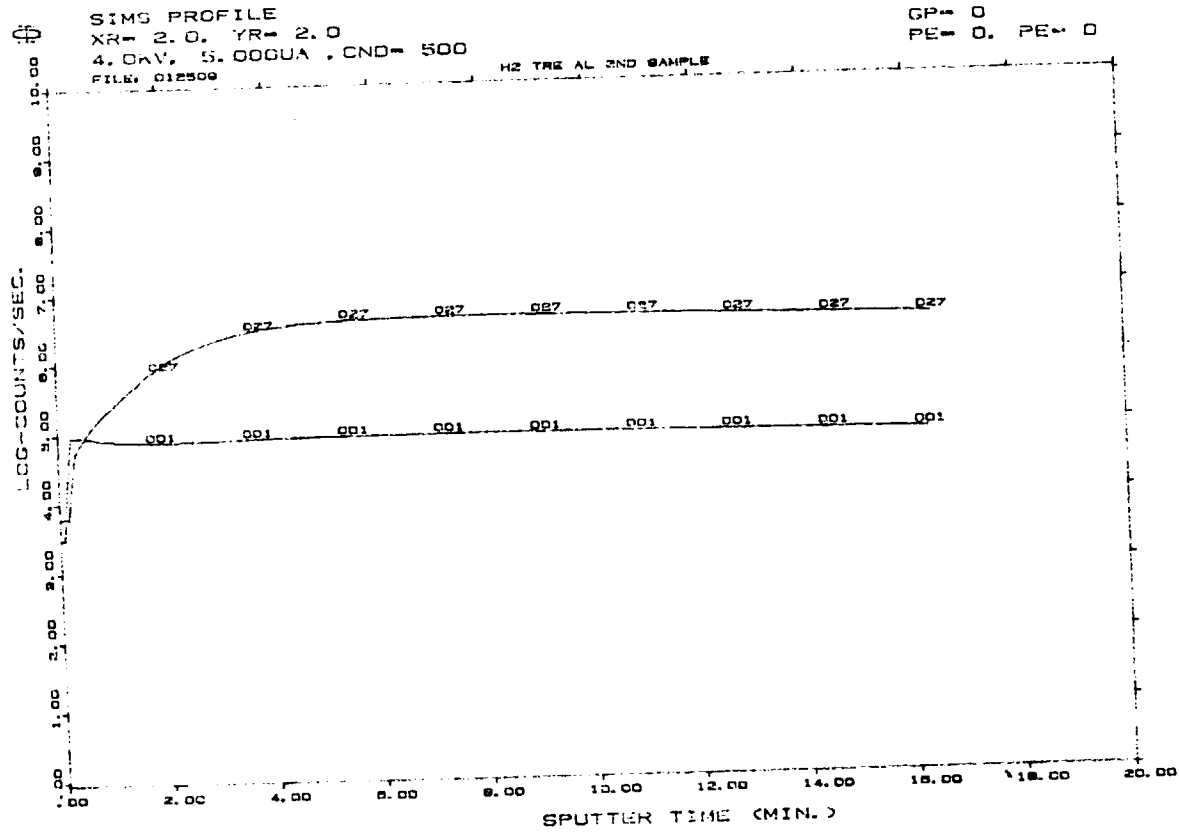
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# SIMS Results



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# SIMS Results



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# Interim Results

## Hydrogen Charging Parameters

- 0.04 N HCL + As<sub>2</sub>O<sub>3</sub> at -3V (1)
- 0.1 N NaOH + As<sub>2</sub>O<sub>3</sub> at -3V (2)
- 0.04 N HCl + As<sub>2</sub>O<sub>3</sub> at -500  $\mu$ A (3)
- 0.04 N HCl + As<sub>2</sub>O<sub>3</sub> at -5000  $\mu$ A (4)

# Interim Results

## Hydrogen Charging Parameters

Solution	Time	Dif. of counts/sec	H content	Surface Roughness RMS ( $\mu\text{m}$ )
(1)	5 hrs	<b>0.057</b>		<b>0.0795</b>
(2)	.5 hrs	-----		<b>0.185</b>
(3)	20 hrs	<b>0.059</b>		<b>0.0772</b>
(4)	20 hrs	<b>0.0185</b>		<b>0.0861</b>
Uncharged	-----	-----		<b>0.0752</b>

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# Interim Results

## Hydrogen Charging Parameters

The two selected charging solutions are:

- 0.04 N HCl+As<sub>2</sub>O<sub>3</sub> at -3 V for 5 hrs
- 0.04 N HCl+As<sub>2</sub>O<sub>3</sub> at -500  $\mu$ A for 20hrs

## Charging Experiments

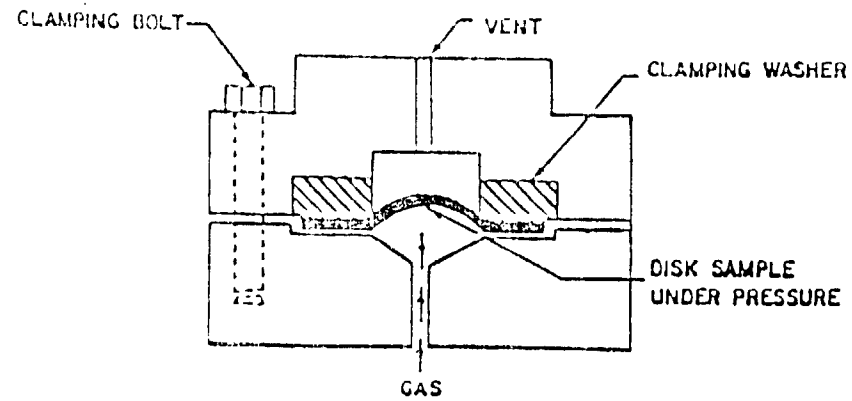
- SIMS technique has not yet been successful
- Evaluating other surface analytical techniques for hydrogen content and hydrogen profile

# 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



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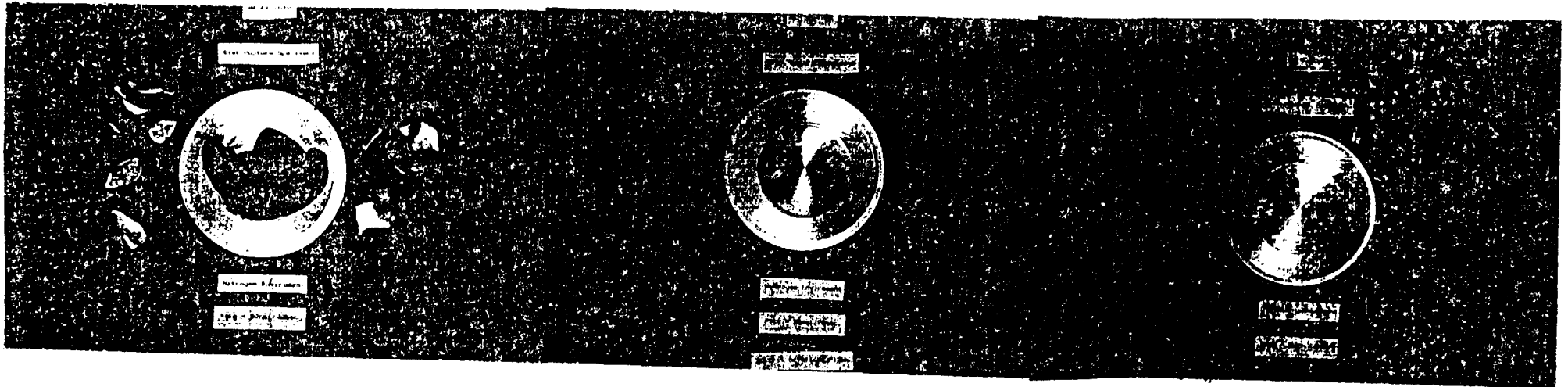
# Interim Results

## Disk Rupture Tests

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

\* Leaked instead of rupture

# Typical Failures for the Disk Rupture Tests



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# Interim Results

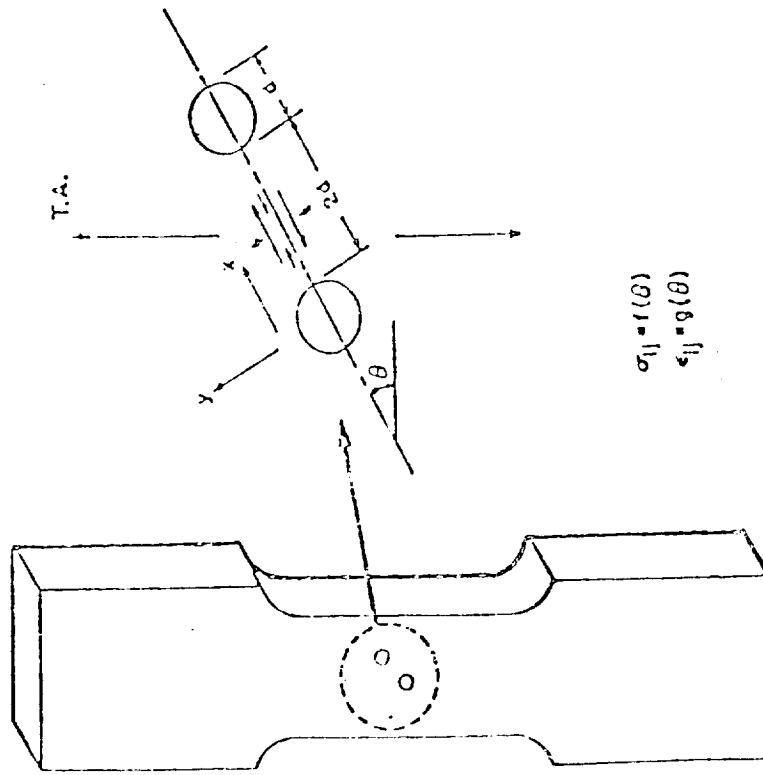
## Disk Rupture Tests

- Minimized hydrogen embrittlement at intermediate strain rate.
- Rough surface results in burst type failure
- Rough surface decreased failure pressure
- The strain rate had no effect in nitrogen

# Tensile Tests

- charged and uncharged
- vary  $\sigma_H$
- vary temperature
- vary gas pressure

# Tensile Tests



$$\sigma_{11} = r(\theta)$$
$$\sigma_{22} = q(\theta)$$

Schematic of Two-Hole Flat Tensile Specimen

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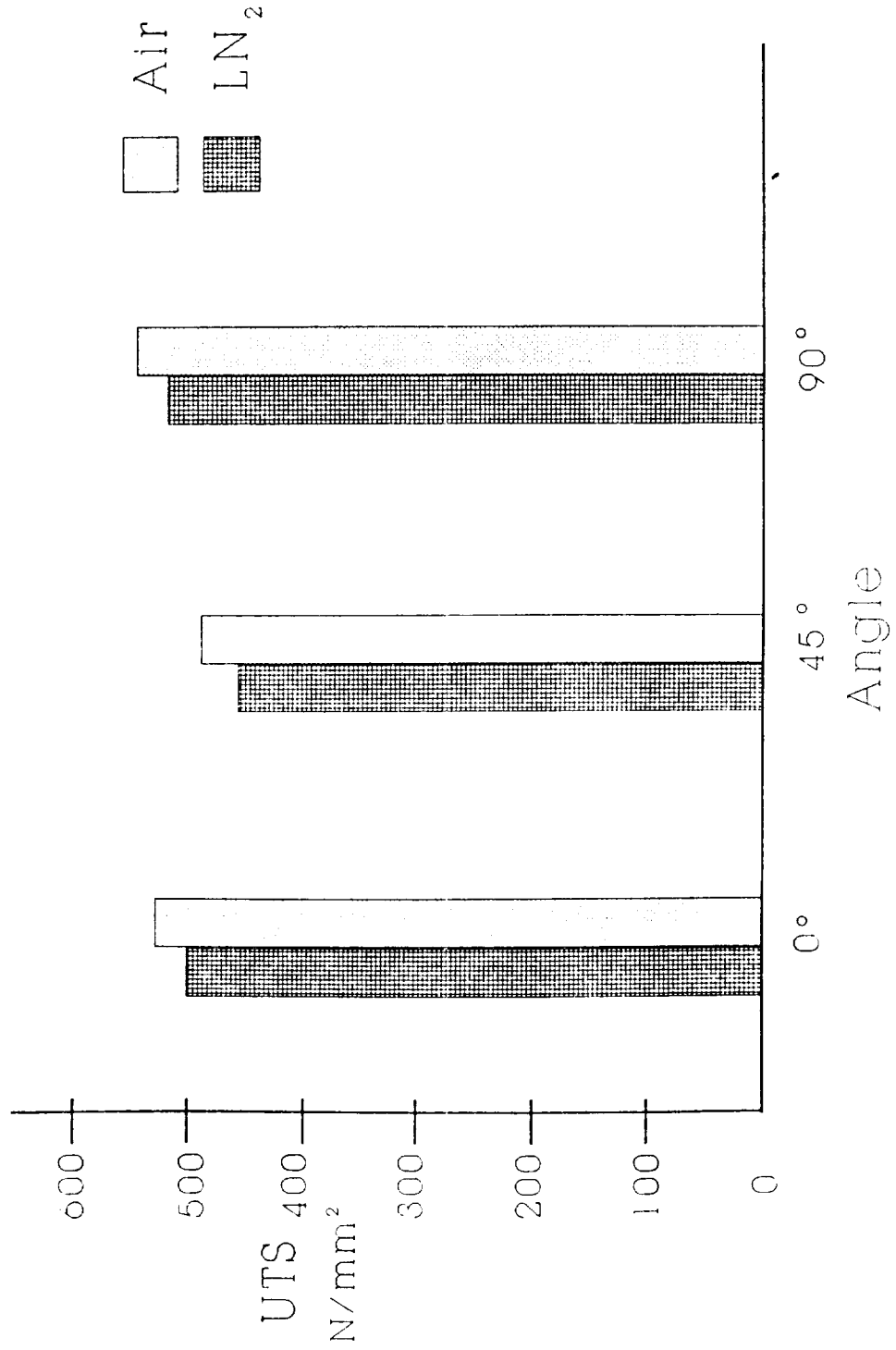
# Interim Results

## Tensile tests

Angle	Envir.	UTS, N/mm <sup>2</sup>	TD, mm	Ef, %
0 deg	Air	500	1.626	3.4
	LN <sub>2</sub>	528	1.321	2.8
45 deg	Air	456	1.232	2.6
	LN <sub>2</sub>	489	1.016	2.1
90 deg	Air	516	1.626	3.4
	LN <sub>2</sub>	546	1.854	3.9

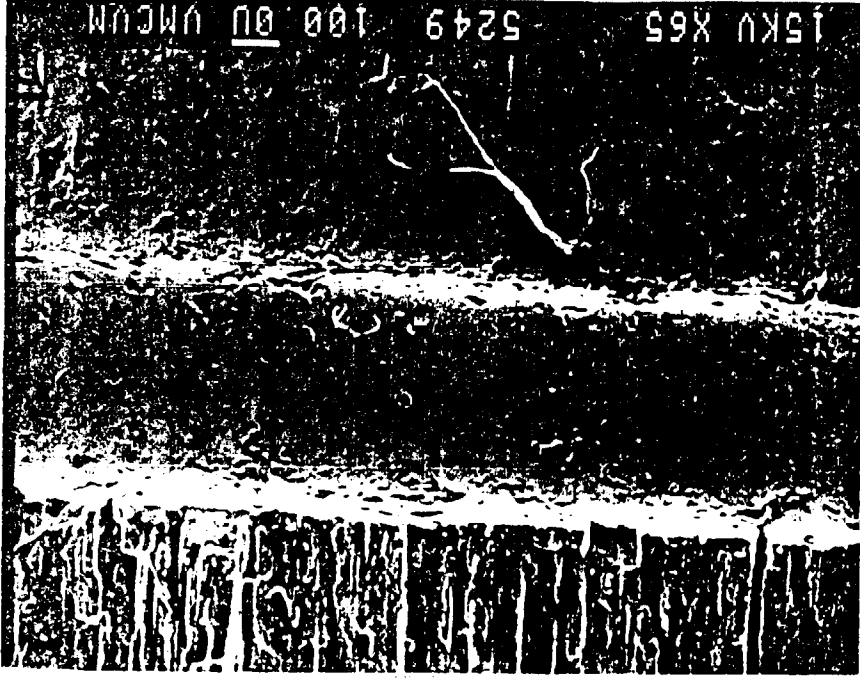
# Interim Results

## Tensile Tests



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# Fractography Tensile Test Specimen at 45



# Interim Results

## Tensile tests

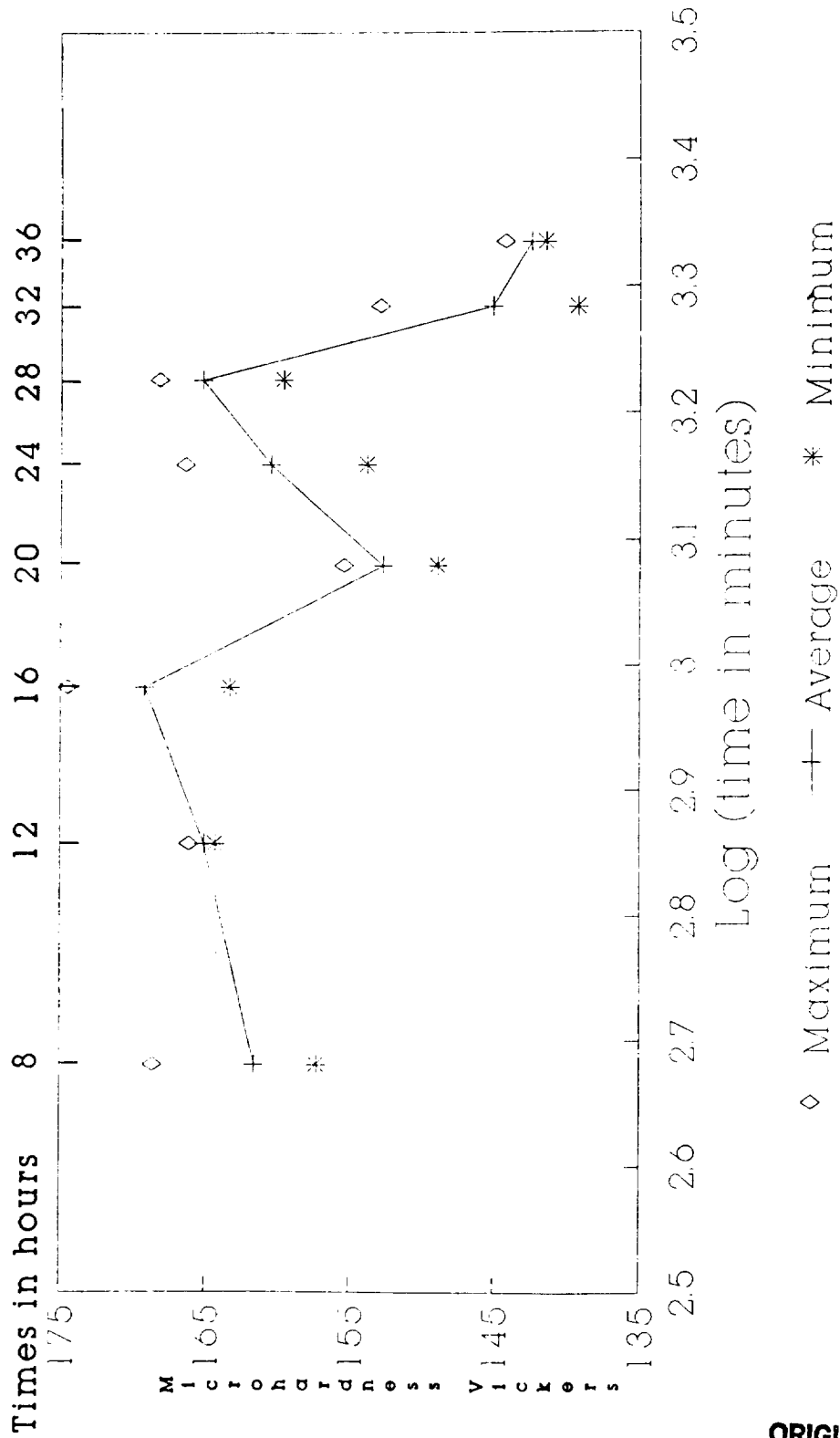
- Greatest UTS for 90 ,  
lowest for 45 .
- No difference between room temperature and  
low temperature.
- Fracture initiation close to the hole  
and rapid propagation.
- Ductile fracture only for 45 , and between  
the holes.

# Aging Experiments

- Aging curves for 2090 T3 & W51
- X-Ray analysis

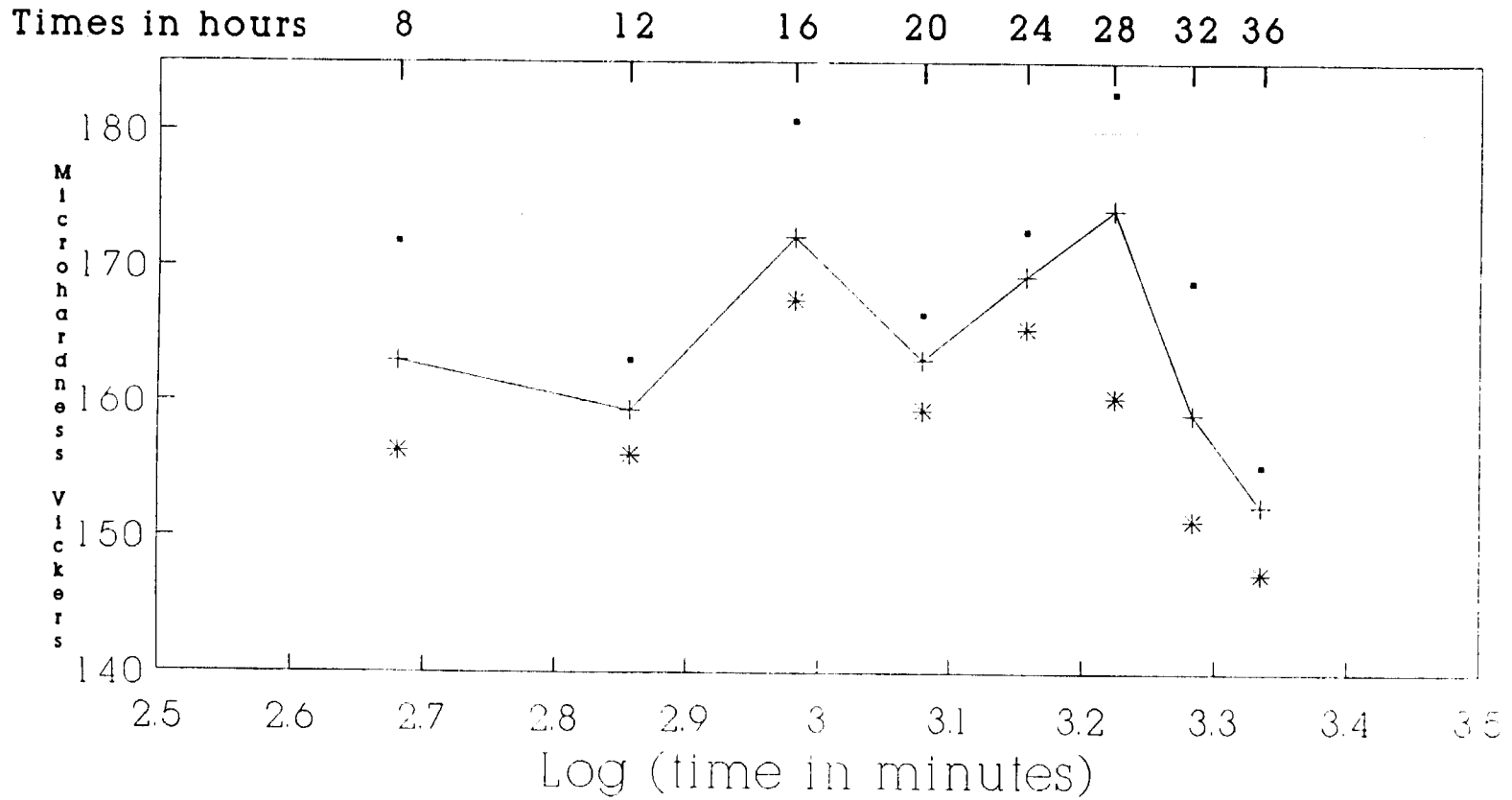


# Aging curve of 2090 T3 at 170 C



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# Aging curve of 2090 W51 at 170 C



• Maximum    —+— Average    \* Minimum

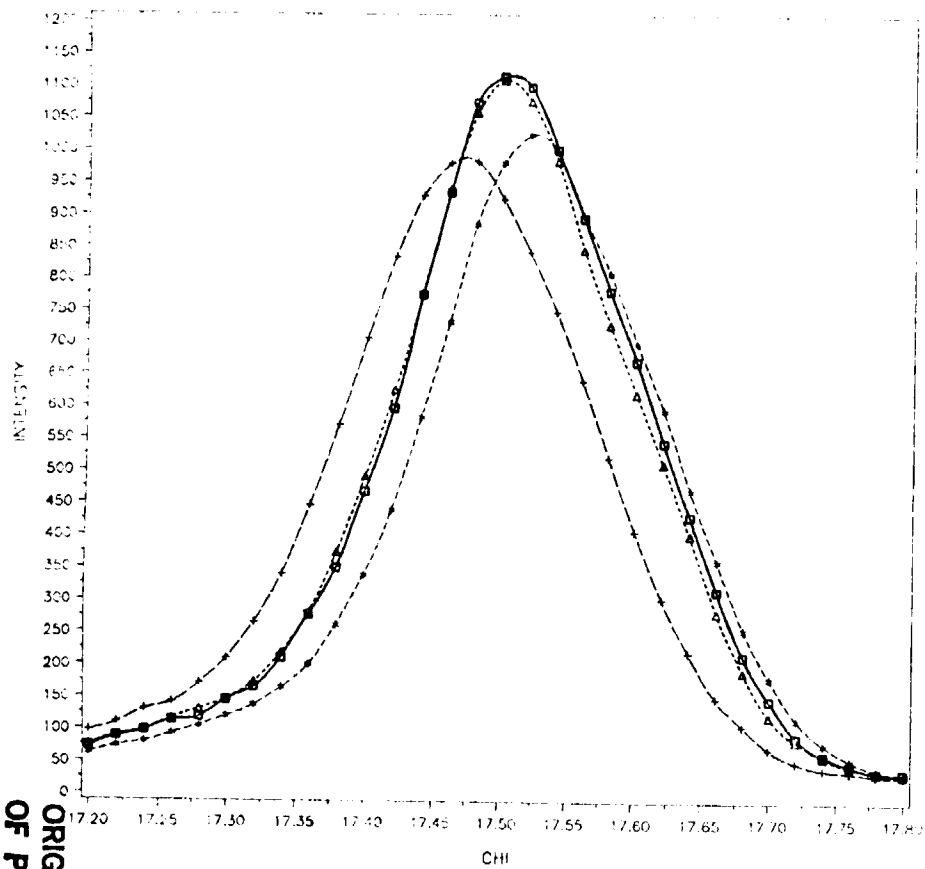
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*Aging Conditions for 2090 T3 & W51*

- 16 hrs at 170 C for 2090 T3
- 16 hrs at 170 C for 2090 W51

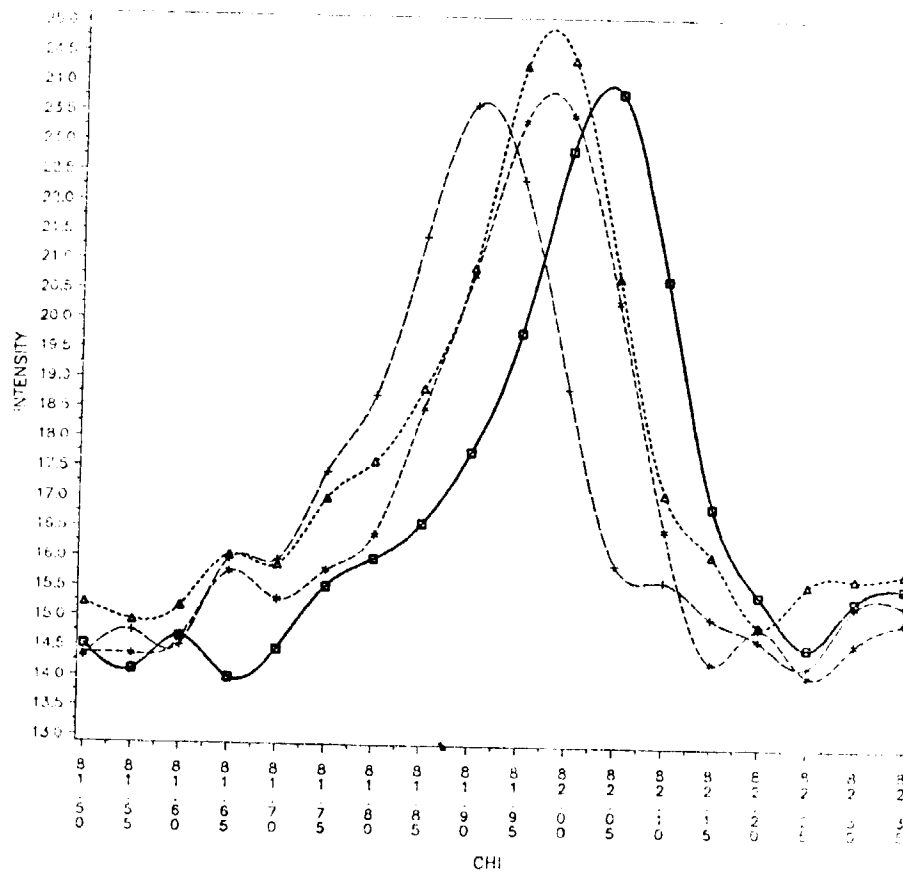
# X-Ray results

INTENSITY FOR 2219  
AL-CU ALLOY



LEGEND:   
 ○—○ AS QUENCHED   
 ▲---▲ FIRST VALLEY   
 +---+ SECOND PEAK

INTENSITY FOR 2219  
AL-CU ALLOY



LEGEND:   
 ○—○ AS QUENCHED   
 ▲---▲ FIRST VALLEY   
 +---+ SECOND PEAK

## *X-Ray results*

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

$8.9 \cdot 10^{-4}$  for the 1st peak

$6.4 \cdot 10^{-4}$  for the 1st valley

$7.9 \cdot 10^{-4}$  for the 2nd peak

# Summary

- Disk Rupture tests:

Rough surface => burst failure.

Intermediate strain rate => less embrittlement.

- Tensile tests:

45 => 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