



**Savannah River
National Laboratory™**

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Effect of Tritium on Cracking Threshold in Aluminum 7075

Tritium 2016

11th International Conference on Tritium Science & Technology

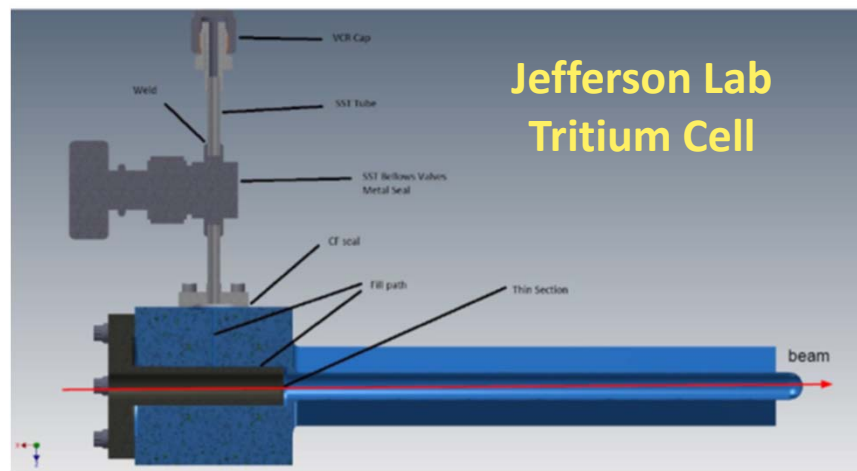
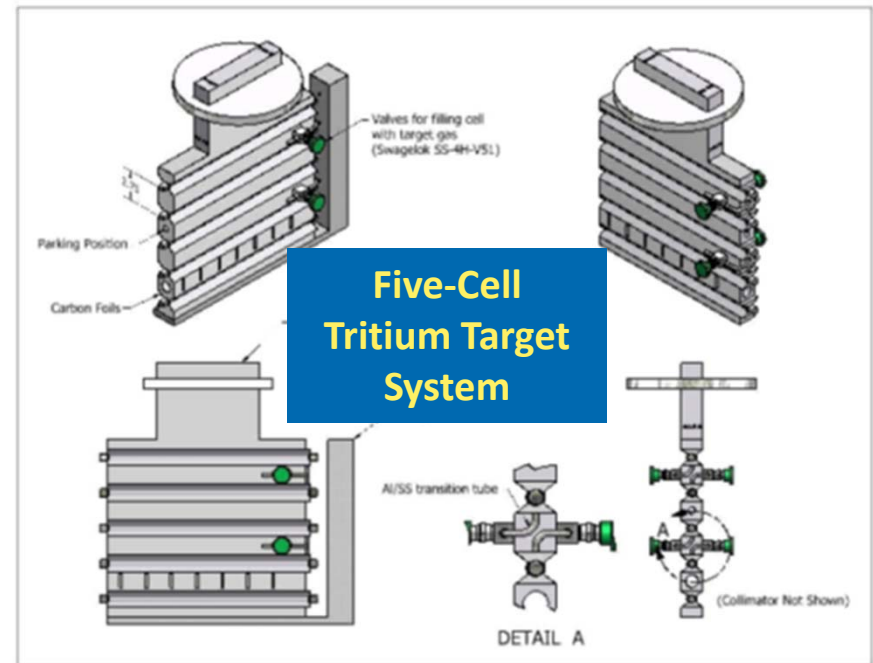
April 17-22, 2016
Charleston Marriott
Charleston, SC

Andrew J. Duncan and Michael J. Morgan

Savannah River National Laboratory: Savannah River Site, Aiken, SC

Purpose and Outline

- SRNS is supplying tritium gas for Physics experiments at Jefferson Labs
- SRNL is conducting experiments to provide additional support for Jefferson Lab safety
- The experiments are designed to measure cracking thresholds in tritium gas for 7075 Aluminum at conditions similar to, and greater than, the operating conditions of the Jefferson Labs tritium cell.
- SRNL interested in understanding tritium effects in aluminum and conducting in-situ testing.
- *Just 4 months into a 12 month experiment*

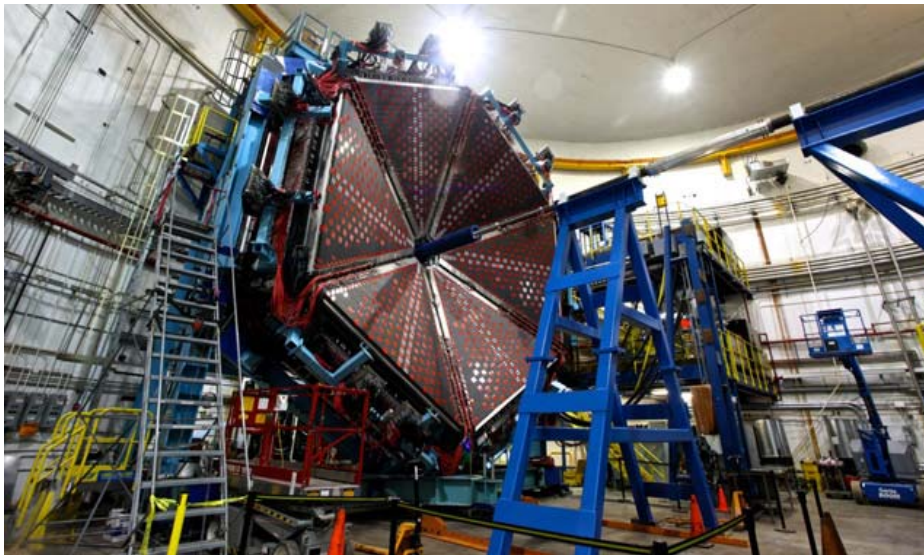


Outline

- Background – Jefferson Labs Experiment
- 7075 Aluminum Tritium Cell
- Effects of Hydrogen/Tritium on Al
- Experimental Approach and Project Plan
- Initial Results – 4 month tritium exposure
- Fracture Mechanics Analysis
- Preliminary Conclusions and Future Work



Jefferson Lab – Measuring the Properties of Nucleons



Proposal to Measure the EMC Effect in Deep Inelastic Scattering off the Tritium and Helium Mirror Nuclei

The JLAB Marathon Collaboration including Dr. David Meekins, of Jefferson Lab, Newport News, VA.

“The primary safety concern for the target is a tritium leak due to mechanical failure of windows due to hydrogen embrittlement, radiation damage, or loss of target integrity from accidental excessive beam heating due to failure of the raster or grossly mis-steered beam.”

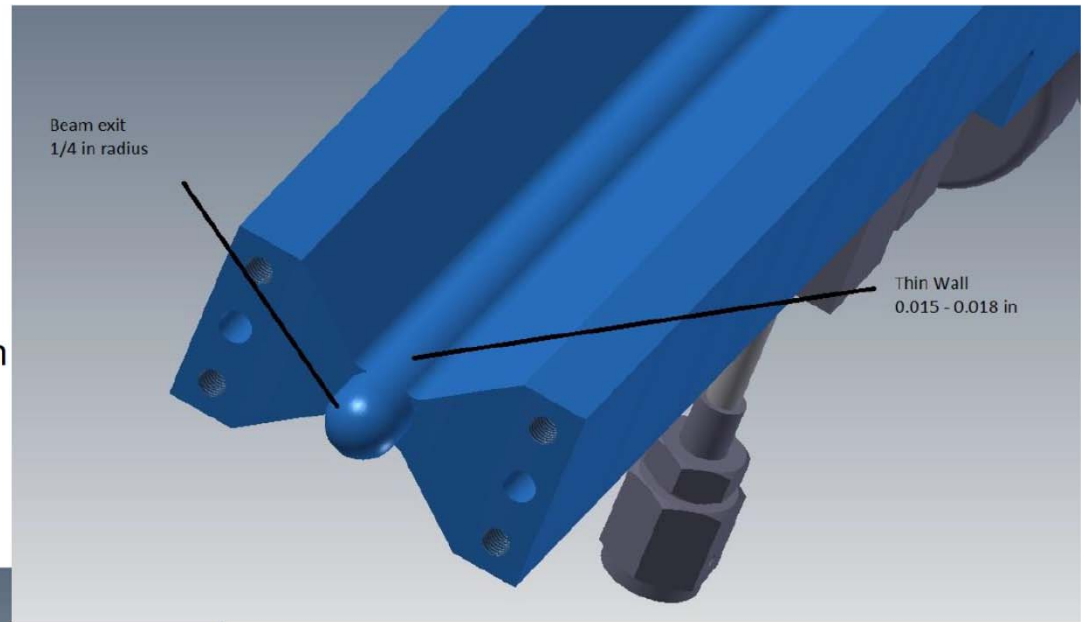
SRNS is providing tritium gas for the experiment and tritium effects data to support the safety basis for experiments designed to establish the internal quark structure of the nucleon.



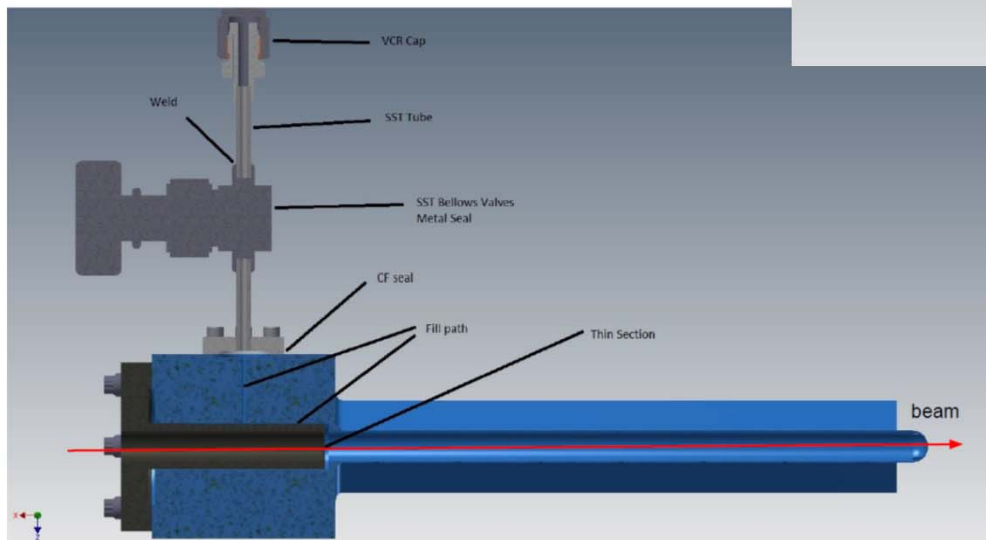
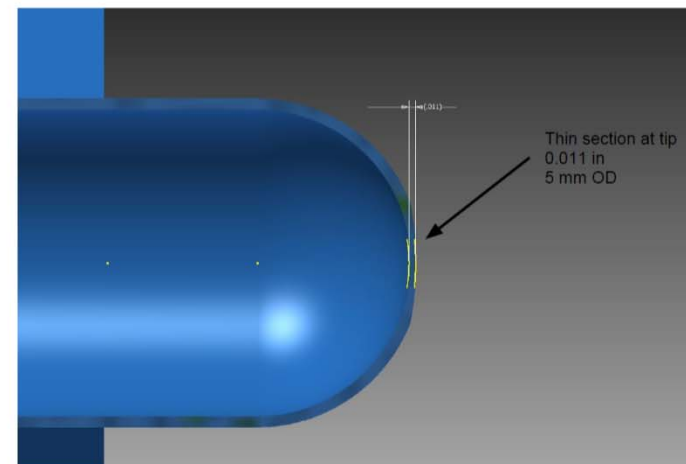
Proposal Awarded Pending "Management Review of Safety Aspects of Tritium Cell"

Experimental Program

- 2 approved experiments
 - Short Range Correlations in 2 and 3 Nucleon Systems
 - Deep Inelastic Scattering on ^3He and ^3H
- Both experiments aim to improve our understanding of the structure of the nucleon



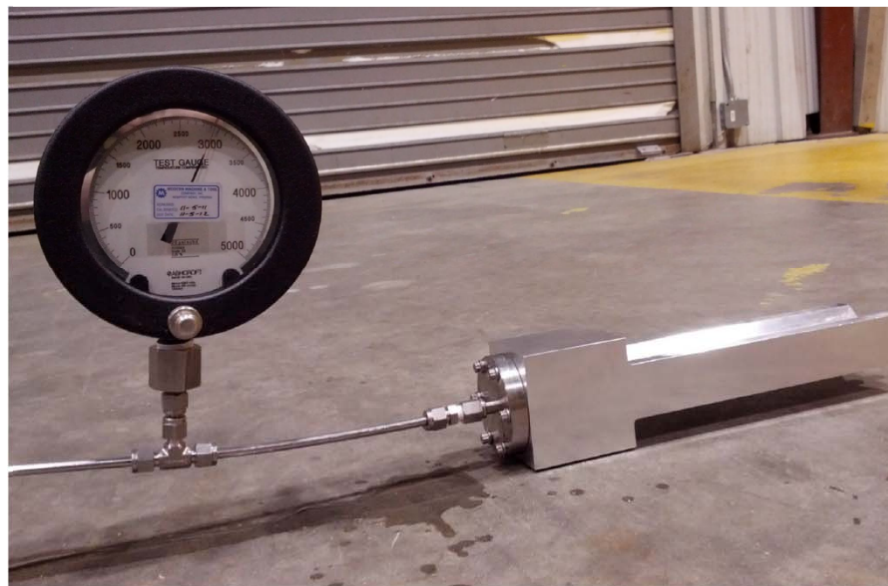
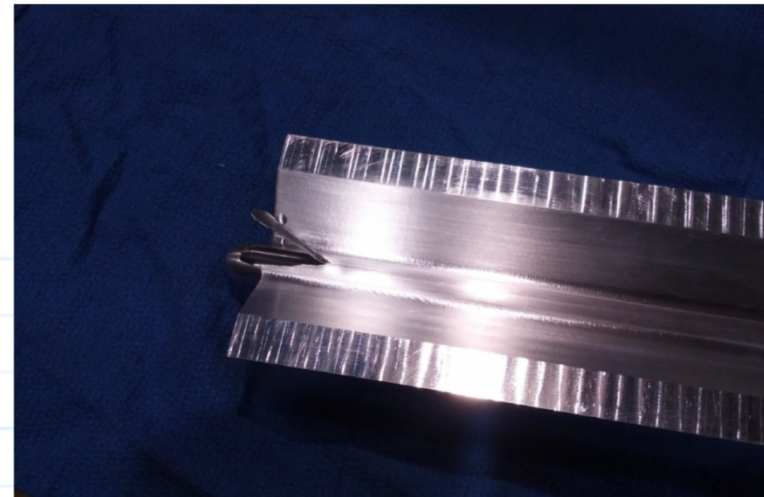
Thin Section



Pressure Estimates and Burst Test of Tritium Cell

Cell pressure at room temperature was estimated from an assumed fill of 1090 Ci (4.03 E13 Bq) of T2 at 100% purity to be 1.38 MPa:

$$P_{cell} := \frac{n_{T2} \cdot Z \cdot T_{room} \cdot R}{V_{cell}} = 199.766 \text{ psi}$$



Burst Pressure was ≥ 20 MPa

Pressure test of new cell

- Test cell wall thickness at thinnest point was 0.014 inch on side wall and 0.012 inch on dome.
 - Note: The actual cell requires 0.015 - 0.018 inch side wall thickness
- Main cell body burst pressure above 3500 psi
 - Factor of >10 for safety
 - Account for cyclic temperature/pressure loads
- Entrance windows
 - Entrance window thickness is 0.010 inch
 - Burst Pressure ~2900 psi

Effective Tritium Pressure for Beam-On Conditions is 21.5 MPa



Fugacity of tritium gas in cell during beam operations:

The beam will dissociate the T2 to T+T which will recombine on the walls of the cell.

this implies that the ratio of T atoms to aluminum atoms may be a factor of 10 larger for beam conditions. The higher concentration/diffusion rate may be considered to result from an effective pressure (temperature) or fugacity. This fugacity is determined below:

solubility is 20x higher

$$C_H := \frac{R_T}{R_{norm}} \cdot C_{150} = 1.455 \cdot 10^{-8}$$

$$P_{eff} := \left(\frac{C_H}{C_0 \cdot e^{\frac{-\Delta H}{R \cdot T_{room}}}} \right)^2 \cdot P_{atm} = (3.114 \cdot 10^3) \text{ psi} \quad \mathbf{3114 \text{ psi} = 21.5 \text{ MPa}}$$

A test in compliance with ASTM E399 shall be performed by SRSTE/SRNL with pressures in excess of 1000 psi. Samples shall be exposed to 4,8,12 months of T2 at room temp and more than 3000 psi. This exceeds the fugacity available by beam on conditions.

SRNL tritium exposures were conducted at 17.2 MPa (2500 psi) at ambient temperature



Composition and Properties of 7075 Aluminum

CHEMICAL COMPOSITION LIMITS (WT. %)

Si	0.40	Zn	5.1-6
Fe	0.50	Ti	0.20
Cu	1.2-2.0	Others, each	0.5
Mn	0.30	Others, total	0.15
Mg	2.1-2.9	Balance, Aluminum	
Cr	0.18-0.28		

Note: Value maximum if range not shown.

TYPICAL FRACTURE TOUGHNESS VALUES Alloy 7075 Plate

ALLOY	TEMPER	K _{IC} :ksi√in. (MPa√m)*	
		L-T	T-L
7075	T651	26 (28.6)	22 (24.2)
	T7351	30 (32.0)	26 (28.6)

*Compact specimen (ASTM E399)

MECHANICAL PROPERTIES

ALLOY 7075 All values are minimum long transverse mechanical properties except where noted.

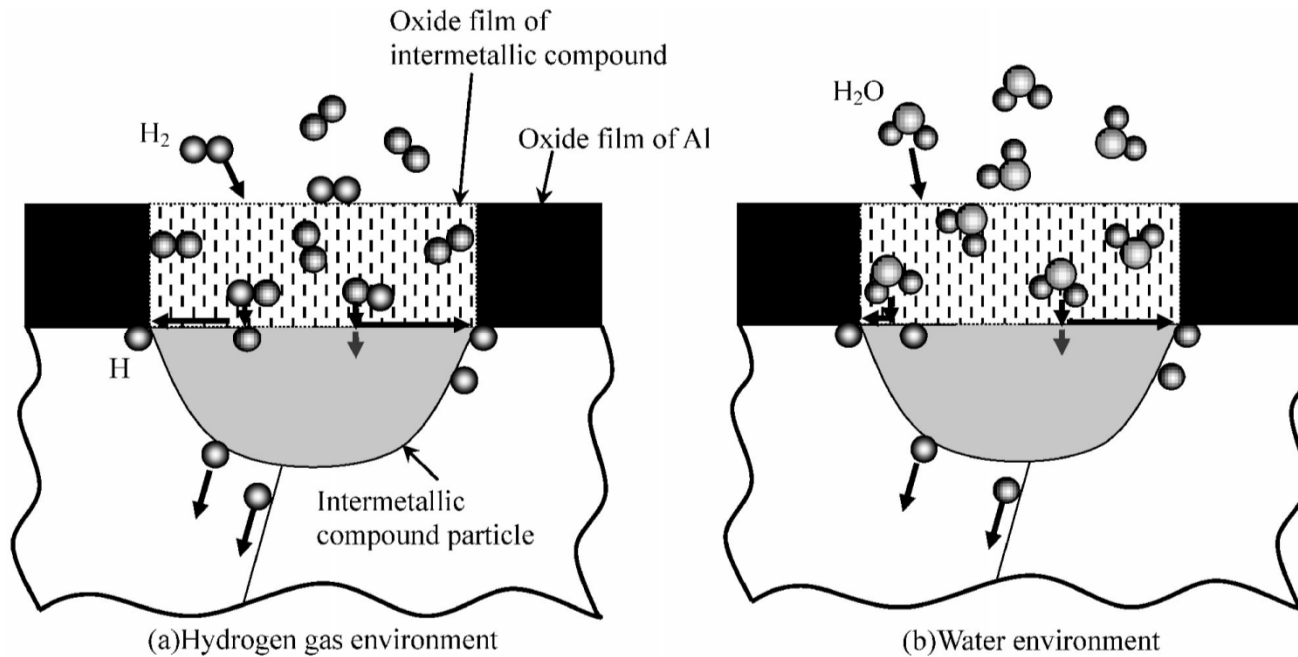
TEMPER	THICKNESS in. (mm)	TENSILE STRENGTH ksi (MPa)	YIELD STRENGTH ksi (MPa)	ELONGATION %
0 Sheet & plate	0.015-2.00 (0.38-50.80)	40 (max) (276)	21 (max) (145)	9-10
T6 Sheet	0.008-0.249 (0.203-6.32)	74-78 (510-538)	63-69 (434-476)	5-8
T651 Plate	0.250-4.000 (6.35-101.60)	78-67 (538-462)	67-54 (462-372)	9-3

We measured K_{IC} to be 24.2 MPa-√m, in agreement with alloy data sheets. Tritium effect on K_{IC}, i. e, K_{TH}, (Cracking Threshold) for 7075 Aluminum is unknown and being measured.



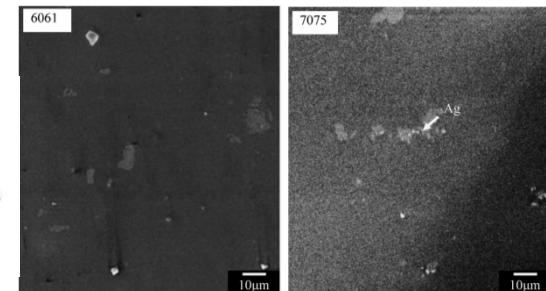
Aluminum Resistant to H₂ Embrittlement - Oxide Film Prevents Dissociation and Permeation

Exceptions – Intermetallic compound particles and water environments



In Jefferson Lab experiment, dissociated tritium atoms are available because of decay and in-beam conditions

Tritium Associated With Intermetallic Compounds in 7075 Aluminum Using Autoradiography*



J. Japan Inst. Met. Mater. Vol. 77, No. 12 (2013), pp. 565–570
Special Issue on Hydrogen and Materials Characteristic in Solids
© 2013 The Japan Institute of Metals and Materials

Tritium Autoradiography Study on Hydrogen Invading Aluminum from Different Environments

Takahito Watakabe^{1,*}, Goroh Itoh² and Yuji Hatano³



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Effect of Humidity and Stress Intensity on SCC Velocity of 7075 Aluminum*

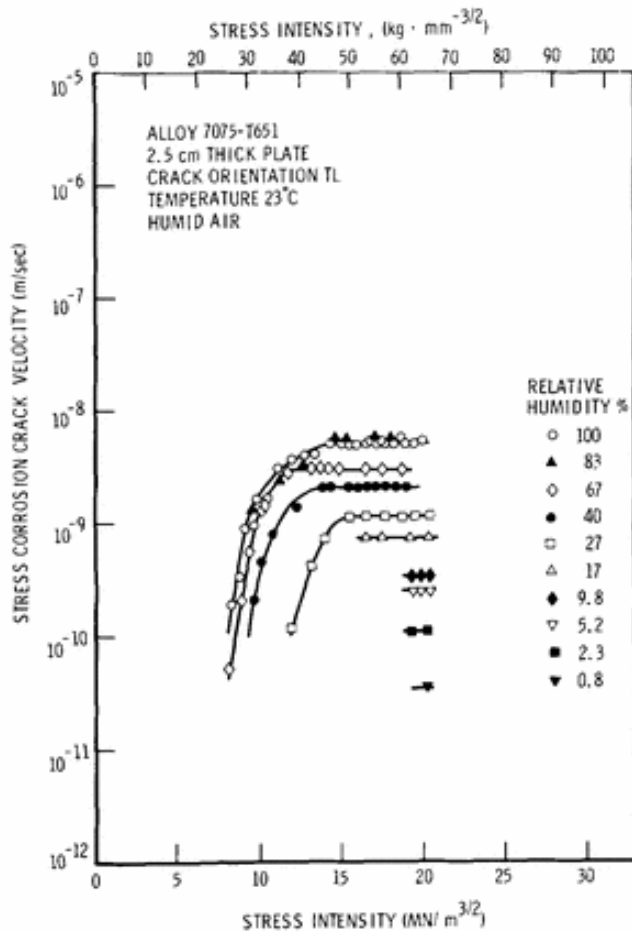
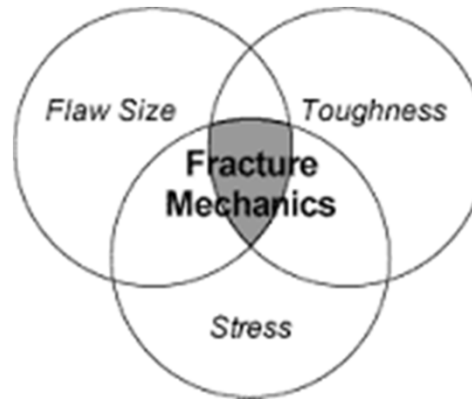
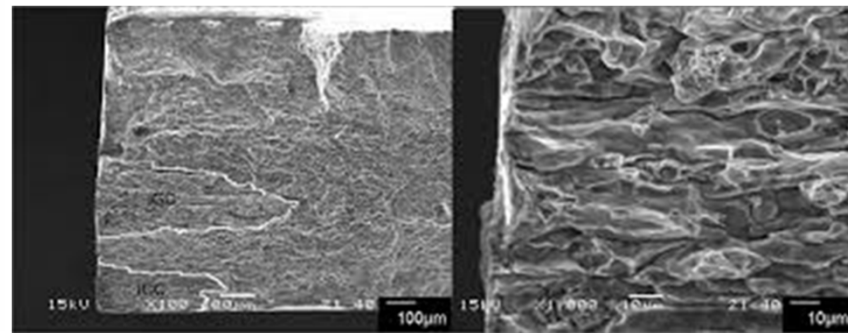


Fig. 5—Effect of humidity and stress intensity on stress corrosion crack velocity of a high-strength aluminum alloy in air.



Environmental Cracking Will Occur at a Critical Flaw Size, Stress Level, and Material Toughness.

Hydrogen Caused Cracks to Grow at Cracking Thresholds (KTH) between 7-10 MPa-√m. Fracture Mode Change to Intergranular Fracture



*Markus O. Speidel, *Met. Trans A Volume 6A April 1975 "Stress Corrosion Cracking of Aluminum Alloys"*

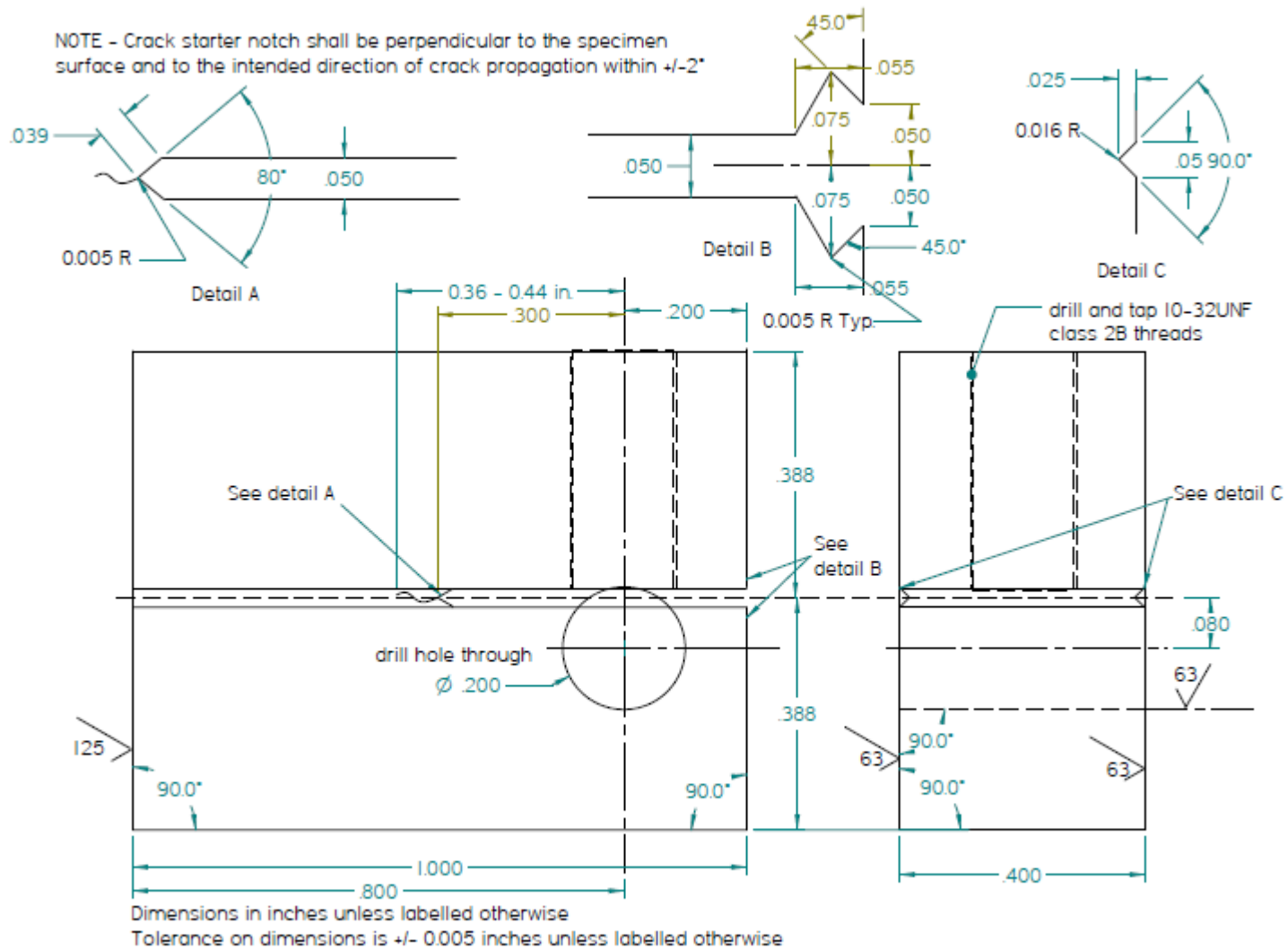


Experimental Procedure & Fracture Mechanics Approach

- Use ASTM E1681 to measure cracking thresholds in tritium gas using Jefferson Lab supplied 7075 Aluminum;
- Fatigue pre-crack specimens and bolt-load to low, medium and high values of stress intensity - corresponding to, and greater than, Jefferson Lab cell conditions;
- Place stressed samples in a pressure vessel and hold in 17.2 MPa (2500 psi) tritium gas for up to twelve months;
- Examine specimens for cracking after four, eight, and twelve months of exposure.
- Tests on companion specimens in moist hydrogen and air are planned to isolate the effects from hydrogen isotopes from decay helium.
- Post exposure characterization will be utilized to ascertain the primary mechanisms of embrittlement.
- Verify cracking thresholds are well below tritium cell stress intensities to ensure safe operating conditions.



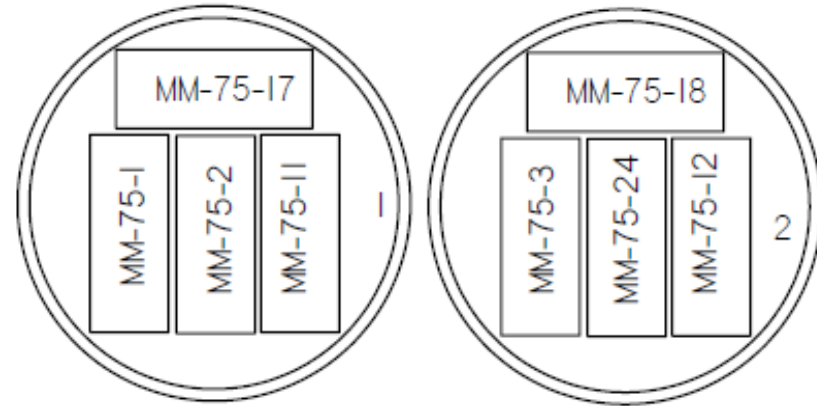
Details Wedge-Opening Loaded (WOL) Specimen (Dimensions in Inches)



Specimen Stress Intensity Levels and Crack Lengths for 4-Month Exposure Test

Specimens Were Stacked In Layers During Tritium Exposure:

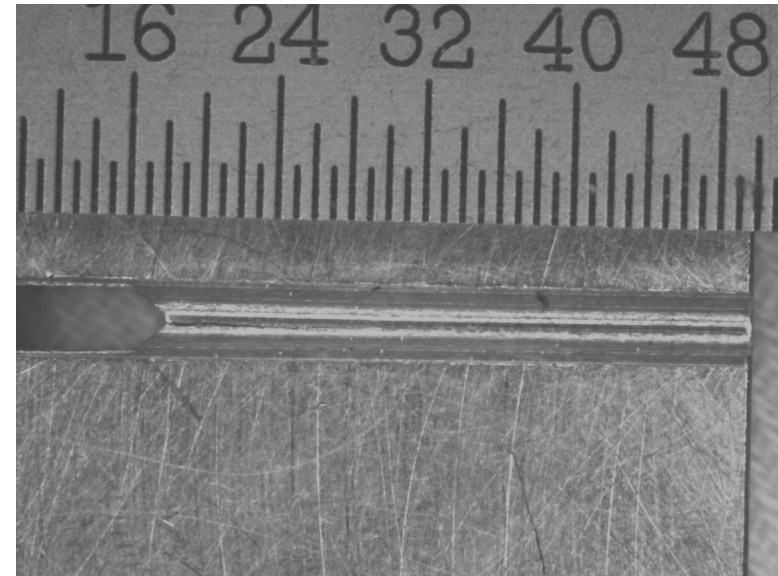
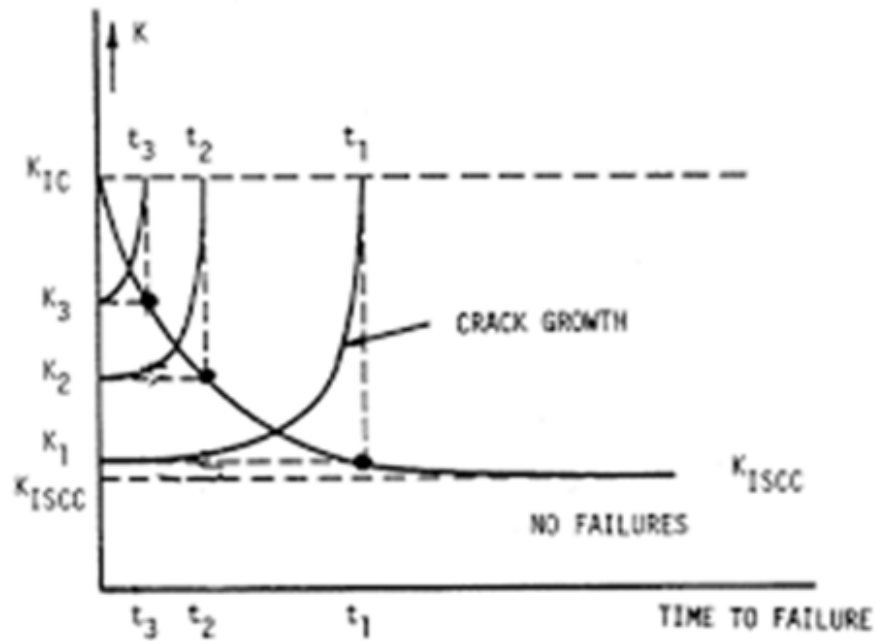
- Layers 1&2 Removed after 4 months
- Layers 3&4 Removed after 8 months
- Layers 5,6&7 Removed after 12 months



Sample	K MPa-√m	COD mm	a/w Initial	a/w Final
MM75-1	19806	.28	.59	.58
MM75-2	20346	.27	.54	.54
MM75-3	19428	.29	.62	.65
MM75-11	17869	.26	.60	.61
MM75-12	18197	.26	.58	.58
MM75-17	15029	.21	.59	.58
MM75-18	14937	.21	.58	.57
MM75-24	11664	.17	.60	.61



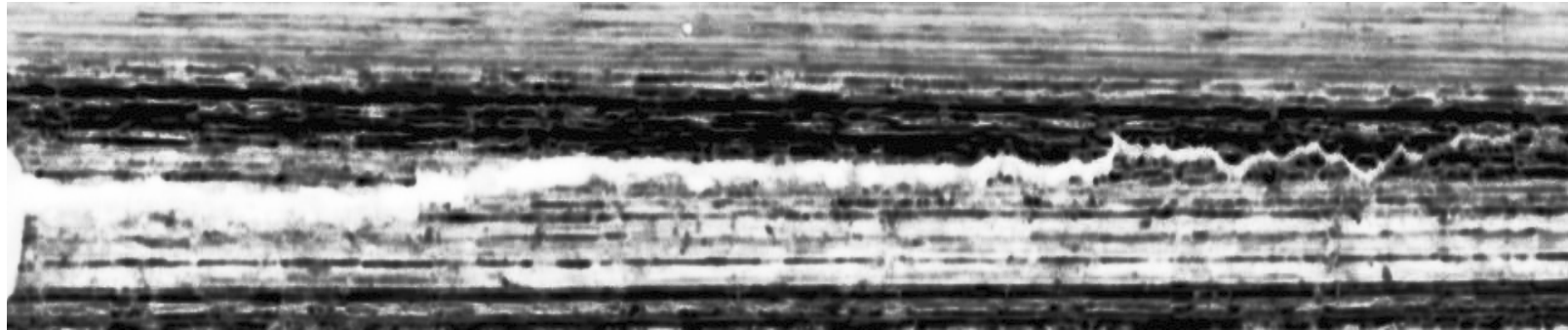
Specimens were Removed from Tritium Gas and Examined Optically for Crack Growth



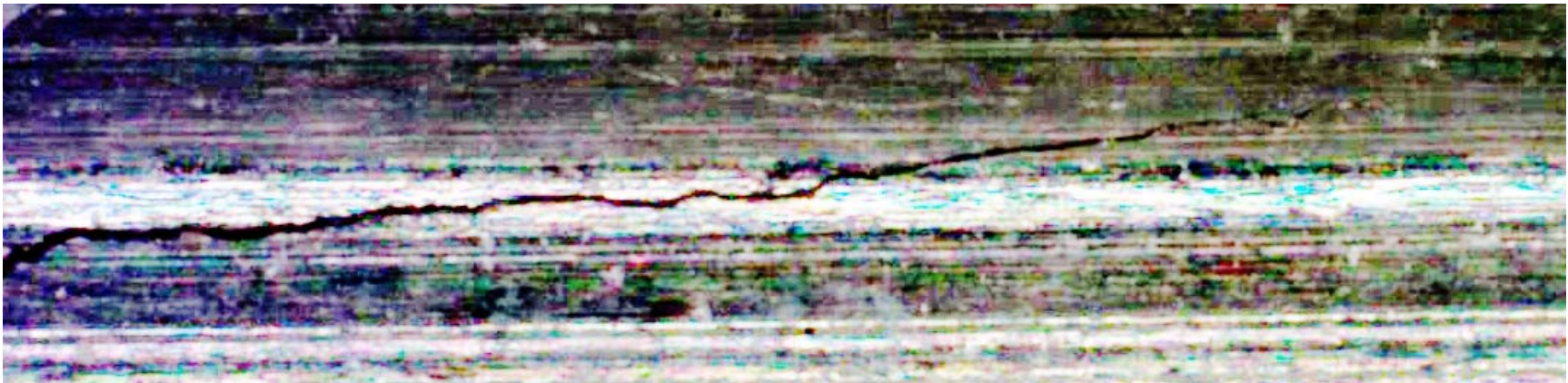
For Bolt-Loaded Samples, Cracks Grow Under Falling K and Stop at Threshold Value



Little or No Crack Growth was Observed after 4 Months Exposure to Tritium Gas



MM75-1 High Stress Intensity (19.8 MPa- \sqrt{m})
)

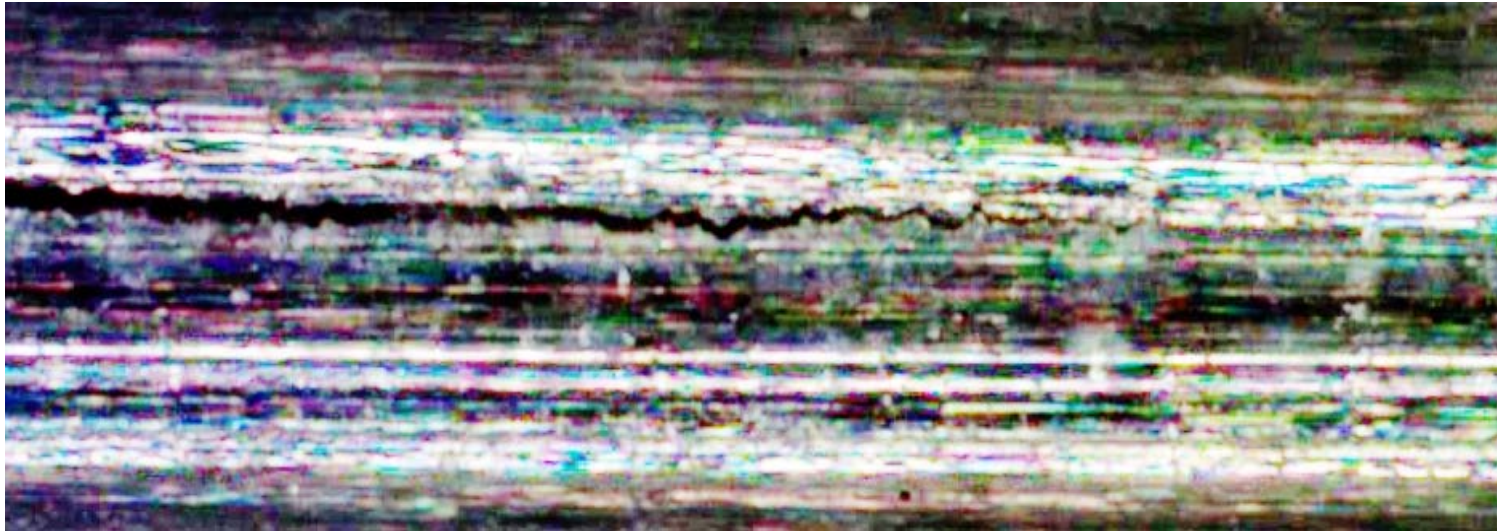


MM75-24 LOW Stress Intensity (11.7 MPa- \sqrt{m})

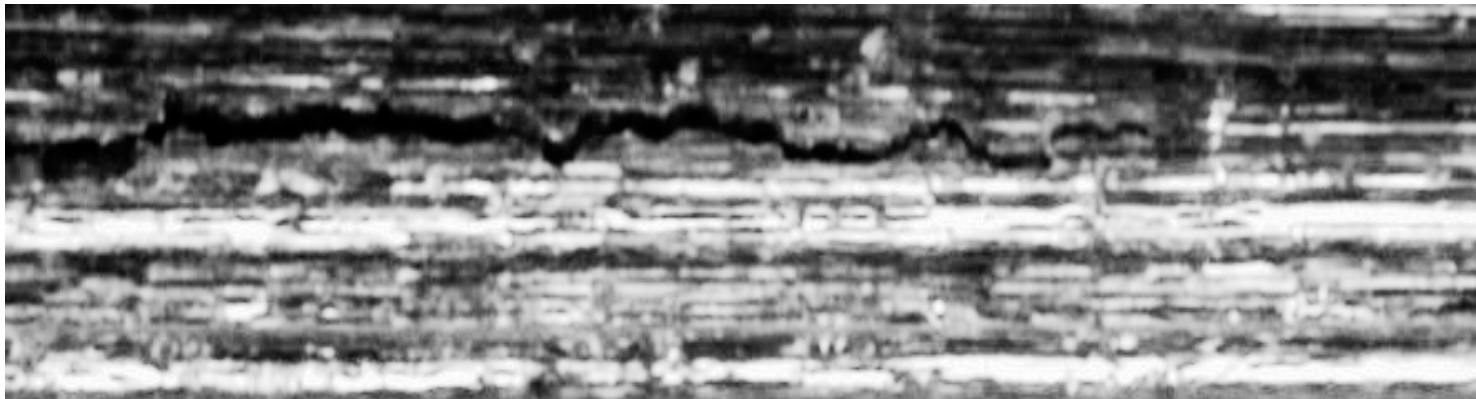
Magnifications ~30 X



Crack Extension **May** have Occurred in Specimens Loaded to **High** Stress Intensities



MM75-2 High Stress Intensity (20.3 MPa-√m)

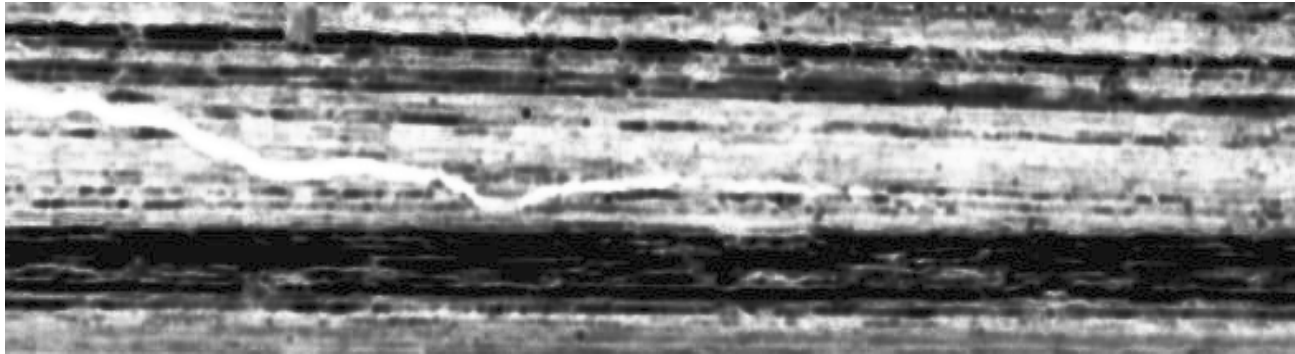


MM75-3 High Stress Intensity (19.4 MPa-√m)

Magnifications ~30 X



No Crack Extension Occurred in Specimens Loaded to **Medium** Stress Intensities



MM75-11 Medium Stress Intensity (17.9 MPa- \sqrt{m})

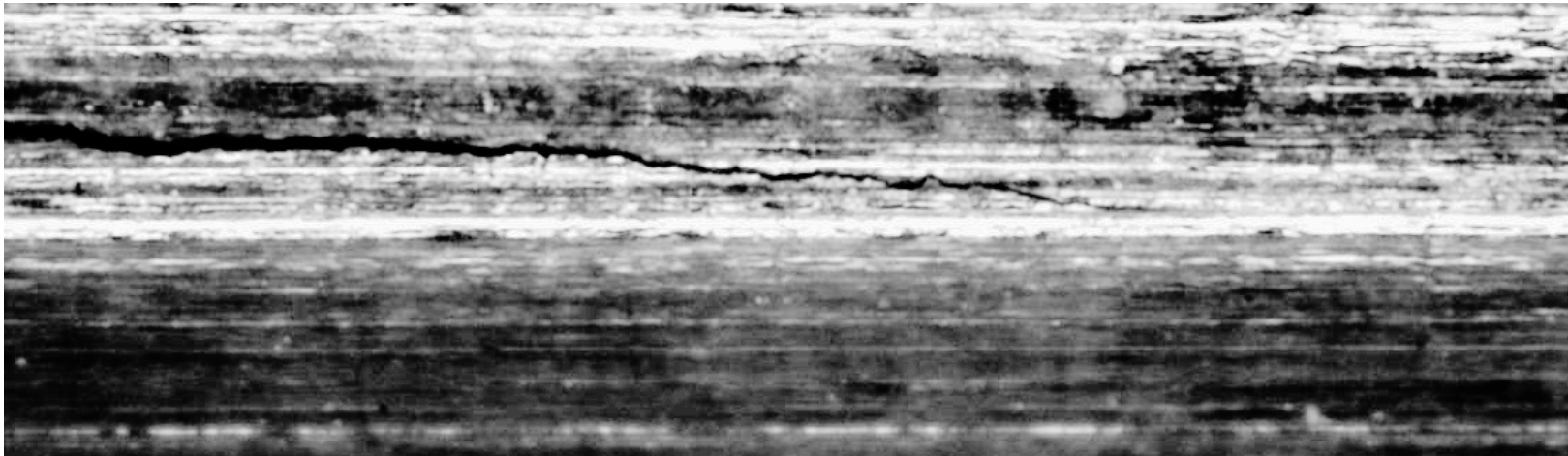


MM75-12 Medium Stress Intensity (18.2 MPa- \sqrt{m})

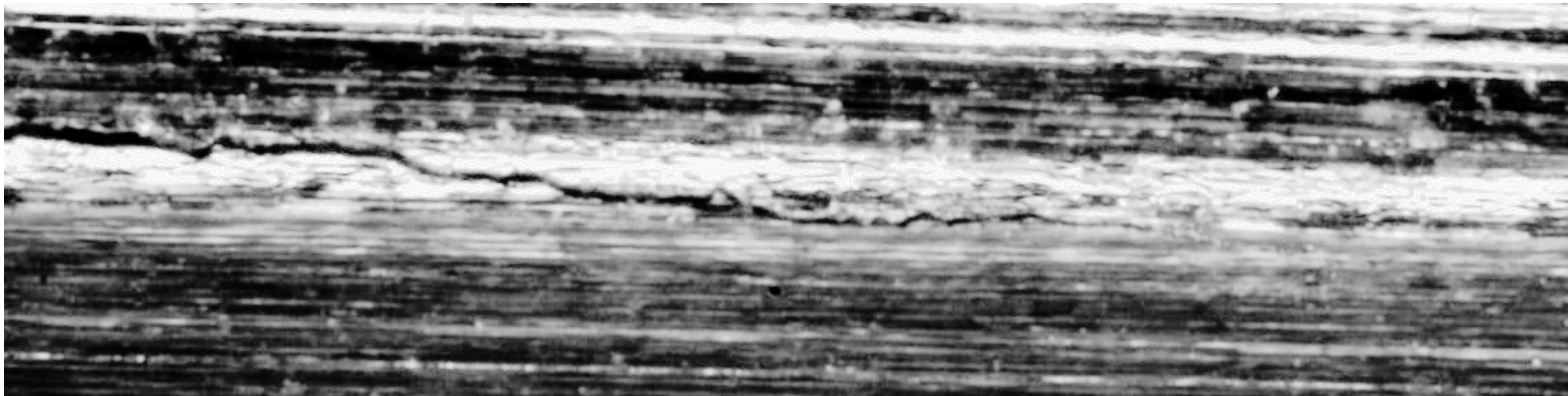
Magnifications ~30 X



No Crack Extension Occurred in Specimens Loaded to **Low** Stress Intensities



MM75-17 Medium Stress Intensity (15.0 MPa- \sqrt{m})

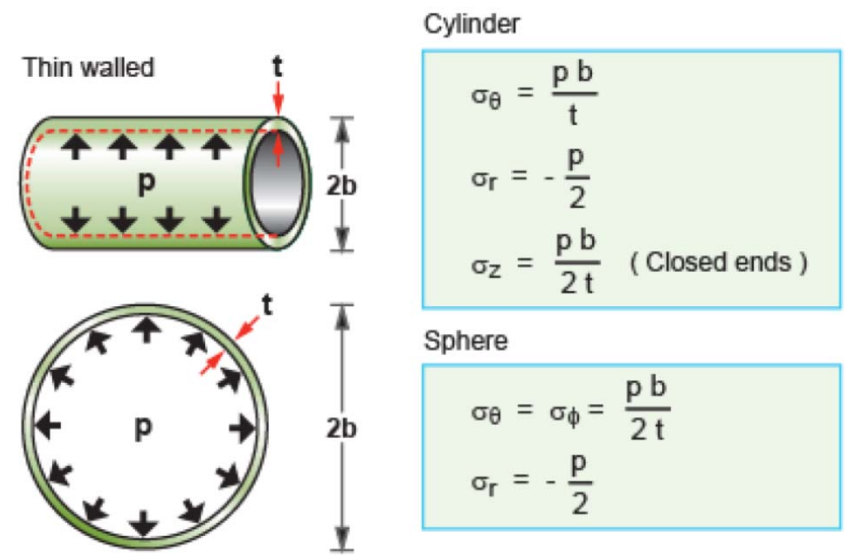


MM75-18 Medium Stress Intensity (14.9 MPa- \sqrt{m})

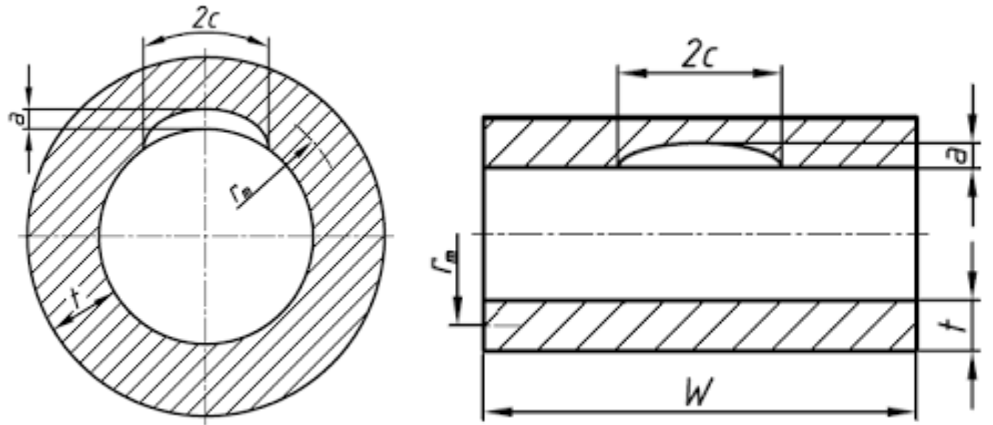
Magnifications ~30 X



Stress Intensity Levels in Jefferson Laboratory Tritium Cell



- Applied stress intensity levels at operating pressure were estimated for the Jefferson Lab tritium cell
- Estimates assumed a variety of flaw shapes and sizes in the cylindrical or spherical portion of the cell
- At operating pressure, Stress Intensity Values were less than 2 MPa-√m.



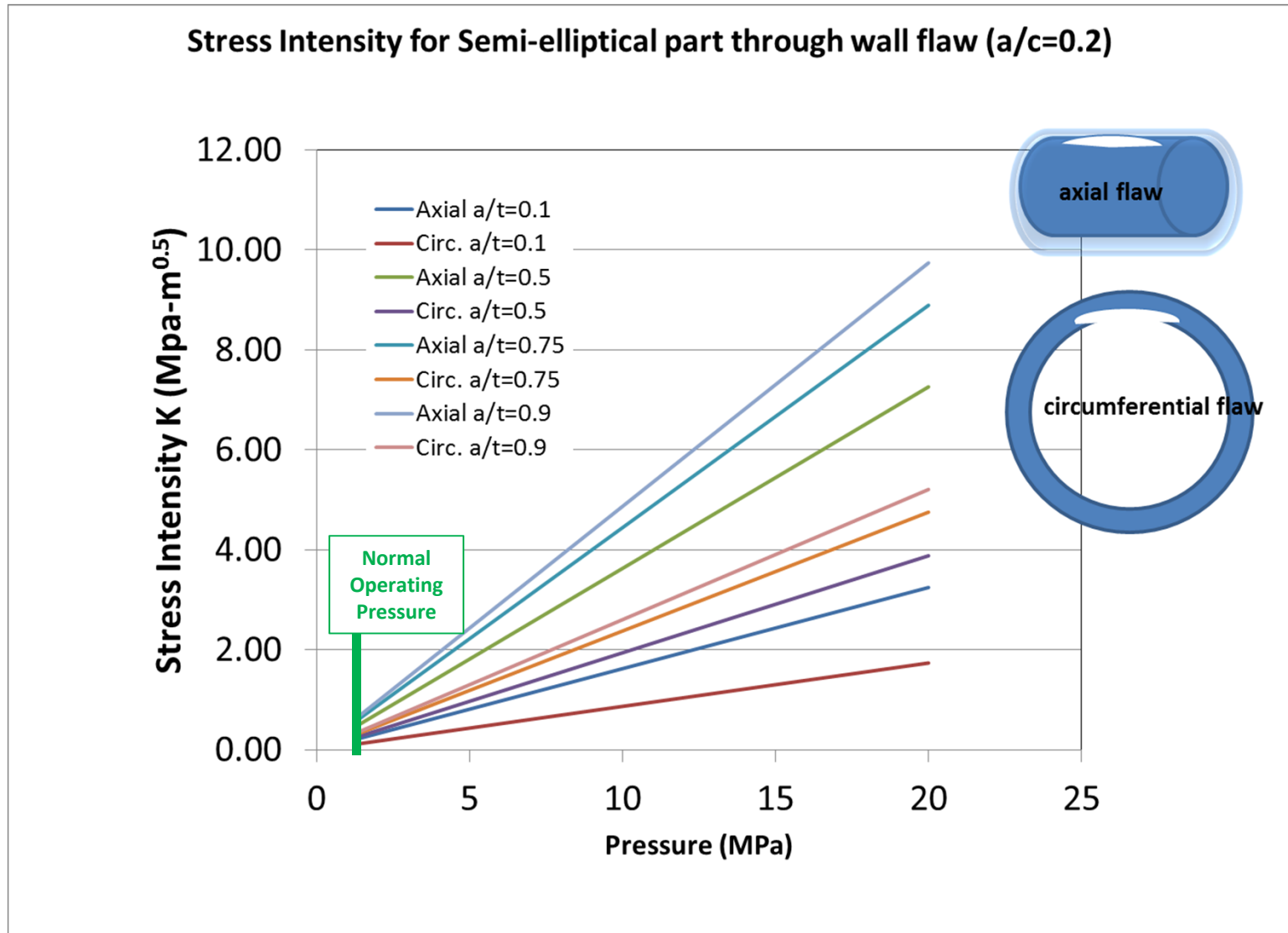
$$K_{I_{max}} = 1.12 \frac{\sigma}{\Phi} \sqrt{\pi a}$$

$$K_{I_{min}} = 1.12 \frac{\sigma}{\Phi} \sqrt{\pi a^2/c}$$

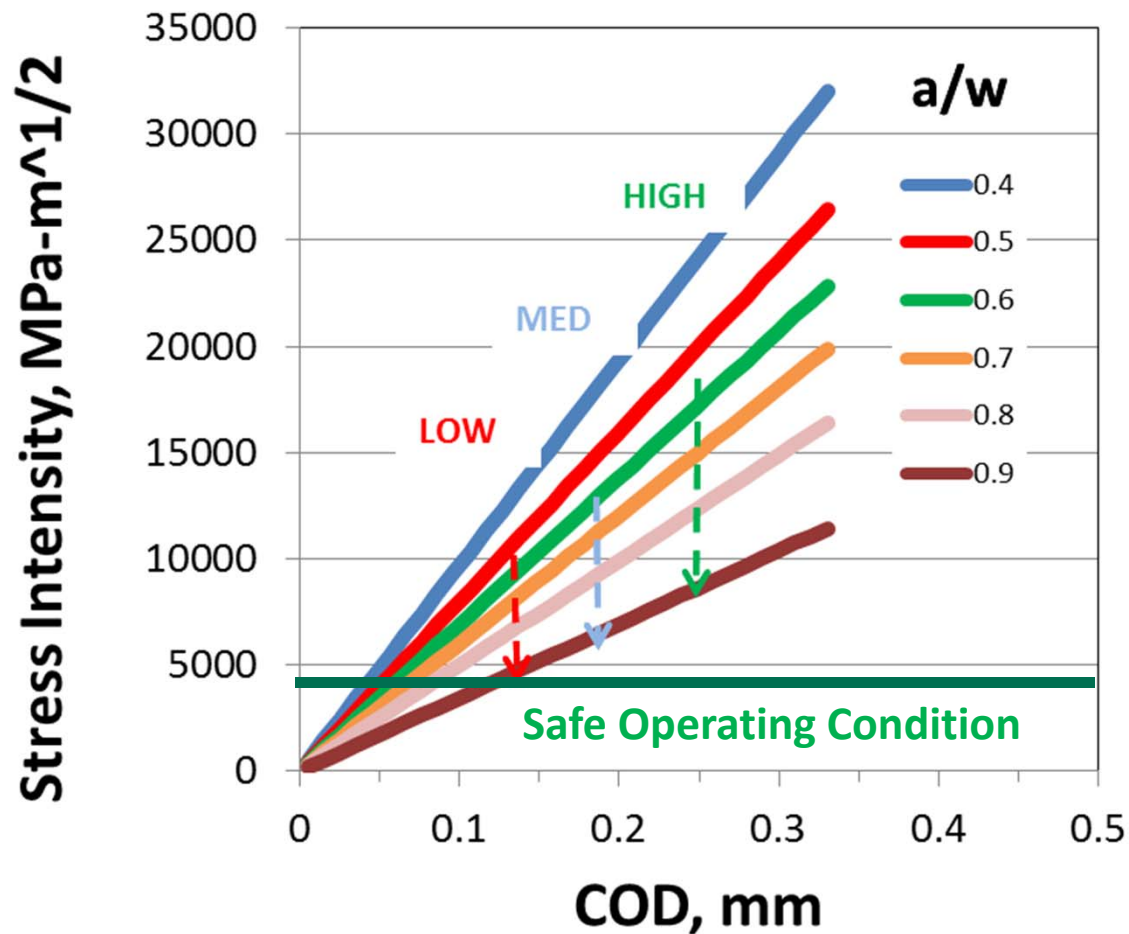
$$\Phi = \int_0^{\pi/2} \left[1 - \frac{c^2 - a^2}{c^2} \sin^2 \varphi \right] d\varphi$$

$$\Phi \approx \frac{3\pi}{8} + \frac{\pi}{8} \frac{a^2}{c^2}$$

Stress Intensity Levels in Jefferson Laboratory Tritium Cell



Specimens Were Loaded to Low, Medium, and High Stress Intensities



- The specimens were loaded well above the operating stress intensities.
- If the material cracking threshold approached the operating stress intensity, large, easily detected crack extension would have occurred in the specimens.

Conclusion:

Since little or no crack growth has been observed after 4 months, no crack growth is expected in the Jefferson Lab tritium cell.



Summary and Preliminary Conclusions

- The fracture toughness, K_{Ic} , of 7075 Aluminum was measured to be 24 MPa- \sqrt{m} for the unexposed condition in agreement with literature values.
- Tritium was expected to reduce the cracking threshold stress intensity values to ~25% K_{Ic} based on hydrogen effects in the literature for 7075 Aluminum in moist air.
- The specimens were exposed to tritium pressures similar to the Jefferson cell effective pressure and loaded to much higher stress intensity levels than the tritium cell.
- Little or no crack growth was observed in 7075 Aluminum specimens stressed between 45-80% of K_{Ic} after 4 months of tritium exposure at 17.2 MPa
- **Since little or no crack growth has been observed after 4 months, no crack growth is expected in the Jefferson Lab tritium cell.**



Future Work

- 4-month exposure specimens will be fractured;
- Measure crack lengths on fracture surfaces;
- Measure actual final load on specimen at end of exposure;
- Recalculate stress intensity levels for true final loads and crack lengths;
- Continue 8-month and 12-month exposures to allow more time for crack nucleation and build-in of helium-3 from tritium decay which could reduce the threshold for cracking;
- Fractography and autoradiography will be conducted on selected specimens to characterize fracture mode and tritium depth of penetration.

Acknowledgments

Dr. David Meekins, Jefferson Labs

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Chad Sweeney, SRNL – SRNL Leak and Pressure Tests

Lee Nigg and Ashley Elizondo, SRNL

Carol Kestin and Ed Stein, SRNS Tritium Materials Test Facility



Thank you for your attention!

SRNL Fast Facts

- > National Laboratory for DOE
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nationally and internationally
- > Applied research, development and
deployment of practical, high-value
and cost effect technology solutions
in the areas of national security, clean
energy and environmental stewardship
- > Operated by Savannah River Nuclear
Solutions for the U.S. Department
of Energy near Aiken, S.C.

Contact Information

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Tritium Effects on Materials

In an effort to ensure the safety of the nation's nuclear weapons stockpile, the Savannah River National Laboratory (SRNL) maintains an active role in the research of tritium's impact on metal and the functionality of weapons systems. Not only does SRNL closely examine the material durability used in the construction of weapons, it examines performance quality in an effort to make certain the weapons stockpile is operating in a safe manner and at its best level. SRNL has been deeply involved in tritium research for over 50 years. Not only do we have the ability, we invented the science.

Putting Our Stockpile to the Test

Tritium reservoirs used in nuclear warheads are constructed of stainless steel and are used for the long-term containment of tritium gas. Tritium and its decay product, helium-3, can cause cracking in these vessels. The tritium embrittlement phenomenon is an aging process that occurs over a long period of time. At SRNL, we have the specialized tools for the delicate testing of reservoirs and tritiated materials. Through an extensive suite of specialized equipment and skilled researchers, SRNL has a unique capability unlike any other laboratory in the world. By investigating the Hydrogen/ Helium embrittlement of materials, SRNL is able to drive the design codes for new technology.



Cracking Thresholds are measured by exposing pre-cracked samples to tritium gas and slowly pulling them apart or by step-loading and holding until crack begins to grow. Crack growth is monitored by using the DC-Potential Drop technique.

