FRACTURE MECHANICS TESTING OF TITANIUM 6AL-4V IN AF-M315E

J. W. Sampson and J. Martinez
NASA Kennedy Space Center
Kennedy Space Center, Florida

C. McLean
Ball Aerospace and Technologies Corp.
Boulder, Colorado

ABSTRACT

The Green Propellant Infusion Mission (GPIM) will demonstrate the performance of AF-M315E monopropellant on orbit. Flight certification requires a safe-life analysis of the titanium alloy fuel tank to ensure inherent processing flaws will not cause failure during the design life of the tank. Material property inputs for this analysis require testing to determine the stress intensity factor for environment-assisted cracking (K_{EAC}) of Ti 6Al-4V in combination with the AF-M315E monopropellant. Testing of single-edge notched, or SE(B), specimens representing the bulk tank membrane and weld material were performed in accordance with ASTM E1681. Specimens with fatigue pre-cracks were loaded into test fixtures so that the crack tips were exposed to AF-M315E at 50°C for a duration of 1,000 hours. Specimens that did not fail during exposure were opened to inspect the crack surfaces for evidence of crack growth. The threshold stress intensity value, K_{EAC}, is the highest applied stress intensity that produced neither a failure of the specimen during the exposure nor showed evidence of crack growth. The threshold stress intensity factor for environment-assisted cracking of the Ti 6Al-4V forged tank material was found to be at least 22 ksi√in and at least 31 ksi√in for the weld material when exposed to AF-M315E monopropellant.

INTRODUCTION

The Green Propellant Infusion Mission (GPIM), managed by Ball Aerospace and funded by the Space Technology Mission Directorate (STMD) at NASA Headquarters, will demonstrate the in-space performance of a new monopropellant, AF-M315E. Developed by the Air Force Research Laboratory (AFRL), AF-M315E provides a higher density and increased specific impulse relative to hydrazine. Because this hydroxylammonium nitrate blend has a lower vapor pressure than hydrazine, it does not require the same degree of personal protective equipment (PPE) during servicing. It is expected that this reduction in PPE will lower the cost of propellant handling. In flight, the propellant will be contained in a pressurized propellant tank on the GPIM satellite. A fracture mechanics analysis is required to verify the safe design life of the pressurized tank for certification by range safety. The objective of the fracture mechanics analysis is to model operating stresses so that a preexisting flaw of an assumed maximum initial size will not grow to a critical size during the service life of the propellant tank. The analysis shows that any crack large enough to cause tank failure would have been seen during inspection. Inputs for this analysis include the crack growth properties of the tank material when exposed to the propellant.

Since NASA's Apollo program, titanium alloy Ti 6Al-4V has been the material of choice for propellant pressure vessels because of its high strength-to-weight ratio and its resistance to corrosion. The structural integrity of Ti 6Al-4V pressure vessels has been studied since the late

1960s. Included in these studies was the sustained load flaw growth of the Ti 6Al-4V and welds of Ti 6Al-4V alloy. Crack growth testing was carried out using common propellants and oxidizers in use during the time period, which included hydrazine, monomethyhydrazine, Aerozine 50, and nitrogen tetroxide. The threshold stress intensity factor for environment-assisted crack growth (K_{EAC}) was determined through test. Since then, all pressurized propellant tanks used on NASA spacecraft have used these combinations of tank material and propellants. With the development of AF-315E monopropellant, testing is required to determine the KEAC of the titanium tank when exposed to this new monopropellant.

The testing documented herein investigates the threshold stress intensity factor for environment-assisted cracking (K_{EAC}) of Ti 6Al-4V in combination with AF-M315E for input into a fracture mechanics analysis. A team of engineers at NASA's Kennedy Space Center (KSC), ATK Space Systems, Air Force Research Laboratories (AFRL) at Edwards Air Force Base, and Ball Aerospace have developed procedures and test hardware to perform this testing. Because the flight tank is fabricated by welding two hemispherical forgings into a sphere, testing will include specimens representing the bulk forging and the weld. Testing of the weld material will be especially important because the design of the tank precludes the possibility of post weld aging heat treatments. Testing will be performed according to ASTM E1681, Standard Test Method for Determining Threshold Intensity Factor for Environment-Assisted Cracking of Metallic Materials. Upon completion of the testing, the resultant stress intensity threshold values will be provided to ATK for analysis using NASGRO[1] fracture analysis software. Historically NASGRO fracture analysis has been performed using design recommendations for Ti 6Al-4V forgings and un-aged welds by Lewis and Kenny[2]. This reference documented testing of uniaxially loaded fracture mechanics specimens containing part-through cracks, where the crack tips were exposed to hydrazine under a sustained load for 24 hours.

PROCEDURES

The purpose of the testing is to produce stress intensity threshold values for Ti 6Al-4V in AF-M315E monopropellant in accordance with ASTM E1681. For this test single-edge bend specimens, annotated as SE(B) specimens, were fatigued to grow sharp crack tips. SE(B) specimens were loaded into test fixtures so that the crack tips were exposed to the propellant at 50°C for a duration of 1,000 hours. This temperature represents the highest temperature that the flight tank is expected to experience on orbit and the worst case for corrosive effects of the monopropellant. The duration of the test was dictated by ASTM E1681. Upon completion of the exposure, SE(B) specimens that did not fail were marked with post-test fatigue cracks. Specimens were then opened to inspect the crack surfaces for evidence of growth during environmental exposure. The threshold stress intensity value, K_{EAC}, is the highest applied stress intensity that produced neither a failure of the specimens during the exposure nor showed evidence of crack growth.

SPECIMEN PREPARATION

The test material, shown in Fig. 1 included a Ti 6Al-4V forging and a weld verification ring. The Ti 6Al-4V forging was provided in the solution treated and aged (STA) condition to represent the bulk tank membrane. The weld verification ring was unaged and represented the Ti 6Al-4V weld on the GPIM tank.

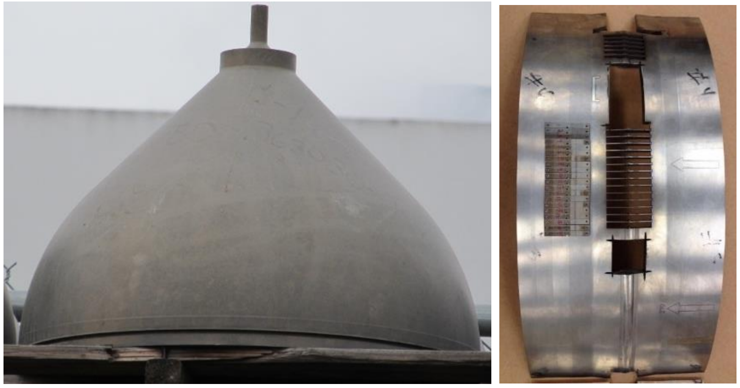

Figure 1. Ti 6Al-4V material. SE(B) specimens were machined from the bulk forging (left) and weld verification ring (right).

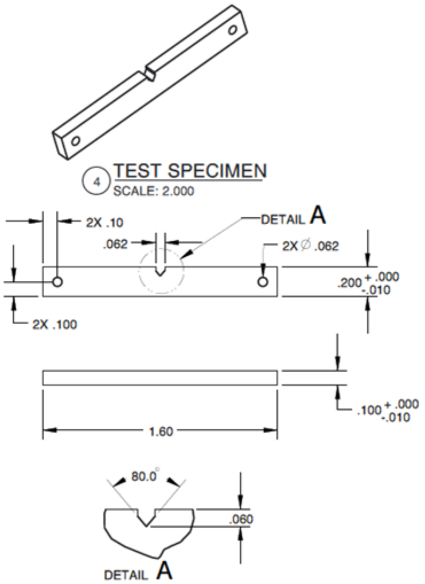

Figure 2. Specimen drawing. Single edge bend specimen, SE(B), dimensions in inches.

SE(B) specimens were machined to the dimensions shown in Fig. 2. Bulk specimens were cut from the forging so that cracks would grow in the L-S direction, which corresponds to crack propagation from hoop stress in a pressurized tank. Weld specimens were cut from the weld ring with the crack in the through-thickness plane and growing parallel to the direction the weld solidification. Specimens were cut using a wire electrical discharge machine (EDM). The faces of the specimen were ground to remove the recast layer and polished to enable view of the pre-cracks on the sidewall of the specimen.

TENSILE, METALLOGRAPHY, AND MICROHARDNESS

It was necessary to determine the tensile properties, specifically the yield strength, of the materials in order to calculate the validity of the fracture toughness and threshold stress intensity results. Tensile properties were determined referencing ASTM E8 for both the bulk material and the weld. Sub-size specimens were cut using the wire EDM. Gauge length was reduced from 1.0 inches to 0.75 inches because of the size of the sections available for testing. Bulk specimens were tested in the wrought direction and weld specimens were tested perpendicular to the direction of the weld solidification.

Metallographic specimens were prepared from the weld ring per ASTM E3. This enabled machining of the SE(B) specimens so that the notch and crack were contained in the weld metal. To locate the heat affected zone (HAZ) from welding, Vickers microhardness testing was performed with a 500 gram-force load in accordance to ASTM E384.

FATIGUE PRE-CRACKING

Fatigue pre-cracks were induced at the notched SE(B) specimens using an MTS 810 servo-hydraulic load frame. A three-point bending test fixture in Fig. 3 was configured referencing ASTM E399 Annex 2 with a load span of 0.8 inch. Cracks were grown using a force shedding method with stress ratio, R, of 0.1. Depending on the fatigue crack length, the maximum load in each cycle was either 110, 90, or 70 lb. Using Eq. (1), the stress intensity was never above 15 ksi√in to prevent a plastic deformation at the crack tip. The fatigue crack length was monitored on the sidewalls of the specimen, as is shown in Fig. 3, using a Keyence digital stereomicroscope. The target length for the fatigue cracks was 0.10±0.01 inch. Specimens were cleaned prior to loading in the test fixtures per ASTM G1.

$$K = \frac{PS}{BW^{3/2}} * 3\sqrt{\frac{a}{W}} * \frac{1.99-\left(\frac{a}{W}\right)*\left(1-\frac{a}{W}\right)*\left[(2.15-3.93\left(\frac{a}{W}\right)+2.7\left(\frac{a}{W}\right)^{2}\right]}{2*\left(1+2*\frac{a}{W}\right)*(1-\frac{a}{W})^{3/2}} \quad (1)$$

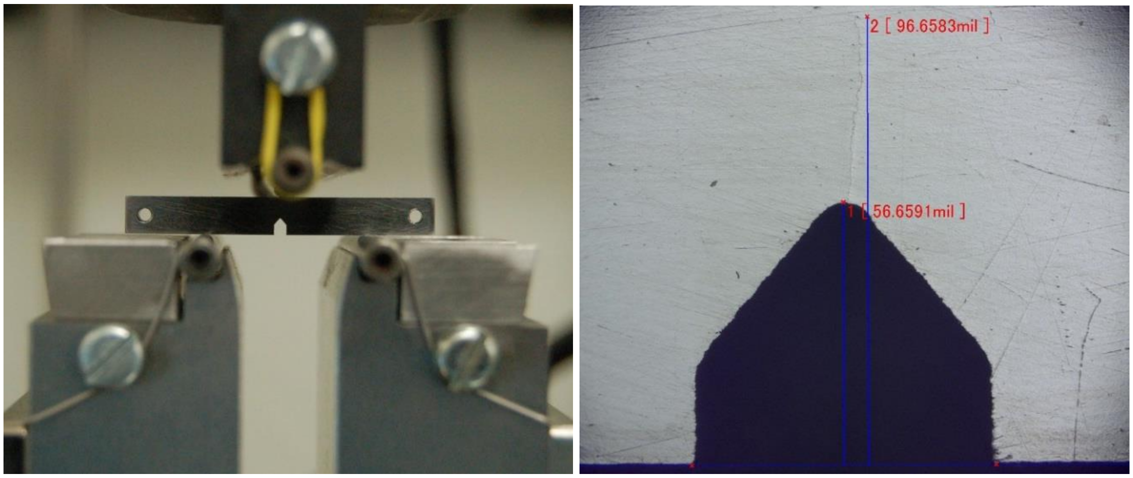

Figure 3. SE(B) specimens in three-point bend fixture (right) and crack length measurement with a stereomicroscope (left).

FRACTURE TOUGHNESS

Fracture toughness testing was performed on specimens representing both the bulk tank material and the weld material in accordance with ASTM E399. Results were used to design and build test fixtures and weights for the threshold stress intensity testing that followed.

PROPELLANT EXPOSURE

Air Force Research Laboratory (AFRL) at Edwards Air Force Base designed test fixtures for loading the specimens while they are exposed to the AF-M315E monopropellant in an oven set to 50°C for 1,000 hours. A cantilever bending apparatus shown in Figure 4 was designed to apply stress intensities up to 80% of the fracture toughness. The fixture was designed such that the notch and crack were surrounded by a flask, which contained the AF-M315E monopropellant. The flask contained through holes for the specimen that were sealed with a rubberized sealant. The test fixtures were designed to hold a set of twelve specimens that were dead weight loaded, exposed to the propellant, and placed in an oven for the duration of the test.

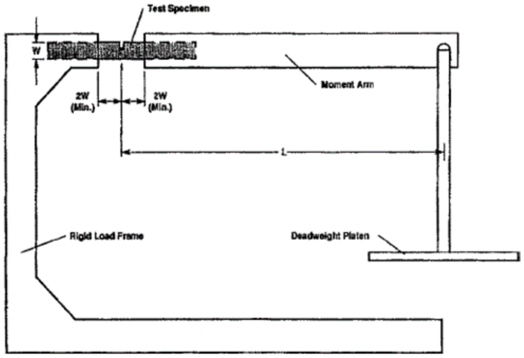

Figure 4. Loading Fixture from ASTM E1681.

POST TEST ANALASIS

After exposure, the fracture surfaces were prepared for viewing with a stereomicroscope. The pre-cracked fracture surface and any crack growth form the threshold testing were marked with an oxide coating in an oven set to 300°C for 30 minutes. Post-test fatigue cracks were then grown using same the pre-cracking fatigue method. This allowed for crack growth during the environmental exposure to be framed between the pre- and post-fatigue cracks. Test specimens were then broken open to view the fracture surfaces. The initial fatigue crack length was measured and the stress intensity of each specimen was calculated using Eq. (2) from ASTM E1681.

$$K = \frac{W_a L_a + W_t L}{B W^{3/2}} * \frac{6\left(\frac{a}{W}\right)^{1/2}}{\left(1-\frac{a}{W}\right)^{3/2}} * \left\{1.9878 - 1.3253\left(\frac{a}{W}\right) + \left(1-\frac{a}{W}\right)\left(\frac{a}{W}\right)\left[-3.8308 + 10.1081\left(\frac{a}{W}\right) - 17.9415\left(\frac{a}{W}\right)^2 + 16.8282\left(\frac{a}{W}\right)^3 - 6.2241\left(\frac{a}{W}\right)^4\right]\right\} \quad (2)$$

RESULTS AND DISCUSSION

Three rounds of SE(B) specimens were exposed to the AF-M315E monopropellant for the duration of the 1,000-hour test. The test specimens representing both the bulk and weld materials were tested at a range of effective K_{EAC} values. After testing, specimens were evaluated to the requirements of ASTM E1681.

TENSILE PROPERTIES, METALLOGRAPHY, AND MICROHARDNESS

Tensile results of the bulk material and weld are displayed in Table 1. The yield strength of the bulk averaged 156 ksi and the tensile strength was 166 ksi. The yield strength of the material in the weld averaged 140 ksi with a tensile strength of 154 ksi. The micrograph of the weld, along with the Vickers microhardness numbers are shown in Fig. 5.

Table 1. Results of Tensile Testing.

Material	Specimen	Thickness (in)	Width (in)	Gauge Length (in)	Maximum Load (lbf)	Yield Strength-Offset 0.2 % (ksi)	Tensile Strength (ksi)	Elongation at Failure (%)
Bulk	B-T1	0.0960	0.2340	0.6504	3727	155.9	165.9	15.7
Bulk	B-T2	0.0960	0.2450	0.6728	3881	156.8	165.0	15.5
Weld	W-T1	0.0860	0.2460	0.6415	3256	139.0	153.9	4.0
Weld	W-T2	0.0845	0.2460	0.6455	3185	140.7	153.2	5.2
Weld	W-T3	0.0850	0.2460	0.6645	3213	141.3	153.7	4.6

EAR 99 -- NO LICENSE REQUIRED
The information contained in the document is technical in content, but not technical data as defined by the International Traffic in Arms Regulations (ITAR) or the Export Administration Regulations (EAR), and therefore is EAR 99 NLR, no export license required. [General Prohibition Six (Embargo) applies to all items subject to the EAR, (i.e., items on the CCL and within EAR 99 NLR). You may not make an export or re-export contrary to the provisions of part 746 (Embargos and Other Special Controls) of the EAR and 22 CFR part 126.1 of the ITAR].

Reference EDR #1631066: NASA KSC Export Control Office, 321-867-9209

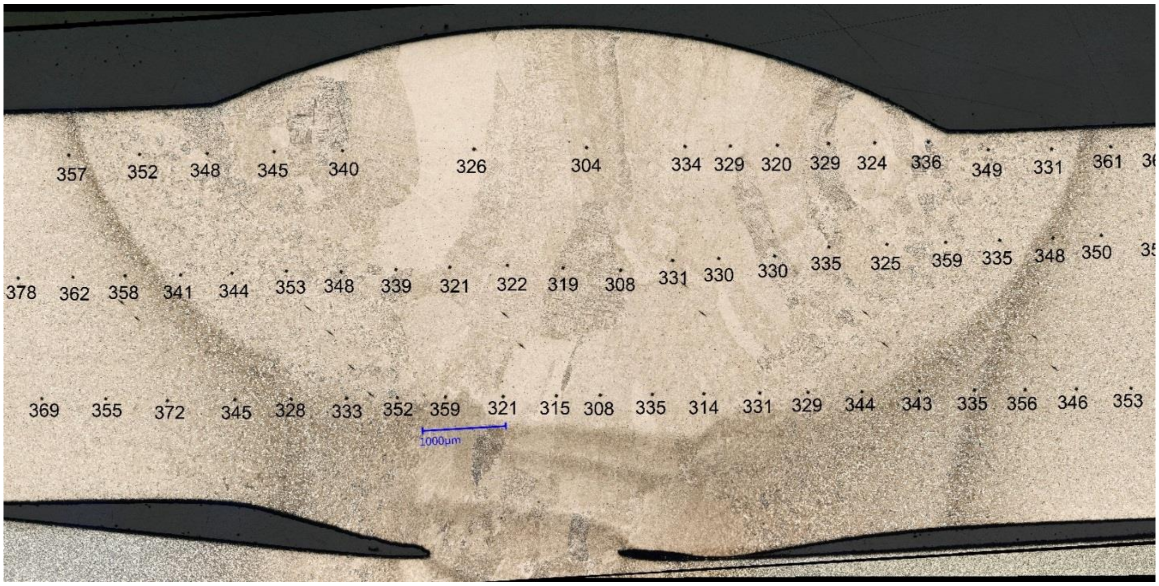

Figure 5. Micrograph of weld. Polished section of weld etched with Kroll's reagent. Vickers microhardness (HV) numbers tested with a 500 gram-force load.

FRACTURE TOUGHNESS RESULTS

Fracture toughness test results are listed in Table 3. The plane-strain validity criteria in ASTM E399 section 9.1.4 was satisfied for neither the bulk nor the weld specimens. Therefore results could not be reported as the plane-strain fracture toughness, K_{Ic}, but rather the conditional fracture toughness result, K_Q. Additionally, the weld specimens failed ASTM E399 section 9.1.3 criteria because of plastic deformation at the crack tip during failure. Further testing of fracture toughness for these weld specimens should be according to ASTM E 1820 to calculate the J_{Ic}, critical elastic-plastic fracture toughness. The average K_Q for the bulk specimens was 45.0 ksi√in and the weld specimens was 49.0 ksi√in.

Table 2. Results of fracture toughness testing. Fracture toughness was tested per ASTM E399.

Material	Specimen	Initial Crack Length, a_0 (in)	P_q (lbf)	P_{max} (lbf)	P_{max}/P_q	Fracture Toughness, K_Q (ksi√in)
Bulk	B01	0.1085	143.2	145.8	1.02	45.4*
Bulk	B02	0.1037	154.6	161.7	1.05	45.1*
Bulk	B03	0.0986	166.1	169.5	1.02	44.5*
Weld	W01	0.0890	214.1	268.7	1.26	49.8†
Weld	W02	0.1033	172.2	216.8	1.26	49.5†
Weld	W03	0.0935	212.1	257.7	1.21	50.0†
Weld	W04	0.1023	166.1	208.8	1.26	46.6†

* *Invalid according to section 9.1.4 of test method ASTM E399*

† *Invalid according to sections 9.1.3 and 9.1.4 of test method ASTM E399*

THRESHOLD STRESS INTENSITY FACTOR RESULTS

Results of the stress intensity threshold testing per ASTM E1681 are listed in Table 4. Bulk and weld specimens failed ASTM E1681 section 9.3.1 validity check for K_{IEAC} where Eq. (3) is less than B, a0, and W-a0. A less restrictive validity check for K_{EAC} was calculated per ASTM E1681 section 9.3.2, where Eq. (4) is less W-a_0. The bulk material passes this criteria at stress intensities less than 43 ksi√in and the weld specimens pass this criteria at stress intensities less than 37 ksi√in. Plane-strain conditions would have been ideal but could not be achieved with these specimens due to the thickness constraints of the material provide for this test.

$$2.5\left(\frac{K_{EAC}}{\sigma_{YS}}\right)^2 \tag{3}$$

$$\frac{4}{\pi}\left(\frac{K_{EAC}}{\sigma_{YS}}\right)^2 \tag{4}$$

Eight of the twelve specimens from the first round of testing failed during dead weight load application. The remaining four specimens showed evidence of crack growth at the fatigue crack. Magnified photographs of some fracture surfaces from the first round of testing are shown in Fig. 6.

The second round of testing included twelve specimens. No specimen failed and after post-test evaluation and no crack growth was detected in any of the twelve. Three of eight specimens representing the weld materials were not exposed to AF-M315E and were used as a baseline, but are not documented in this report. Magnified photographs of some facture surfaces from the second round of testing are shown in Fig. 7.

The third round of testing included twelve specimens. No specimen failed and after post-test evaluation and no crack growth was detected in any of the twelve specimens. Magnified photographs of some facture surfaces from the third round of testing are shown in Fig. 8.

Table 3. Results from first round of stress intensity threshold testing.

Material	Specimen	Thick-ness, B (in)	Width, W (in)	Initial Crack Length, a_0 (in)	Stress Intensity, K_{EAC} (ksi√in)	Test Result
Bulk	B08	0.0955	0.1970	0.0930	36.4	Crack growth detected after 1000 hours exposure to AF-M315E at 50°C
Bulk	B16	0.0950	0.1965	0.0942	37.6	Crack growth detected after 1000 hours exposure to AF-M315E at 50°C
Bulk	B20	0.0950	0.1965	0.0965	38.8	Crack growth detected after 1000 hours exposure to AF-M315E at 50°C
Bulk	B06	0.0950	0.1970	0.0924	38.9	Immediate fracture upon loading
Bulk	B11	0.0950	0.1970	0.0945	40.2	Crack growth detected after 1000 hours exposure to AF-M315E at 50°C
Bulk	B15	0.0950	0.1970	0.0984	42.6	Immediate fracture upon loading
Bulk	B14	0.0950	0.1960	0.0956	44.3	Immediate fracture upon loading
Bulk	B12	0.0955	0.1970	0.0953	46.0	Immediate fracture upon loading
Bulk	B19	0.0950	0.1960	0.0956	47.1	Immediate fracture upon loading
Bulk	B10	0.0950	0.1965	0.0969	47.7	Immediate fracture upon loading
Bulk	B22	0.0950	0.1965	0.1010	47.9	Fracture approx. 5 minutes after loading
Bulk	B18	0.0955	0.1965	0.1074	52.8	Immediate fracture upon loading

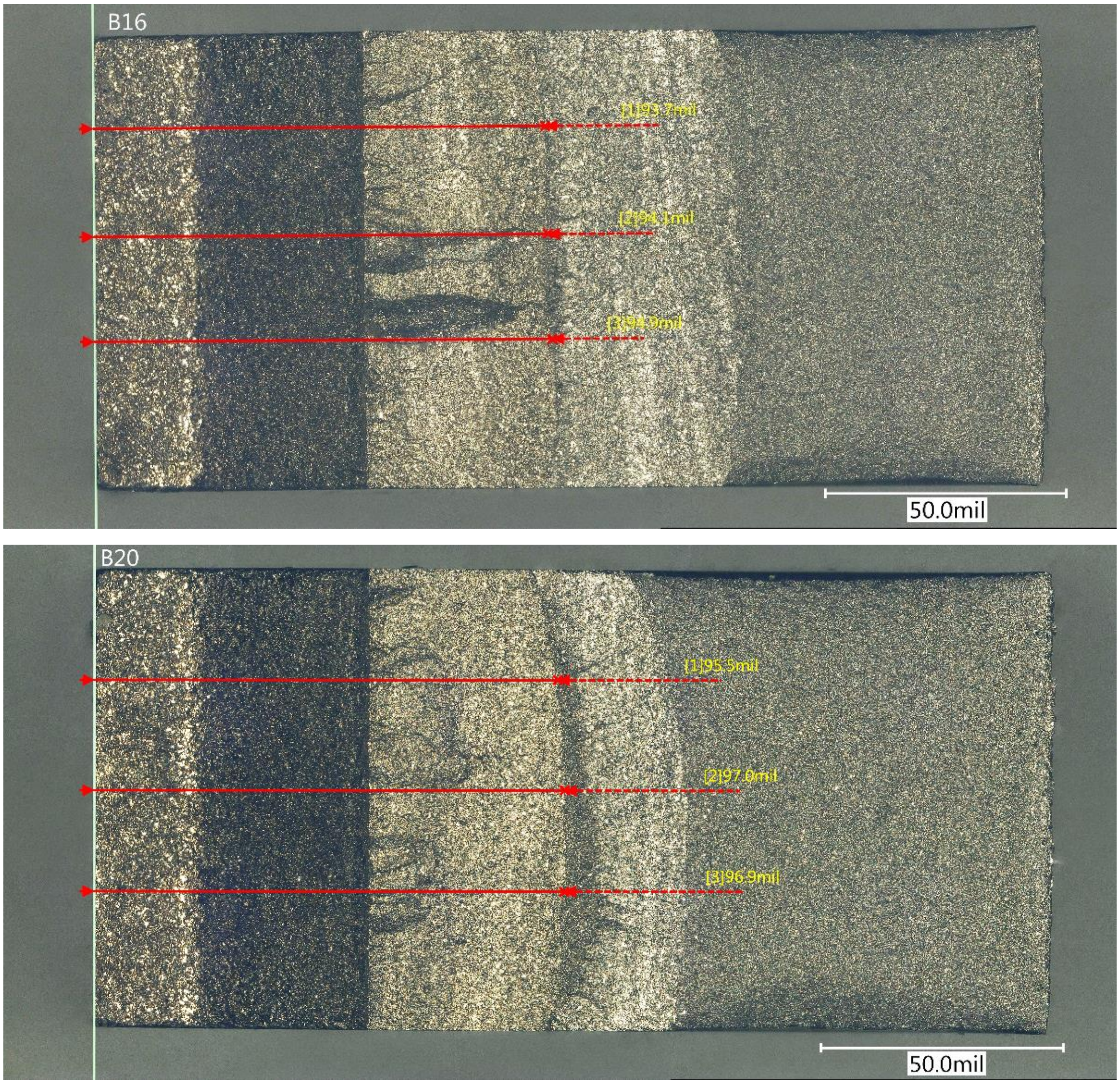

Figure 6. Fracture surfaces of bulk specimen B16 (top) and B20 (bottom) showed evidence of crack growth after 1,000 hours of exposure to AF-M315E at 50˚C. The length of the fatigued pre-crack is annotated.

Table 4. Results from second round of stress intensity threshold testing.

Material	Specimen	Thick-ness, B (in)	Height, W (in)	Initial Crack Length, a_0 (in)	Stress Intensity, K_{EAC} (ksi√in)	Test Result
Bulk	B26	0.0950	0.1970	0.1045	22.0	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B27	0.0950	0.1970	0.1038	22.5	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B28	0.0950	0.1965	0.0979	21.6	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B29	0.0950	0.1975	0.1036	22.1	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W08	0.0910	0.1980	0.1033	30.8	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W10	0.0920	0.1980	0.1089	32.4	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W12	0.0930	0.1990	0.1040	31.2	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W13	0.0915	0.1980	0.1019	31.4	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W14	0.0920	0.1985	0.1050	30.7	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C

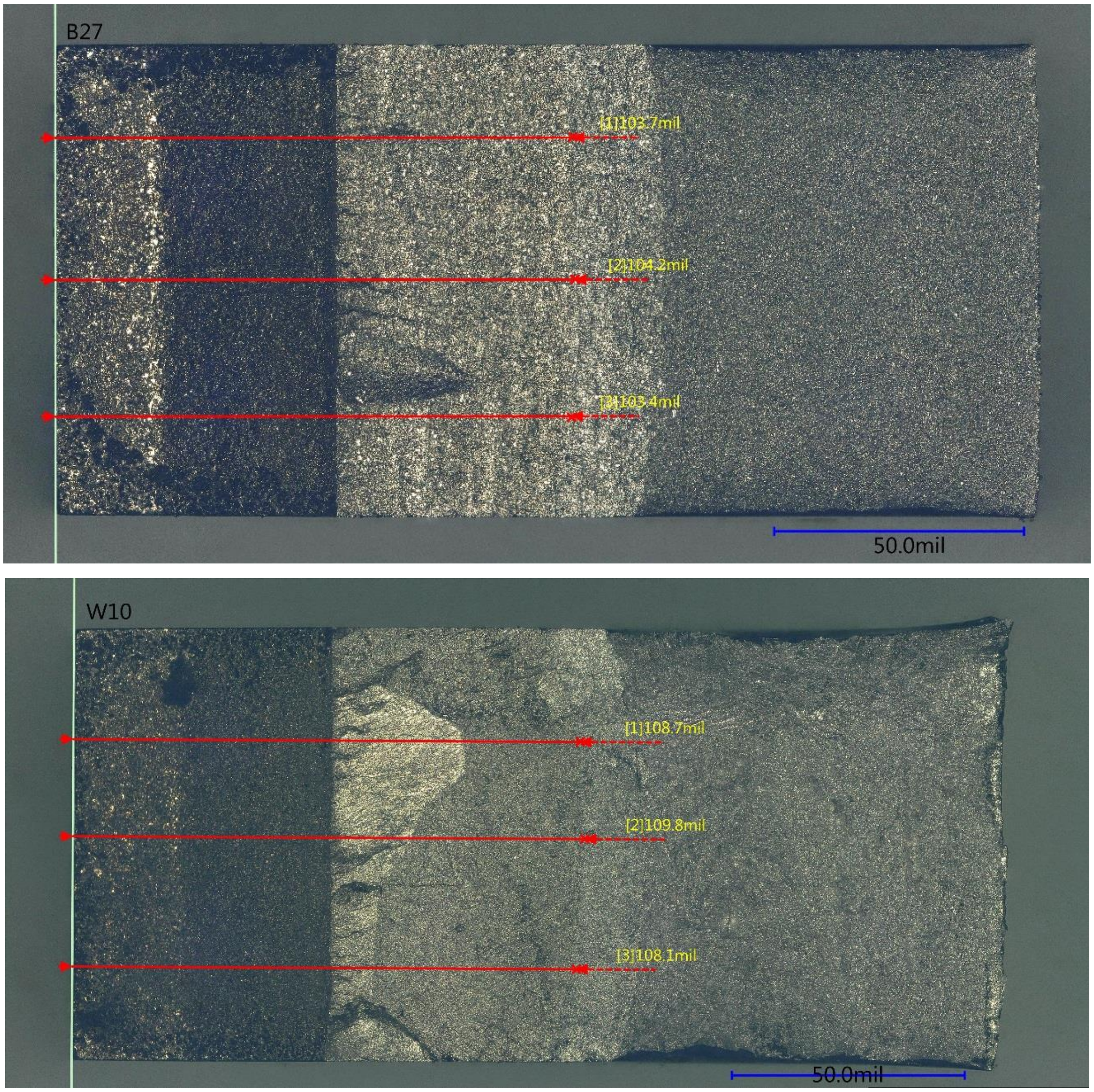

Figure 7. Fracture surfaces of bulk specimen B27 (top) and weld specimen W10 (bottom). There was no crack growth after 1,000 hours of exposure to AF-M315E at 50˚C. The length of the fatigued pre-crack is annotated**.**

Table 5. Results from third round of stress intensity threshold testing.

Material	Specimen	Thick-ness, B (in)	Height, W (in)	Initial Crack Length, a_0 (in)	Stress Intensity, K_{EAC} (ksi√in)	Test Result
Bulk	B31	0.0840	0.1955	0.1034	21.6	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B32	0.0835	0.1950	0.1049	22.9	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B33	0.0890	0.1960	0.1011	22.7	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B34	0.0880	0.1970	0.1027	21.8	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B35	0.0875	0.1980	0.1023	21.3	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Bulk	B36	0.0875	0.1965	0.1046	21.1	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W22	0.0930	0.1990	0.1055	30.5	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W23	0.0915	0.1980	0.0950	28.7	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W24	0.0920	0.1985	0.0994	29.2	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W25	0.0925	0.1980	0.1067	31.0	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W27	0.0925	0.1990	0.1081	32.3	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C
Weld	W26	0.0920	0.1985	0.1048	30.8	No failure or crack growth after 1,000 hours of exposure to AF-M315E at 50°C

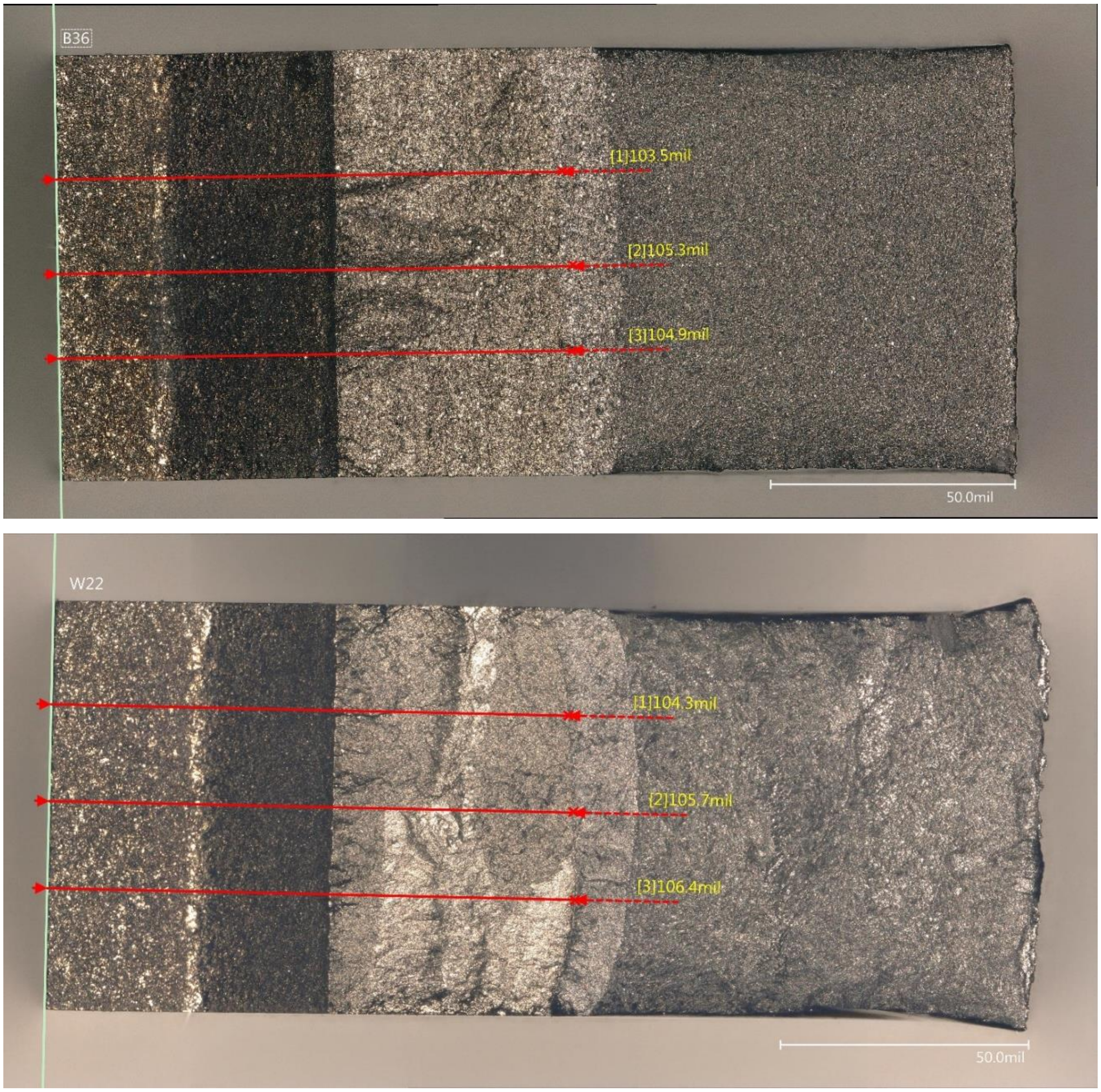

Figure 8. Fracture surfaces of bulk specimen B36 (top) and weld specimen W22 (bottom). There was no crack growth after 1,000 hours of exposure to AF-M315E at 50˚C. The length of the fatigued pre-crack is annotated.

SUMMARY AND CONCLUSIONS

The threshold stress intensity factor for environment-assisted cracking was determined for titanium alloy propellant tanks when exposed to AF-M315E monopropellant. Material representing the Ti 6Al-4V tank forging was found to have a K_{EAC} of at least 22 ksi√in, and the weld material was found to be at least 31 ksi√in. Plane-strain conditions would have been ideal but could not be achieved due to the thickness constraints of the material provided for testing. However, the bulk forging used to produce these test specimens is considerably thicker than the tank wall of the GPIM flight tank. Since fracture toughness decreases as material thickness and width increases[3], the threshold stress intensity results are considered conservative for analysis of the GPIM propellant tank.

FUTURE WORK

Not all combinations of materials and environment will result in environment-assisted cracking. Subcritical crack growth will result at stress intensity values close to the fracture toughness of the material in any environment. Future work is recommended to further refine the environment-assisted crack threshold of Ti 6Al-4V tanks when exposed to AF-M315E. Additional testing of the bulk material between stress intensities in the range of 22-35 ksi√in and weld material in the range of 33-49 ksi√in will result in an increased factor of safety for the safe-life analysis of GPIM flight tanks.

ACKNOWLEDGMENTS

Authors would like to thank Tim Smith of NASA Marshall Space Flight Center; Amy Brown of Ball Aerospace; Walter Tam and Richard Bahng of Orbital ATK; Paul Zuttarelli, Adam Brand, and Jeffrey Jacobs at Edwards Air Force Base for all programmatic and testing support.

REFERENCES

1. **NASGRO**, Southwest Research Institute, San Antonio, Texas.
2. Lewis, C.J. and Kenny, J.T. **Sustained Load Crack Growth Design Data for Ti-6Al-4V Titanium Alloy Tanks Containing Hydrazine**, AIAA/SAE 12th Propulsion Conference. AIAA Paper No. 76-769, 1976
3. Hertzberg R. W., **Deformation and Fracture Mechanical of Engineering Materials**, 4th ed., John Wiley & Sons, Inc., 1996, pp 342-343