We put science to work.™

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

A U.S. DEPARTMENT OF ENERGY NATIONAL LABORATORY • SAVANNAH RIVER SITE • AIKEN, SC

Effect of Tritium on Cracking Threshold in 7075 Aluminum

Andrew J. Duncan and Michael J. Morgan

February, 2017

SRNL-STI-2017-00002, Revision 0

SRNL.DOE.GOV

DISCLAIMER

Printed in the United States of America

Prepared for
U.S. Department of Energy

Keywords: *Stress Intensity Factor, Hydrogen, Fracture Toughness, Embrittlement, Pressure Vessel*

Retention: *Permanent*

Effect of Tritium on Cracking Threshold in 7075 Aluminum

Andrew J. Duncan
Michael J. Morgan

February, 2017

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

Savannah River National Laboratory™
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

REVIEWS AND APPROVALS

AUTHORS:

Andrew J. Duncan, Materials Science and Technology Date

Michael J. Morgan, Materials Science and Technology Date

TECHNICAL REVIEW:

Poh-Sang Lam, Materials Science and Technology Date

APPROVAL:

Justin Halverson, Manager Date
Materials Science and Technology

ACKNOWLEDGEMENTS

The authors would like to acknowledge several individuals who assisted in producing this work. Lee Nigg and Greg Staack assisted in coordination of the project task and laboratory resources. Ashley Elizondo assisted in planning and selection of specimen design. Chad Sweeney, Calvin Clamp, and Dante Pilgrim assisted in conducting the tritium exposures and specimen recovery operations. Eddie Stein, Carol Kestin and Brenda Bordon assisted in post-exposure characterization.

EXECUTIVE SUMMARY

The effect of long-term exposure to tritium gas on the cracking threshold (K_{TH}) of 7075 Aluminum Alloy was investigated. The alloy is the material of construction for a cell used to contain tritium in an accelerator at Jefferson Laboratory designed for inelastic scattering experiments on nucleons. The primary safety concerns for the Jefferson Laboratory tritium cell is a tritium leak due to mechanical failure of windows from hydrogen isotope embrittlement, radiation damage, or loss of target integrity from accidental excessive beam heating due to failure of the raster or grossly mis-steered beam. Experiments were conducted to investigate the potential for embrittlement of the 7075 Aluminum alloy from tritium gas.

Aluminum alloys are generally resistant to embrittlement by hydrogen isotopes because of their passive oxide layer which prevents hydrogen ingress into the alloy. However, hydrogen embrittlement has been observed in 7075 Aluminum in moist air, because the water molecule reduces the effectiveness of the aluminum oxide surface and allows dissociated hydrogen to diffuse into the alloy. Although embrittlement has not been observed in dry hydrogen gas, embrittlement may occur in dry tritium gas because other dissociation phenomena are possible. In accelerator applications, tritium will dissociate during beam-on conditions. Tritium molecules also dissociate upon radioactive decay of one of the two atoms that form the molecule. The dissociated tritium may then diffuse into the aluminum and induce embrittlement leading to failure by slow crack growth in service.

This investigation used a fracture mechanics approach to evaluate the potential for embrittlement of the tritium cell. Because data on the effects of tritium on the fracture properties of high strength aluminum alloys is very limited, in-situ cracking threshold experiments were conducted in high-pressure tritium gas on 7075 Aluminum specimens supplied by Jefferson Laboratories. Twenty-eight specimens were fatigue pre-cracked and bolt-loaded to different stress intensity factors according to the test procedure described in ASTM E1681. The stress intensity factors ranged between 40% and 90% of the fracture toughness value (K_{Ic}) of 7075 Al. The stressed specimens were placed in a pressure vessel and held in 17.2 MPa of tritium gas for up to ten months at ambient temperature. Examinations of specimens were conducted after four, eight, and ten month intervals and showed little or no evidence of crack growth at any of the stress intensity factors.

The results were used for estimating the potential for cracking of the tritium cell. To do this, stress intensity factors for the cell were estimated using handbook formulae and the cell geometry, its operating pressure, and postulated flaws. The stress intensity factors for the cell at normal operating pressure were estimated to be less than 1 MPa-√m. Since little or no crack growth was observed in the specimens exposed to tritium gas for up to 10 months at stress intensity factors more than ten times (12-20 MPa-√m) these values, no crack growth is expected in the Jefferson Lab cell.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Al	Aluminum
ASTM	American Society for Testing Materials
ASME	American Society of Mechanical Engineers
COD	Crack opening displacement
K	Stress Intensity Factor
K_{Ic}	Material Fracture Toughness
K_{TH}	Environment-assisted cracking threshold stress intensity factors
MPa	Megapascal
SRNL	Savannah River National Laboratory

1.0 Introduction

The effect of long-term exposure to tritium gas on the cracking threshold of 7075 Aluminum Alloy was investigated. The alloy is the material of construction for a pressure cell used to contain tritium in an accelerator at Jefferson Laboratory designed for inelastic scattering experiments on nucleons (1). The primary safety concern for the target is a tritium leak due to mechanical failure of windows from hydrogen isotope embrittlement, radiation damage, or loss of target integrity from accidental excessive beam heating due to failure of the raster or grossly mis-steered beam (1). The purpose of this study was to investigate the potential for tritium embrittlement under conditions expected in the Jefferson Lab cell during normal and above normal operating conditions.

In general, the 7075 Aluminum is resistant to embrittlement by hydrogen isotopes except under specific environmental conditions. This is because the passive oxide layer acts as a barrier to hydrogen dissolution by preventing disassociation of the molecule (2, 3). Watakabe, *et al.* (2) used tritium autoradiography to show that there are exceptions to this as tritium was shown to dissociate and permeate near intermetallic compounds and from water environments. In pure aluminum, tritium invasion was not detected, while tritium was observed in the constituent particles in two alloys: 6061 Al through AlFeSi phase particles and, 7075 Al alloy through Al_7Cu_2Fe phase particles (Figure 1) (2). It is unclear if the amount of tritium associated with these second phase particles would be enough to cause embrittlement of the alloy. In another study, subcritical cracking associated with hydrogen embrittlement at low stress intensity factors has been observed in 7075 Al tested in humid air (3). Speidel measured the effect of humidity and stress intensity on stress corrosion crack velocity of 7075-T651 Aluminum alloy plate in air (Figure 2). Speidel also showed that cracking only occurred in moist environments and that, dry hydrogen, dry nitrogen, dry oxygen, dry air, and dry argon do not support stress corrosion crack growth, and propagating stress-corrosion cracks stopped when specimens were transferred to such dry gasses (3).

In the case of dry tritium gas exposure, free tritium atoms may be available for dissolution on the surface because of radioactive decay. Tritium that diffuses into the aluminum could lower the threshold for cracking leading to delayed failure by slow crack growth. Research on tritium effects on high strength aluminum alloys is limited and the data do not include engineering parameters such as crack growth rates and threshold stress intensity factors. Measured cracking thresholds in tritium gas may also depend on the time of exposure, because of additional embrittlement from its radioactive decay product, helium-3.

To evaluate the potential for embrittlement of the tritium cell, this investigation used a experimental fracture mechanics approach. In-situ cracking threshold experiments were conducted in high-pressure tritium gas on 7075 Aluminum specimens supplied by Jefferson Laboratories. The results of the experiments were used along with a fracture mechanics analysis of the tritium cell to evaluate the potential for tritium-induced cracking in the tritium cell.

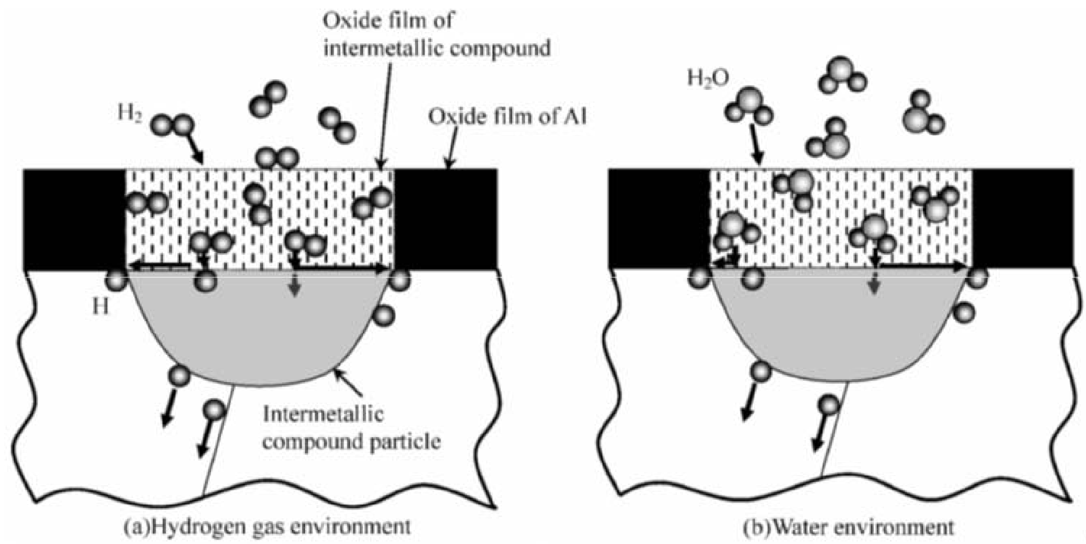


Figure 1: Mechanism for hydrogen invasion into an aluminum alloys from two environments. Normal resistance to H_2 embrittlement from the oxide film can break down near intermetallic compound particles (a) and water environments (b) (2).

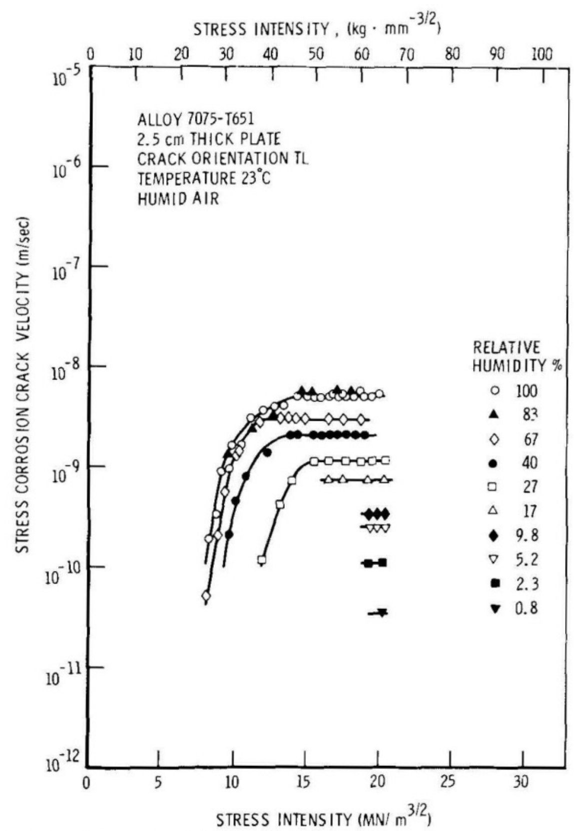


Figure 2: Effect of humidity and stress intensity on stress corrosion crack velocity of 7075-T651 aluminum alloy plate in air (3).

2.0 Experimental Procedure

Cracking thresholds for 7075 Aluminum were measured in high pressure tritium gas using ASTM E 1681-03 (4). The test standard method is used to determine environment-assisted cracking threshold stress intensity factors (K_{TH}) for metallic materials from constant-displacement testing of fatigue precracked bolt-load compact fracture specimens. The specimen design was adopted from the bolt-loaded compact tension specimen referenced in the standard and is shown in Figure 3. The specimens were fabricated from 7075 Al bar stock in the T651 conditions in the TL orientation and supplied to SRNL from Jefferson Labs. Typical composition and properties of 7075 Aluminum are shown in Figure 4. Pre-cracks were initiated in the specimens by fatigue in air to intermediate crack lengths (a/w ≅ 0.50-0.65). Fatigue cracking was conducted at stress intensity factors as low as possible consistent with developing a crack in the specimens within 24 hours. The maximum fatigue loads and their corresponding stress intensity factors are shown in Table 1.

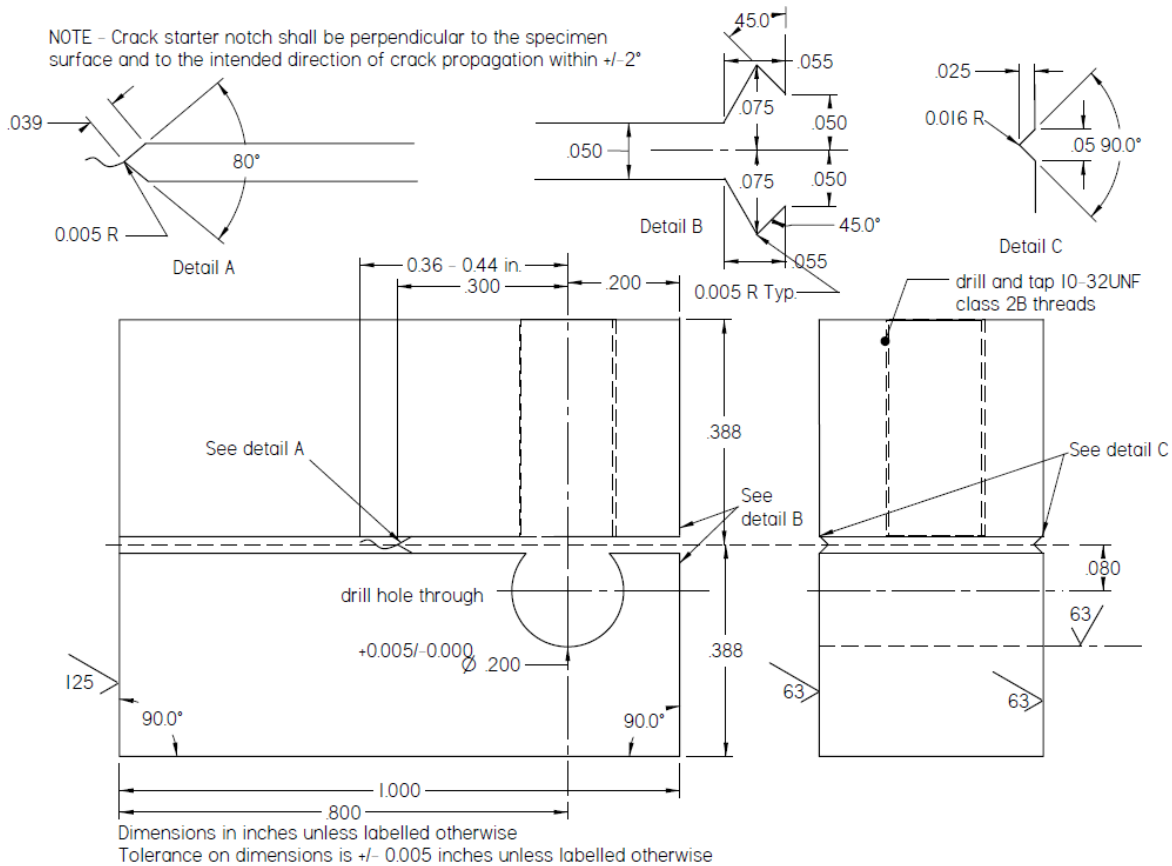


Figure 3: Bolt-loaded compact tension specimen.

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	K_{Ic}:ksi√in. (MPa√m)* T-L
7075	T651 T7351	26 (28.6) 30 (32.0)	22 (24.2) 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

Figure 4: Composition and mechanical properties of 7075 Aluminum (5).

After fatigue cracking and prior to exposure to tritium gas, the specimens were cleaned in concentrated nitric acid to remove any heavy oxide scale, washed in dilute alconox solution to remove any oils from the surface and rinsed with isopropyl alcohol and dried with a heat gun to ensure adequate drying. The cleaned specimens were then loaded to desired stress intensity factors. This was achieved by first elastically loading and unloading specimens in an Instron mechanical testing machine while recording load and crack opening-displacement (COD). The specimens were then reloaded in a vice with a bolt to a critical COD value corresponding to the desired stress intensity factor (Figure 5).

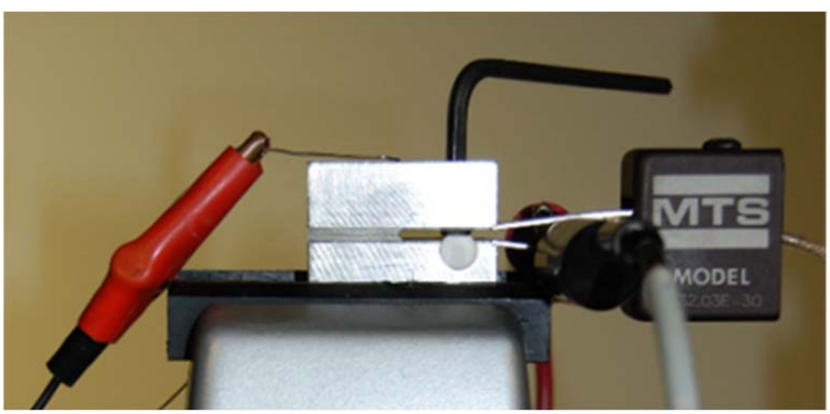


Figure 5: Specimens were loaded in a vice to desired stress intensity factors by monitoring a crack opening displacement clip gage.

After loading, the specimen crack lengths were measured on both exposed surfaces using an optical microscope. The actual loads on the specimens were estimated from the individual load-COD curve. The individual specimen dimensions, COD values, crack lengths, and fatigue loads are listed in Table 1. In general, all specimens were loaded to at least 1.5 times the stress intensity factors needed for fatigue precracking to minimize residual stress effects at the fatigue crack tips. Specimens that had low fatigue loads were used for bolt loading to low stress intensity factors; specimens that saw high fatigue loads were loaded to high stress intensity factors. The actual stress intensity factors achieved ranged from 40-90% of the fracture toughness of the material (K_{Ic}) measured in laboratory air. K_{Ic} of Alloy 7075-T6 in TL orientation was measured to be 24.2 MPa-√m which is consistent with ALCOA data (5).

The intent of the experimental plan was to load the specimens to a range of stress intensity factors that were reasonably possible for the Jefferson Lab cell. We wanted to load the specimens to stress intensity factors that corresponded to, and were greater than, normal operating conditions. To do this, conservative values of stress intensity factors were estimated for the cell using known values of the cell operating pressure and handbook formulae by assuming the presence of a flaw in the cell wall. The estimates were based on a variety of flaw shapes and sizes. The details of the calculations are given in the Experimental Analysis section. It should be emphasized that visual inspection of the cells after fabrication did not reveal flaws. The presence of flaws having different sizes and shapes was assumed in order to estimate fracture mechanics parameters. Even for the worst possible flaws assumed, the largest value of stress intensity in the cell was estimated to be less than 1 MPa-√m. Fatigue cracks could not be initiated at such low stress intensity factors. Instead, the lowest factors achievable were used (~10 MPa-√m). The highest factors achieved were ~20 MPa-√m which is approximately 90% of the stress intensity factor that would cause fracture in the absence of tritium, i.e., the material fracture toughness value, K_{Ic}. Further details are given in the Experimental Analysis section below.

After the specimens were bolt-loaded, they were stacked into a pressure vessel and exposed to 17.2 MPa tritium gas for 4, 8 and 10 months at ambient temperature (Figure 6). In this way, cracking thresholds could be measured in-situ in high pressure tritium gas. The exposure pressure was the maximum allowed for the pressure vessel available at this time. The pressure used is similar to, but not as large as estimates of the effective pressure of tritium during beam-on conditions for the Jefferson cell, 21.5 MPa (6). Although the tritium cell will be filled at ambient temperature with 1090 Curies of pure tritium to a pressure of 1.38 MPa, the fugacity of tritium gas in the cell during beam operation is increased because the beam will dissociate the tritium molecules which will recombine on the cell walls. The effective pressure is used for estimating the concentration of tritium on the cell walls and not for estimating stresses. The fill pressure is used for stress calculations.

Table 1: Experimental data set including exposure, specimen dimensions, fatigue and preloading details

Sample ID	Exposure	W	B	Bn	b avg	a avg	a/w avg	COD	P	f(a/w) (COD)	K(COD)	Fatigue P_{Max}	K_{Max} (Fat)	K(COD)/ K_{Max} (Fat)
description	"time at pressure"	"width"	"thickness"	"net thickness"	"remaining ligament"	"crack length"		"crack open displ"	"load"	"constant"	"stress intensity factor"	"max load"	"max SI factor in fatigue"	
Units	Months	mm	mm	mm	mm	mm		mm	N		MPa-√m	N	MPa-√m	
MM75-9	10	20.3	10.2	8.9	9.1	11.2	0.551	0.279	2108	0.147	20.7	1023	9.0	2.3
MM75-2	4	20.3	10.1	8.9	9.4	11.0	0.540	0.271	2224	0.149	20.3	1468	12.5	1.6
MM75-6	10	20.3	10.1	8.9	8.5	11.8	0.583	0.285	1957	0.141	20.1	961	9.5	2.1
MM75-5	8	20.3	10.2	8.9	8.5	11.8	0.583	0.283	1942	0.141	20.0	1019	10.1	2.0
MM75-4	8	20.3	10.1	8.9	6.6	13.6	0.673	0.319	1373	0.125	20.0	890	12.9	1.5
MM75-10	10	20.3	10.1	8.9	8.6	11.7	0.577	0.279	2055	0.142	19.9	1010	9.8	2.0
MM75-8	10	20.3	10.2	8.9	8.1	12.1	0.598	0.287	2037	0.138	19.9	1023	10.8	1.8
MM75-7	10	20.3	10.2	8.9	8.2	12.1	0.594	0.285	1988	0.138	19.9	961	10.0	2.0
MM75-1	4	20.3	10.2	8.9	8.4	11.9	0.586	0.281	1935	0.140	19.8	1357	13.6	1.5
MM75-3	4	20.4	10.2	8.9	7.8	12.7	0.619	0.289	1753	0.134	19.4	965	11.0	1.8
MM75-13	8	20.3	10.2	8.9	8.6	11.7	0.577	0.257	2001	0.142	18.3	1063	10.3	1.8
MM75-14	8	20.3	10.2	8.9	8.6	11.6	0.574	0.255	1926	0.142	18.3	983	9.4	1.9
MM75-16	10	20.3	10.1	9.0	8.6	11.7	0.575	0.255	2004	0.142	18.3	988	9.5	1.9
MM75-12	4	20.3	10.2	8.9	8.5	11.8	0.582	0.257	1902	0.141	18.2	974	9.6	1.9
MM75-15	10	20.4	10.1	8.9	8.4	11.9	0.586	0.256	1908	0.140	18.0	992	9.9	1.8
MM75-11	4	20.3	10.1	8.9	8.2	12.1	0.598	0.258	1706	0.138	17.9	979	10.3	1.7
MM75-22	10	20.3	10.1	8.9	8.8	11.6	0.569	0.214	1673	0.143	15.4	1028	9.7	1.6
MM75-23	10	20.3	10.2	8.9	8.5	11.8	0.581	0.216	1488	0.141	15.3	970	9.6	1.6
MM75-20	8	20.3	10.1	8.9	8.9	11.4	0.560	0.208	1690	0.145	15.2	1005	9.2	1.7
MM75-17	4	20.3	10.1	8.9	8.4	11.9	0.585	0.213	1558	0.140	15.0	1005	10.1	1.5
MM75-21	8	20.3	10.2	8.9	8.4	11.9	0.585	0.212	1539	0.140	14.9	1023	10.2	1.5
MM75-18	4	20.3	10.1	8.9	8.5	11.9	0.584	0.211	1587	0.140	14.9	1010	10.0	1.5
MM75-27	8	20.4	10.2	8.9	8.8	11.6	0.569	0.184	1375	0.143	13.2	681	6.4	2.1
MM75-28	10	20.4	10.1	8.9	7.8	12.6	0.618	0.180	1117	0.134	12.2	678	7.7	1.6
MM75-24	4	20.3	10.1	8.9	8.2	12.1	0.595	0.168	1103	0.138	11.7	685	7.1	1.6
MM75-26	8	20.3	10.2	8.9	8.4	11.9	0.584	0.158	1081	0.140	11.2	667	6.7	1.7
MM75-29	10	20.4	10.2	8.9	8.2	12.2	0.597	0.160	1019	0.138	11.1	667	7.0	1.6
MM75-25	10	20.3	10.1	8.9	8.7	11.6	0.573	0.142	1032	0.143	10.2	685	6.5	1.6

Cracking thresholds were measured by examining the specimens after exposure using an optical microscope and measuring the cracking lengths on both sides of the specimen surfaces. The measured crack lengths were compared to the initial values measured for each specimen. If tritium were to cause cracking at the stress intensity factors used, which would indicate the need for further analysis of the Jefferson Cell, a significant amount of crack growth would occur on the specimen surfaces. We estimated that cracks would grow from their initial values of ~50%-65% of the section width to 75%-90% of the section width. This could easily be seen using the optical microscopes available. Furthermore, for bolt-loaded samples, cracks have the inherent advantage in that they grow under a falling stress intensity factor and will arrest at the threshold value (K_{TH}). This technique ensures that, if cracks propagate during exposure, they will arrest before specimen failure and can be measured at the end of the exposure period.

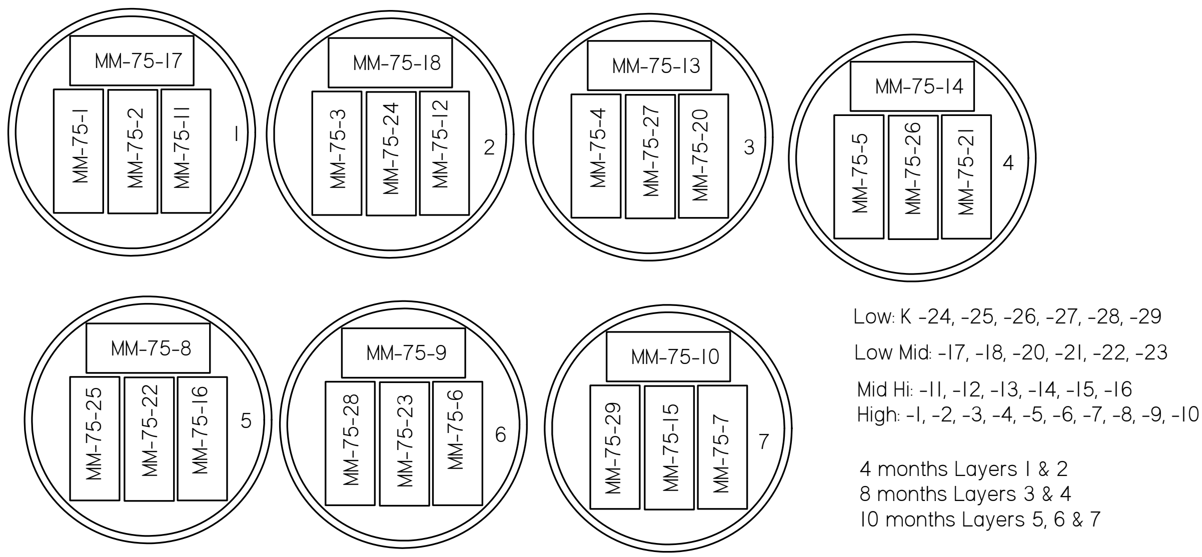


Figure 6: Placement of samples in autoclave pressure vessel and schedule for withdrawal (specimens were withdrawn after 10 months to satisfy project needs).

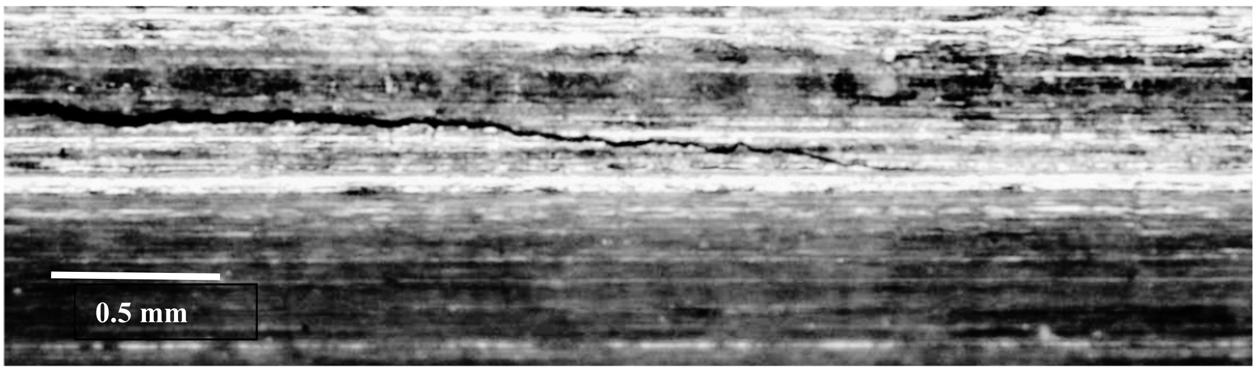
Additional examinations including tritium autoradiography (to measure tritium depth of penetration), specimen fracture, and scanning electron fractography were planned but not conducted. The aluminum specimens had excessive residual surface tritium contamination and high off-gassing levels for the laboratory hoods available for these additional examinations. The tritium activity was much higher than those generally observed for the more common stainless steel examinations conducted at SRNL after tritium exposures. Furthermore, the tritium off-gassing levels did not diminish over time, which is commonly observed for stainless steel specimens, after many weeks in the fresh air hoods. The activity apparently results when the tritium contaminated aluminum surfaces react with laboratory air. The moist air apparently makes tritium stick to the surfaces. These off-gassing and tritium contamination issues limited the specimen handling and examinations that could be conducted. It is not clear at this time whether the handling issues we experienced with tritium contaminated aluminum would have any bearing on the handling of the Jefferson-Lab tritium cell after it is filled with tritium gas in dry nitrogen boxes. Recommendations are given below to help resolve possible concerns.

3.0 Experimental Results

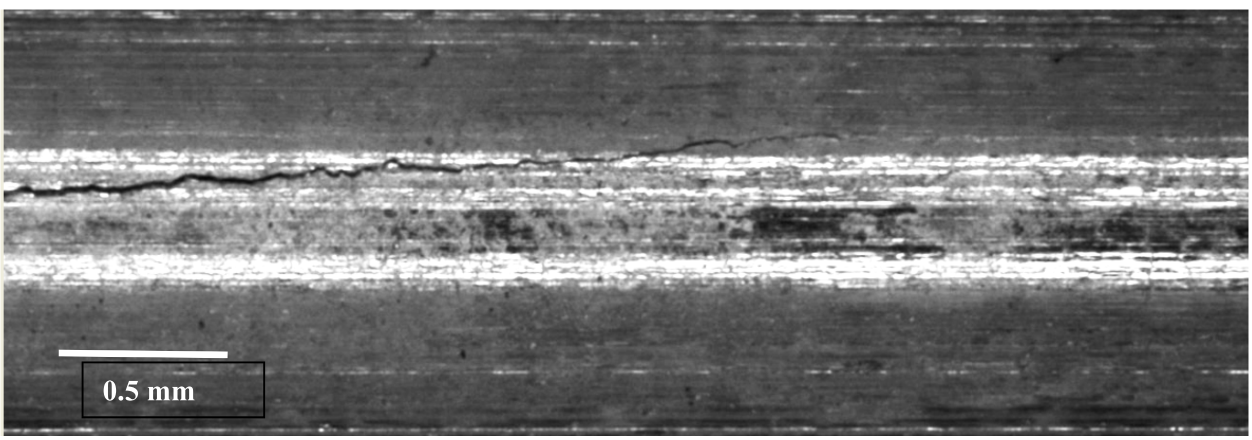
Cracking thresholds were measured by examining the specimens after 4, 8, and 10 months of exposure to tritium gas using an optical microscope and measuring the cracking lengths on both sides of the specimen surfaces. Typical optical micrographs of the surface cracks are in Figure 7. The measured crack lengths were compared to the initial values measured for each specimen. If tritium were to cause cracking at the stress intensity factors used, and during the time of exposure, further analysis of the Jefferson Cell would be warranted. The specimens were designed so that significant crack growth would easily be detected -from the initial value of 50%-65% of the section width to 75%-90% of the section width.

The measurements are summarized in Table 2. In all but three specimens, no significant crack extension was observed. Specifically, for 25 of the 28 specimens the final crack lengths were all equivalent to the original crack lengths, given the margin of error for the technique. What crack extension was observed for specimens MM-75-3, MM75-16, and MM75-27 was small and likely insignificant as well. For the specimen with the largest crack growth, MM75-3, the measurements indicated that the crack grew from 62% to 65% of the section width after four months of exposure. Although these values are smaller than what the technique was designed to detect, K_{TH} can be estimated from the final crack length to be 18.6 MPa√m, somewhat smaller than the stress intensity factor that the specimen was originally loaded to, i.e. 19.4 MPa√m. The observed crack growth, if real, was even smaller for specimens MM75-16 (eight-month exposure) and MM75-3 (four-month exposure). For specimens MM75-16 and MM75-27, the cracks grew from 57% to 59% of the remaining ligament. Based on these values, K_{TH} for MM75-16 was estimated to be 17.8 MPa√m (originally loaded to 18.2 MPa√m) and for MM75-27, 12.8 MPa√m (originally loaded to 13.2 MPa√m).

MM75-2, K = (20.3 MPa-√m), 4 month exposure



MM75-17, K = (15.0 MPa-√m), 4 month exposure



MM75-25 K = (10.2 MPa-√m), 10 month exposure

Figure 7: Examples of optical crack length measurements.

Table 2: Crack Growth Data for specimens as a function of exposure

Time of exposure	Sample	K MPa-√m	COD mm	a/w Initial	a/w Final
4 months	MM75-1	19.8	.28	.59	.58
	MM75-2	20.3	.27	.54	.54
	MM75-3	19.4	.29	.62	.65
	MM75-11	17.9	.26	.60	.61
	MM75-12	18.2	.26	.58	.58
	MM75-17	15.0	.21	.59	.58
	MM75-18	14.9	.21	.58	.57
	MM75-24	11.7	.17	.60	.61
8 months	MM75-4	20.0	.32	.67	.66
	MM75-5	20.0	.28	.58	.57
	MM75-13	18.3	.26	.58	.57
	MM75-14	18.3	.26	.57	.57
	MM75-20	15.2	.21	.56	.56
	MM75-21	14.9	.21	.59	.59
	MM75-26	11.2	.16	.58	.59
	MM75-27	13.2	.18	.57	.59
10 months	MM75-6	20.1	.28	.58	.59
	MM75-7	19.9	.29	.59	.60
	MM75-8	19.9	.29	.60	.59
	MM75-9	20.7	.28	.55	.56
	MM75-10	19.9	.28	.58	.57
	MM75-15	18.0	.26	.59	.59
	MM75-16	18.3	.26	.57	.59
	MM75-22	15.5	.21	.57	.56
	MM75-23	15.4	.22	.58	.58
	MM75-25	10.2	.14	.57	.57
	MM75-28	12.1	.18	.62	.62
	MM75-29	11.1	.16	.60	.60

4.0 Fracture Mechanics Analysis

To evaluate the potential for cracking of the tritium cell (see Figure 8), stress intensity factors of postulated semi-elliptical flaws are needed. A thorough analysis would use code based solutions like those in ASME Section XI, Appendix A (7). To do this, more information is required than was available to SRNL (e.g., materials specific stress-strain curves, detailed design parameters, stress analyses, etc.). For the purposes of this study, stress intensity factors for the cell were estimated using standard formulae for semi-elliptical flaws in a pressurized cylinder loaded by membrane stresses and pressure on the crack faces (Figures 9-10). The estimates were calculated from the cell geometry, its operating pressure, and a variety of postulated flaws (Figure 10). For this analysis, it was assumed that the tritium cell will be filled at ambient temperature with 1090 Curies of pure tritium to a pressure of 1.38 MPa.

Figure 9 shows the calculation formulae that were used for estimating the membrane stresses in the cylindrical and spherical regions of the tritium cell, i.e., standard handbook formulae for estimating stresses in cylindrical and spherical vessels. Figure 10 depicts the postulated flaw shapes and geometric parameters used in the calculations and shows the stress intensity factor estimation formulae for the assumed flaws given the vessel dimension and stress levels. The results of the calculations are summarized in Figure 11. Note from Figure 11 that the stress intensity factors for the cell at normal operating pressure for a variety of axial and circumferential flaws were estimated to be less than 1 MPa-√m.

The analysis was also used to demonstrate if cracking would be expected in the Jefferson Lab cell given the results and the calculated stress intensity factors. Figure 12, shows the calculated relationship between crack growth and stress intensity factor for the falling K bolt-load test taken from Equation 4 of ASTM E1681 (4). The figure shows the estimated cracking thresholds if crack growth to 90% of the section width had been detected in various specimens. For example, for specimens loaded to high stress intensity factors (17-20 MPa-√m), if the crack grew from the initial value of ~65% of the section width to 90% of the section width, the test would indicate that the tritium had reduced the threshold for cracking to 7.5 MPa-√m. If a similar amount of crack growth had been observed for specimens loaded to medium stress intensity factors (13-15 MPa-√m), the data would indicate a reduction in the cracking threshold from tritium exposure to 6 MPa-√m and to 5 MPa-√m for those specimens originally loaded to low stress intensity factors. These reduced values are still above the 1 MPa-√m estimate for the Jefferson Lab cell. Since little or no crack growth was observed in the specimens exposed to tritium gas for up to 10 months at stress intensity factors more than ten times these values (12-20 MPa-√m), no crack growth is expected in the Jefferson Lab cell.

The results and analysis show that cracking of this alloy for the exposure conditions used in the present study (i.e., high pressure tritium gas) is difficult to induce. However, a longer nucleation period may be required for tritium dissociation and diffusion than the time period used in this study. During beam-on conditions in the cell, diffusion could be accelerated due to radiation induced dissociation which may shorten this nucleation period (6). Figure 11 shows the stress intensity factor expected for postulated flaws as a function of pressure for the tritium cell. The stress intensities expected are well below those used in this study. In addition, no cracking was detected after 10 months. Hence, it is not expected to occur during the planned operation period.

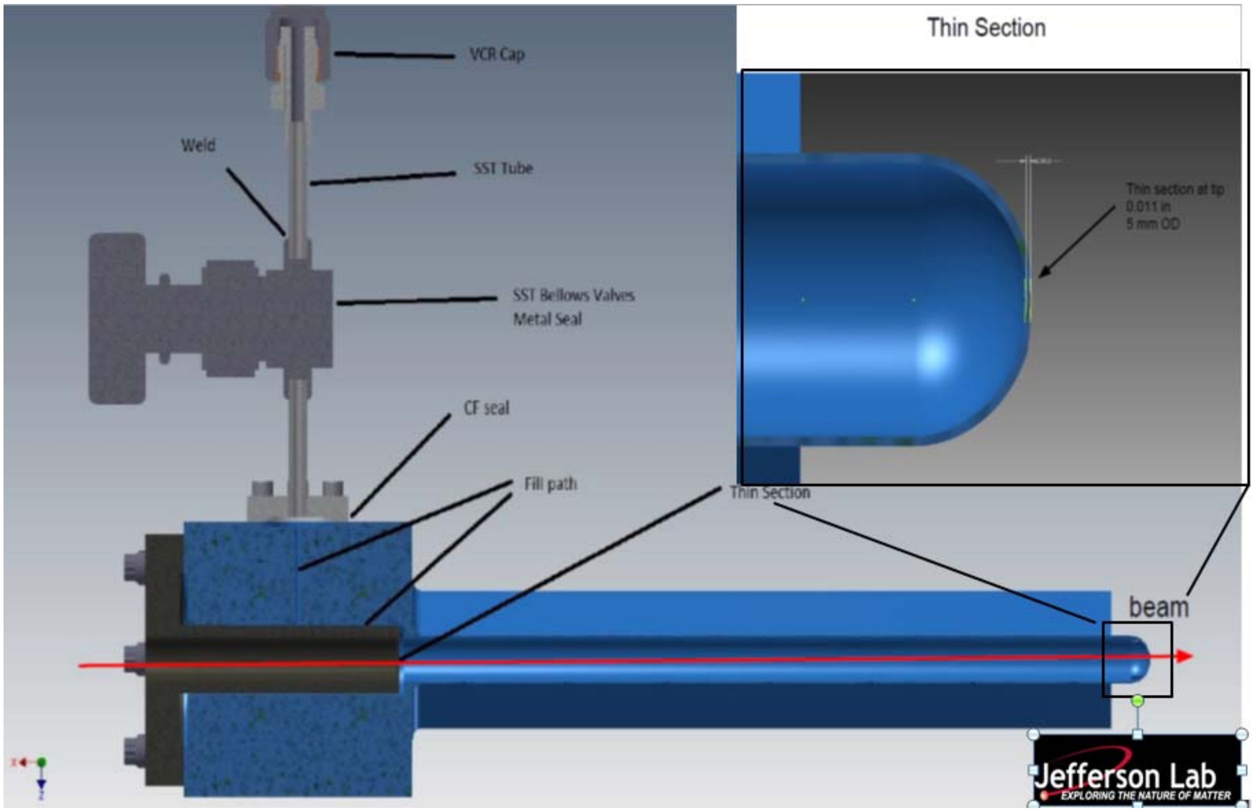


Figure 8: Tritium Cell Design for proposed Jefferson Lab experiment.

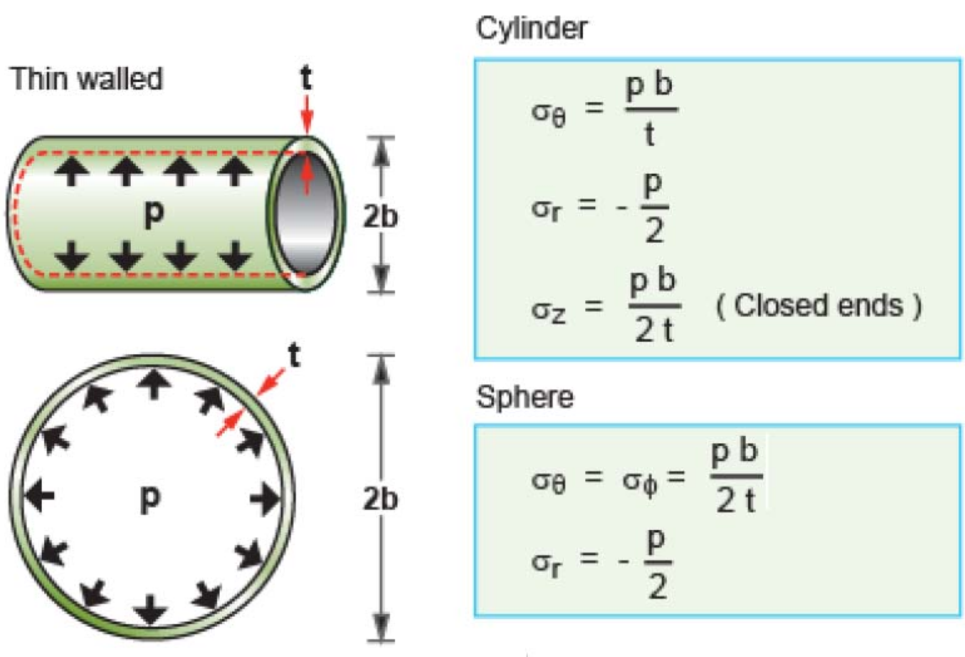
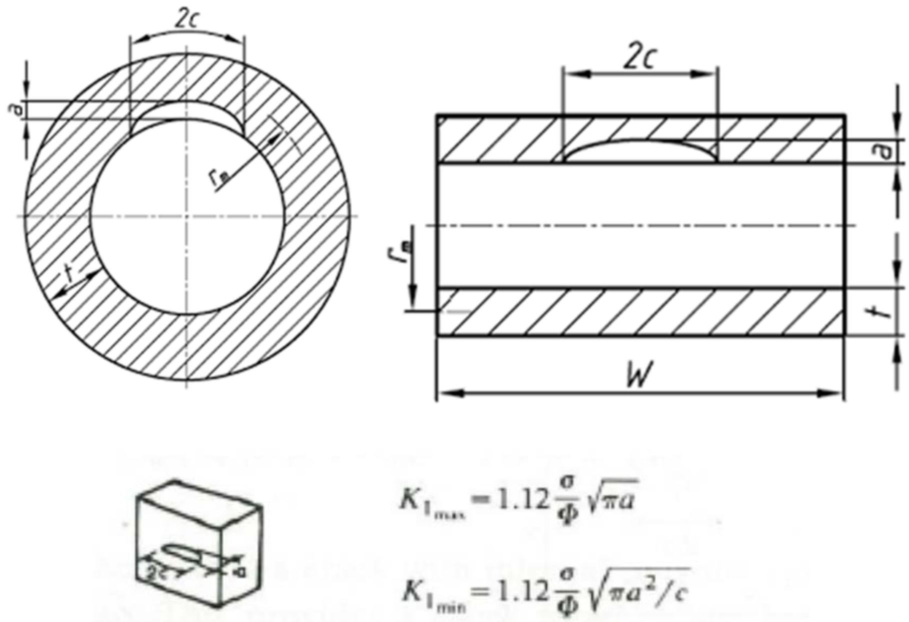


Figure 9: Stress formulas used to approximate membrane stress in pressure vessel (8).



$$K_{1_{max}} = 1.12\frac{\sigma}{\Phi}\sqrt{\pi a}$$

$$K_{1_{min}} = 1.12\frac{\sigma}{\Phi}\sqrt{\pi a^2/c}$$

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

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

Figure 10: Postulated flaws used to estimate stress intensity factors for Jefferson cell as a function of pressure (9).

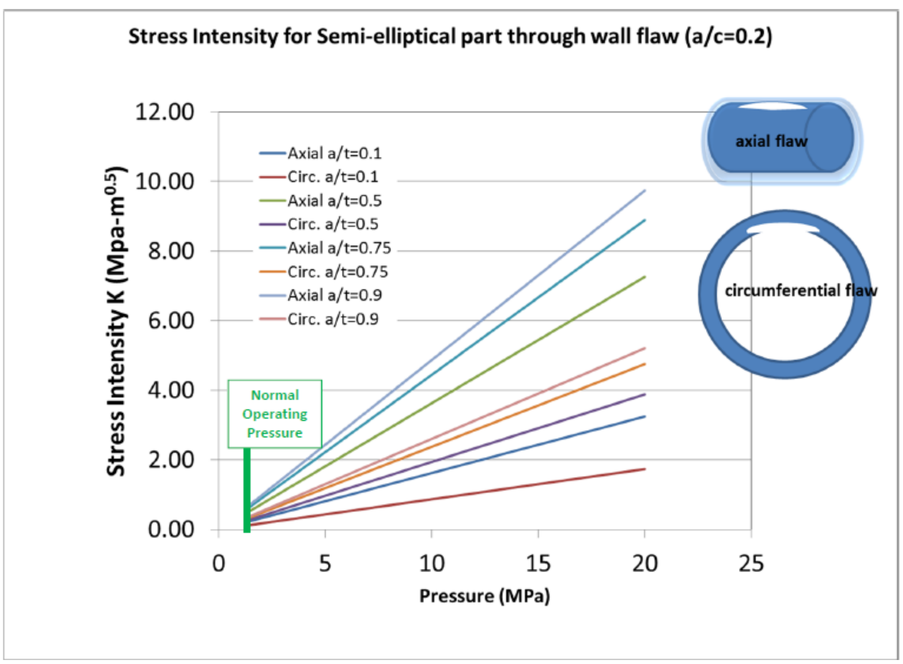


Figure 11: Estimated stress intensity factors as a function of pressure for postulated flaws in Jefferson Laboratory Tritium Cell.

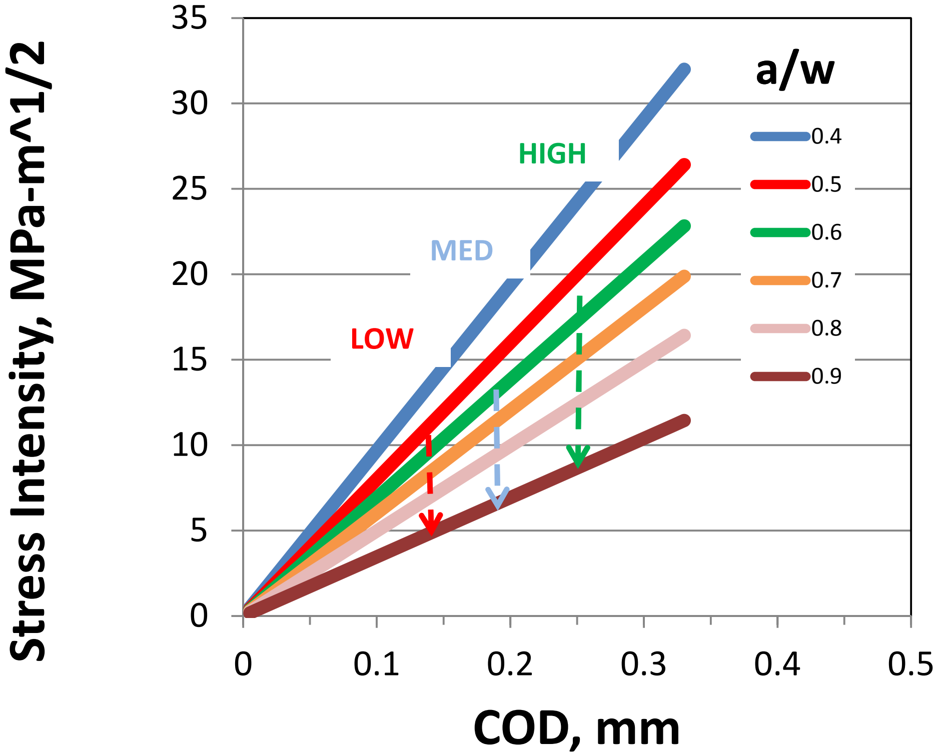


Figure 12: Relationship between crack growth and stress intensity factor for falling K slow crack growth test calculated from ASTM E1681 (4).

5.0 Summary and Conclusions

- The fracture toughness, K_{Ic}, of 7075 Aluminum was measured to be 24.2 MPa-√m for the unexposed condition in agreement with literature values.
- Literature data indicate that hydrogen-induced cracking may occur in 7075 Aluminum threshold stress intensity factors as low as ~25% K_{Ic} based on hydrogen ingress from moist air. No significant hydrogen effect on cracking is observed in dry hydrogen environments (3).
- Twenty-eight specimens were loaded to various stress intensity factors and exposed to tritium gas at a pressure similar to the Jefferson Lab cell effective pressure during beam-on conditions to determine the potential for cracking.
- Little or no crack growth was observed in 7075 Aluminum specimens stressed between 40-90% of K_{Ic} after 4, 8, and 10 months exposure to tritium gas at 17.2 MPa and ambient temperature.
- A fracture mechanics analysis of the cell shows that, since little or no crack growth was observed after 10 months and stress intensity factors expected in service are low, no crack growth is expected in the Jefferson Lab tritium cell.

6.0 Recommendations, Path Forward or Future Work

The excessive tritium surface tritium contamination levels and off-gassing rates from the aluminum specimens was unexpected. The handling and examination of the specimens was difficult and some of the examinations could not be conducted because tritium contamination levels and off-gassing rates were higher and did not diminish with time like that for stainless steel, the typical material examined at SRNL. The data suggest that tritium is tied up on the surface of the aluminum alloys after exposure and particularly after examinations in fresh air hoods. It is not clear if external tritium contamination could be transferred to the Jefferson Lab cell after filling operations in the dry nitrogen glove boxes which could make it difficult to handle. Experiments with unexposed aluminum specimens placed in the nitrogen box where filling would occur could be conducted to see if the aluminum surfaces pick up excessive contamination from the residual tritium in the box.

7.0 References

1. Measurement of the *d/u* **ratios** and A=3 EMC Effect in Deep Inelastic Electron Scattering Off the Tritium and Helium Mirror Nuclei Jefferson Lab PAC37 Proposal, December 2010 J. Arrington and The JLab MARATHON Collaboration.

2. Takahito Watakabe, Goroh Itoh and Yuji Hatano, J. Japan Inst. Met. Mater. Vol. 77, No. 12 (2013), pp. 565-570.

3. M. O. Speidel, Met. Trans A Volume 6A April 1975 “Stress Corrosion Cracking of Aluminum Alloys”.

4. ASTM E 1681-03 Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials, ASTM, PA (2013)

5. ALCOA Alloy 7075 Technical Data Sheet SPD-10-037 ALCOA Mill Products, P.O. BOX 8025, BETTENDORF, IOWA 52722, www.millproducts-alcoa.com.

6. Project: PS-TGT-14-001 Hall A Tritium Target Title: Estimated pressure in tritium cell and permeation rate Document Number: TGT-CALC-103-010 Revision: Original Author: Dave Meekins, Date: 8/13/2014, Jefferson Laboratory.

7. ASME. 2017. "Rules for Inservice Inspection of Nuclear Power Plant Components, Section XI. Appendix A - Analysis of Flaws In ASME” Boiler and Pressure Vessel Code – An International Code, American Society of Mechanical Engineers, New York.

8. Mike Ashby, **Useful solutions for standard problems**, 9th Edition, July 2011, Engineering Department, Trumpington Street, Cambridge CB2 1PZ, UK

9. David Broek, **Elementary Engineering Fracture Mechanics**, Martinus Nijhoff Publishers Publishers, AD Dordrecht, The Netherlands (1986) pp. 85-86.