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STRESS-CORROSION CRACKING OF Ti-6A1-4V ALLOY IN METHANOL

by Robert L. Johnston, Robert E. Johnson, Glenn M. Ecord, and Willard L. Castner

Manned Spacecraft Center
Houston, Texas

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

The report presents the results of an investigation to determine if an incompatibility exists between methanol and Ti-6Al-4V solution-treated and aged alloy. The test specimens were obtained from virgin solution-treated-and-aged sheet material and from the remnants of two Apollo service propulsion system fuel tanks which failed while containing methanol under pressure. The investigation shows that methanol and stressed Ti-6Al-4V alloy are incompatible and result in tank failure because of a stress-corrosion mechanism.

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SUMMARY

An investigation was undertaken to determine if an incompatibility of methanol with Ti-6Al-4V solution-treated and aged alloy contributed in major proportion to failures of pressurized Apollo fuel tanks containing methanol. This paper presents the results of fatigue, constant load, ultimate tensile strength, and cathodic protection tests on the alloy exposed to various fluids, including methanol. Also presented are data from tests designed to gain preliminary insight into the mechanism of the methanol attack and subsequent material failure. Specimens were obtained from samples of ruptured Apollo fuel tanks and from virgin solution-treated-and-aged titanium alloy sheet. Test results indicated that the alloy is highly notch-sensitive in methanol. Stress-corrosion cracking in the alloy when exposed to methanol was also indicated. Methods for preventing this attack have not been investigated in depth; however, there is evidence that moisture (as little as 1 percent) in methanol appreciably inhibits the adverse reaction.

INTRODUCTION

During spacecraft systems checkout, the liquid propellant tanks of the Apollo service module are subjected to pressurizations and loadings with simulated propellants. Because of their physical properties and flow characteristics, methanol and Freon MF have been selected to simulate Aerozine-50 and N_2O_4, respectively, for cold-flow testing of the service propulsion system. These simulated propellants enable realistic testing without creating the hazards associated with the use of this hypergolic combination of propellants. Although several spacecraft systems which included these fuel tanks have been successfully tested, failures of two fuel tanks occurred while the tanks were under pressure and filled with methanol. These failures suggested the possibility of tank material with inferior mechanical properties or a basic incompatibility of the methanol with the tank material, Ti-6Al-4V solution-treated and aged (STA) alloy.

As a result, an investigation was undertaken at the NASA Manned Spacecraft Center (MSC) to determine, first, if an incompatibility of methanol with the Ti-6Al-4V-STA fuel-tank alloy existed. Then, the investigation was extended to determine whether such an incompatibility contributed in major proportion to the fuel tank failures. High-stress fatigue cycling of fuel-tank specimens in a variety of fluids, including methanol, was performed to provide, in the shortest possible time, comparison data for an insight into the problem. These preliminary fatigue test data did, indeed, indicate that specimens immersed in methanol would fail in relatively few cycles. Hence, following this preliminary indication that a methanol environment might significantly affect the service life of the Ti-6Al-4V-STA alloy, constant load tests on the same types of specimens were considered to be the most expeditious means of proving the incompatibility of methanol and Ti-6Al-4V-STA alloy. Fluids used in the tests were methanol, distilled water, air, isopropyl alcohol, Freon MF, ethylene glycol/water solution, and Aerozine-50.

Specimens from virgin Ti-6Al-4V-STA sheet with no previous exposure to methanol were tested for comparison of data with specimens from the failed tanks. Tensile tests were performed on specimens which survived the constant load tests to determine the effects of the combinations of various fluids and stresses on the ultimate tensile strength of the alloy. A few cathodic protection tests were performed to find a quick means of gaining insight into the mechanisms of the methanol/titanium alloy incompatibility problem.

To confirm the incompatibility of titanium with anhydrous methanol, several companies, NASA centers, and other government agencies were asked to perform the same types of tests as those being performed by MSC. Information tending to confirm the results of the MSC investigation has been received from the Grumman Aircraft Engineering Corporation, the Space and Information Systems Division of North American Aviation, Langley Research Center, Marshall Space Flight Center, the Martin Company, the Naval Research Laboratories, and the Boeing Company.

The authors wish to thank the Titanium Metals Corporation of America for expeditiously providing the virgin Ti-6Al-4V-STA alloy sheet used in this study.

TEST PROGRAM

Materials and Specimen Preparation

Test specimens. - Test specimens were taken from both failed tanks and virgin sheet material. The specimens from the failed service module fuel tanks (Apollo spacecraft 017 and 101) were obtained from the parent metal of the tanks, as well as from welded areas, with the longitudinal axes of the specimens parallel to the longitudinal axes of the tanks. Samples were taken from the side of the tank opposite the cracks (figs. 1 and 2). The virgin specimens were machined from Ti-6Al-4V-STA sheet

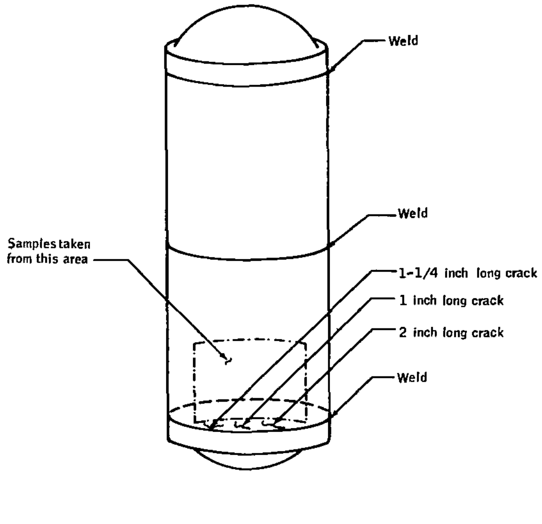


Figure 1. - Spacecraft 101 failed tank showing failure pattern and location of samples used for specimens. (Not to scale.)

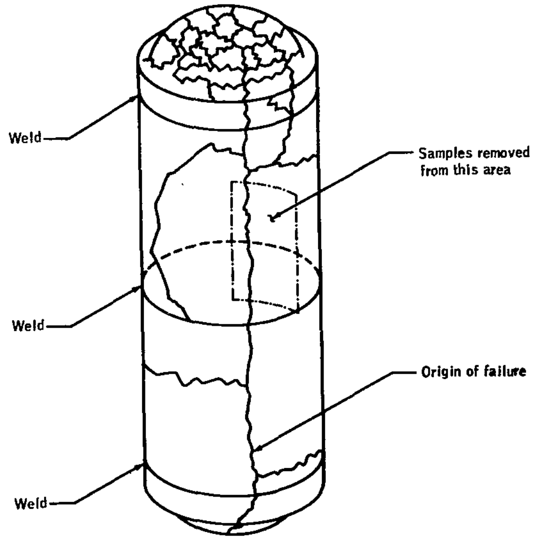


Figure 2. - Spacecraft 017 failed tank showing failure pattern and location of samples used for specimens. (Not to scale.)

which had no previous methanol exposure. The STA tank material differed from the STA virgin sheet in that the tank material came from a forging and, in addition, had been stress-relieved after being welded. The stress-relieving process left a blue oxide film on the tank surface. Most specimens were the notched (fig. 3) and standard unnotched (0.5 inch by 8.0 inches) shapes with the exception of two double test-section specimens. These specimens were twice as long as the standard specimens and contained two active gage lengths in series (fig. 4). The purpose of testing double test-section specimens was to determine the effect of prefracturing on ultimate tensile strength. After having been subjected to constant loading until one section failed, the unbroken section (in which cracks had started during the constant load testing) was tested to determine ultimate tensile strength.

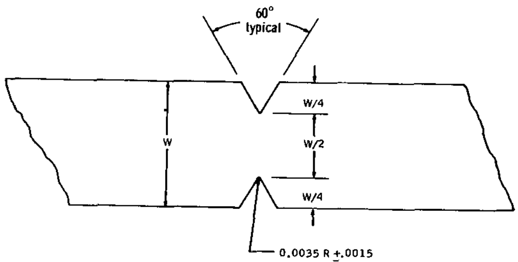


Figure 3. - Notched specimen. Notches opposite within 0.001 inch.

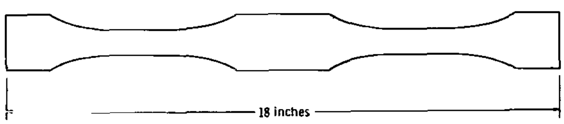


Figure 4. - Double test-specimen. Gage lengths are contoured according to American Society for Testing Materials Standard E-8.

The 2-inch gage length of the standard test specimens was enclosed within a cup made of glass tubing when the test fluid was other than air (fig. 5). These cups were sealed on the bottom with paraffin when the testing fluid was distilled water, methanol, isopropyl alchol, ethylene glycol, or water with sodium

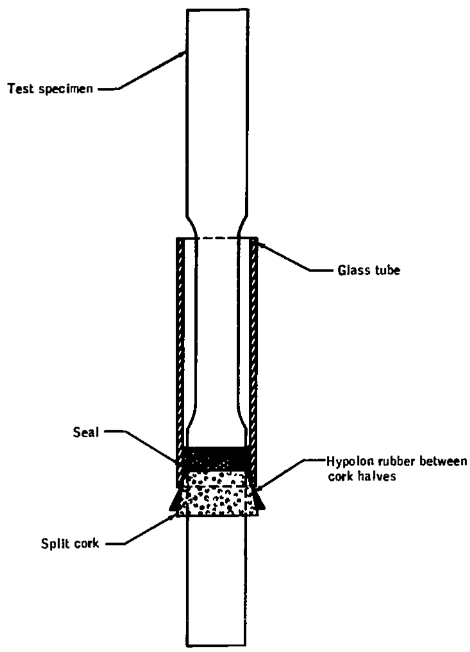


Figure 5. - Test specimen with fluid cup.

chromate inhibitor; Dow Silastic 732 RTV was used as the sealant when the test fluid was Freon MF; and Apiezon grease was used when the test fluid was Aerozine-50. No specific tests were conducted to determine the effects of the sealants on the test fluids or specimens, but no apparent reaction was noted. In the case of Aerozine-50, a piece of plastic sheet was wrapped around the cup and specimen to protect the fluid from atmospheric contamination.

X-ray examination. - Prior to testing, the material from the spacecraft 101 and spacecraft 017 fuel tanks was X-ray inspected to determine the presence of cracks which could have caused the tanks to fail. No crack indications were noted during this X-ray examination. Specimens which did not fail during constant load testing were X-rayed again for an indication of cracks before being tested to determine ultimate tensile strength. Either type AA or type M film was used in these examinations.

Penetrant inspection. - Samples of spacecraft 101 and spacecraft 017 tank materials were also penetrant-inspected prior to testing to determine the presence of cracks which could have caused the tanks to fail. The spacecraft 017 samples were completely inspected on both the inner and outer tank wall surfaces. The spacecraft 101 sample was also inspected; however, inspection of the outer tank wall was restricted by the presence of a white paint coating. Zyglo ZL22 spray-type penetrant was applied to each specimen. The specimens were then washed and developed in Zyglo ZP5. The penetration time was 30 minutes and the excess removal time was 5 minutes. Examination of the samples using a 10-magnification eyepiece showed no crack indications. Several porosity indications were noted in the weld but were considered to be so small as to be of no consequence. Numerous scratches were also visible. These were attributed to machining grooves which were only slightly subsurface, and their effects are still being evaluated. Specimens were also penetrant-inspected for indications of cracks after undergoing sustained load testing.

Test fluids. - The test specimens were immersed in air, reagent grade anhydrous methanol, methanol/air, distilled water, water with 500 ppm sodium chromate inhibitor, Aerozine-50, or ethylene glycol/water (87 percent ethylene glycol, 13 percent water).

Test Procedures

Testing of the specimens was performed in the Structures Laboratory at MSC. Primary equipment used included stress-rupture (creep) machines and fatigue-testing machines.

Fatigue tests. - A cyclic stress ranging from 7 to 140 ksi was applied to each specimen at 6 cpm until the specimen failed or until 1500 cycles had been accumulated. Four specimens were tested with each fluid used.

Constant load tests. - A dead-weight loading which produced stresses of 75, 90, 100, 120, 130, or 140 ksi was applied to each specimen. In most cases, specimens were tested at one stress level only. The exceptions to this are noted in table I. In those tests in which specimens had not failed after being subjected to constant loads in different fluids or at different stresses, the specimens were tensile-tested to determine ultimate tensile strength. In general, four or more specimens were tested with each fluid. In addition to the testing of standard specimens, two specimens of the double test-section type were tested in this manner. After one section of the double test-section specimen failed, the other section was X-rayed and penetrant-inspected to determine if cracks existed before the section was tensile-tested.

Ultimate tensile-strength tests. - In addition to previously mentioned specimens obtained from failed tanks and virgin sheet, the specimens which survived the constant load tests were then tested to determine their ultimate tensile strength. The test history at MSC of these tensile specimens is noted in table I.

Fluid analysis. - By the use of arc-emission spectroscopy, reagent grade methanol with no previous exposure to metal was analyzed for the presence of titanium, aluminum, and vanadium. The reagent grade methanol was then exposed to stressed and unstressed specimens and similarly analyzed.

Cathodic protection tests. - Three sets of specimens from spacecraft 101 were used in the cathodic protection tests, each set containing a stressed and an unstressed specimen. The power source was a 6-volt battery and the fluid used was methanol. Three tests were performed. For two of the tests, the stressed specimen was attached to the negative terminal and the unstressed specimen to the positive terminal. When it became apparent that the stressed specimen would not fail after a substantial period of time in this condition, the polarity was reversed and held constant until failure occurred. For the third test, the stressed specimen was attached to the positive terminal and the unstressed specimen to the negative terminal and held in that condition until failure occurred. Specimens were stressed to 120 ksi for the cathodic protection tests.

Microscopic Examinations

Microscopic examinations were conducted to gain an insight into the mode of failure of the specimens exposed to methanol. Metallographic and fractographic examinations were used for this part of the investigation.

Metallographic examination. - A metallographic examination was performed on specimens to investigate the characteristics of the alloy structure and the crack propagation. A Bausch and Lomb metallograph, with Keller's Etch No. 5 (1.0 ml HF, 1.5 ml HCl, 2.5 ml HNO_3, and 95.0 ml H_2O) as the etchant, was used for this examination.

Fractographic examination. - A fractographic examination was performed on specimens to investigate characteristics of the fracture surfaces. An electron microscope (JEM Model 7) was used for this examination.

RESULTS AND DISCUSSION

Data obtained from exposing the specimens to all fluids and types of loading during this investigation are shown in tables I to III. Review of these data indicates a definite reduction in the ultimate tensile strength of the Ti-6Al-4V-STA alloy when exposed to methanol. These data also provide substantial indications that the alloy becomes highly notch-sensitive while simultaneously under stress and in contact with methanol. This was particularly evidenced during the constant load tests on virgin material when comparison was made between the types of fluids and the respective average times to failure. In addition, metallographic examination of the samples indicated that fracture occurred as a result of a stress-corrosion cracking mechanism. While the exact nature of this mechanism has not been determined at this time, additional evaluation and testing have been initiated.

Fluid Analysis

Analysis of reagent-grade test methanol prior to exposure to Ti-6Al-4V alloy showed no trace of titanium, aluminum, or vanadium. Analysis of the same methanol exposed to stressed titanium alloy for an average of 15 minutes showed an amount of titanium and aluminum approximately two orders of magnitude greater than the amount found in the methanol exposed to unstressed titanium for 24 hours. No trace of vanadium was detected.

Fatigue Tests

The specimens which were fatigue-loaded in methanol failed in a relatively low number of cycles when compared to specimens loaded in other test fluids (table II). With this indication that methanol significantly affected the service life of the alloy, contant load tests were accomplished in order to verify this incompatibility.

Constant Load Tests

The constant load tests point out more dramatically the effect of methanol on titanium (table III). Specimens obtained from the failed tanks and subjected to fluids other than methanol survived these tests by periods of time at least two orders of magnitude greater than the times for those specimens being tested in methanol. In fact, no specimens tested in fluids other than methanol failed during the constant load tests. In addition, the presence of physical irregularities, such as scratches on the surfaces of the alloy, was found to cause the stress-corrosion effects of methanol to increase. Unstressed specimens swabbed and/or rinsed with methanol showed no reduction in tensile strength when subsequently tested in air. Specimens obtained from the virgin STA sheet survived the tests for longer periods than the specimens obtained from the failed tanks, when both were tested in methanol. This might be attributed to undetected flaws in the failed tank material, to the previous methanol exposure history of the tank material, to effects of the oxide film on the tank material, to the heat-treat condition of the tank material, or to any combination of the four conditions.

Another finding of these tests was that water in methanol acts as an inhibitor. As little as 1 percent of water in methanol markedly reduced or inhibited the stress-corrosion capability. Several specimens were exposed to methanol without failure for extended periods of time at stresses below 100 ksi. However, the methanol, because of its hygroscopic properties, was progressively diluted with water (absorbed from the atmosphere) to various amounts which influenced the time-to-failure mode. Specimens immersed in methanol, tested later in the program, were wrapped with a plastic sheet to prevent this water absorption.

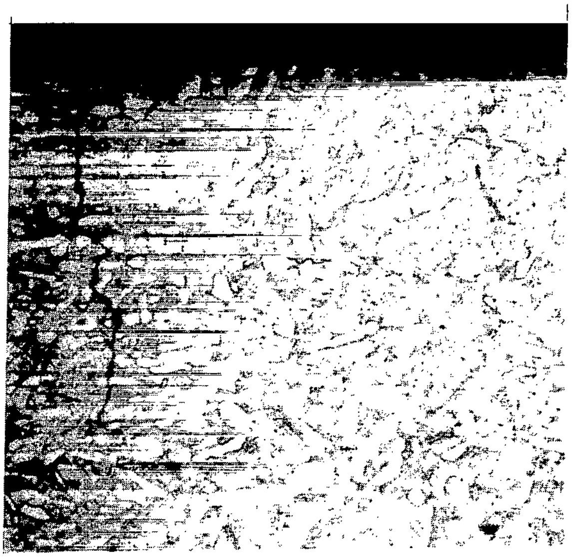
Cathodic Protection Test

During the first cathodic protection test, the stressed specimen was attached to the negative terminal and the unstressed specimen to the positive terminal. After the specimens had remained in this condition for more than 3 hours without failing, the polarity was reversed; the stressed specimen then failed within 2 minutes. The second test setup was the same as the first. After the specimens remained unbroken for 12 hours, the polarity was reversed and the stressed specimen broke within 3-1/2 minutes. For the third test, the stressed specimen was immediately attached to the positive terminal and the unstressed specimen to the negative terminal; the stressed specimen failed within 2-1/2 minutes. These results indicate the electrochemical nature of the methanol attack and suggest a possible means of preventing such attack.

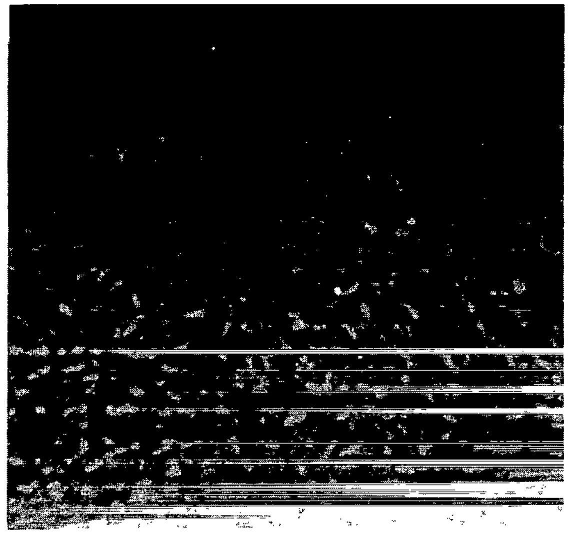
Microscopic Examination

The structure of the failed tank material at, and near, the fracture surface in the tensile specimens showed a normal appearance when examined through the Bausch and Lomb metallograph. However, branch cracking was observed, as shown in figures 6(a) and 6(b). A view normal to a specimen surface, exhibiting a branch crack located near a notched area, is shown in figure 7. This specimen had broken in the notch after 25 minutes in methanol at 100 ksi. A similar crack in an unnotched specimen was also observed (fig. 8). Figure 9 shows a fracture profile with nearby cracks.

The metallographic study determined the existence of cracks having characteristics of stress corrosion or, possibly, hydrogen stress cracking. The results of the cathodic protection test, however, suggested that hydrogen embrittlement was only a remote possiblity, since failure did not occur when hydrogen was collected at the stressed surface of the cathode.



(a) View 1.



(b) View 2.

Figure 6. - Branch cracking of alloy structure (270X).

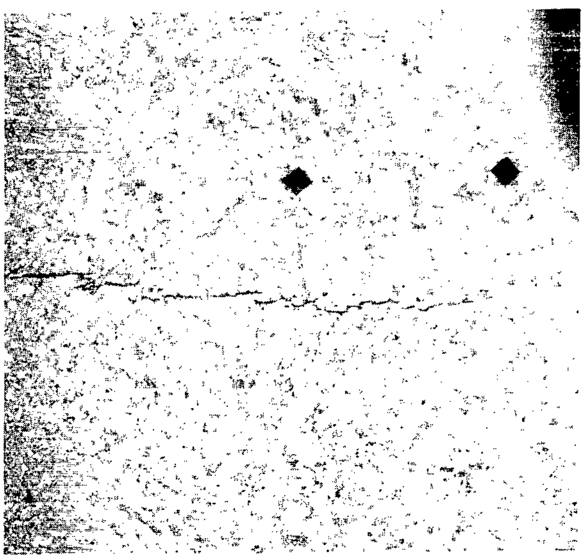


Figure 7. - Branch crack near a notched area (100X).

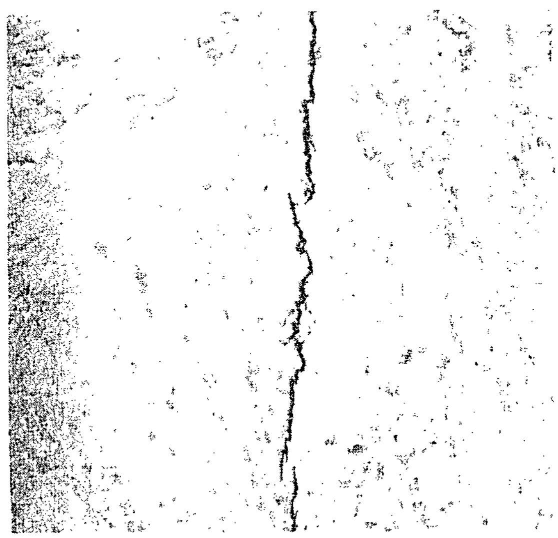


Figure 8. - Branch crack in an unnotched specimen (100X).

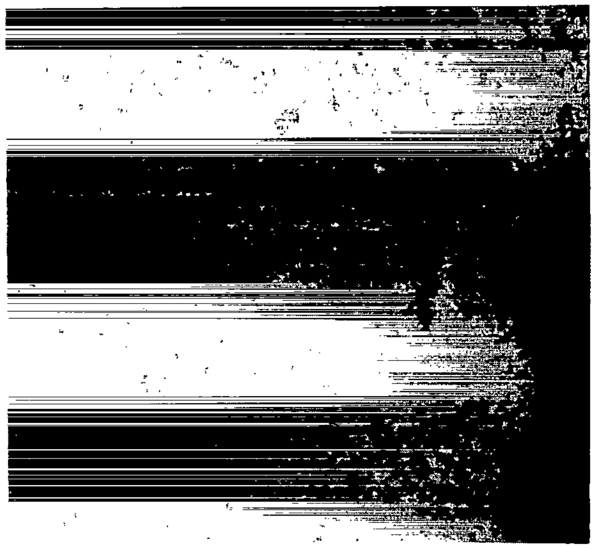
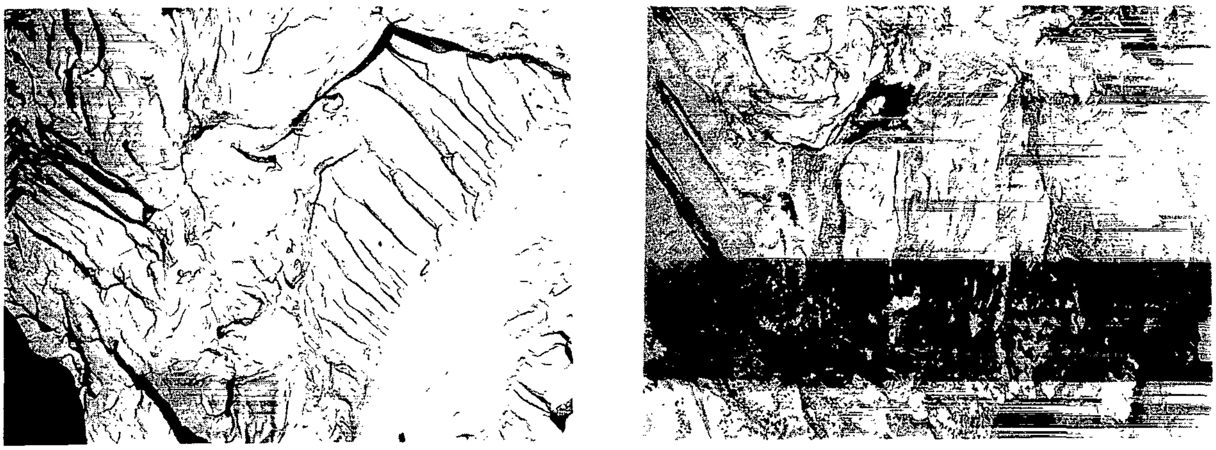


Figure 9. - Fracture profile with nearby cracks (100X).

A fractographic study was made of the fracture surfaces of the tensile specimen to investigate further the mode of failure. Figure 10 shows the brittle appearance in the fracture origin; figure 11, the ductile shear area of the fracture; and figures 12(a) and 12(b), the appearance of the fracture between the brittle zone and ductile shear area. Characteristics observed in figure 12(a) are considered to be representative of stress corrosion.

Figure 10. - Brittle area in fracture origin (2500X).

Figure 11. - Ductile shear area of fracture (2500X).



(a) View 1.

(b) View 2.

Figure 12. - Fracture between brittle zone and ductile shear area (2500X).

CONCLUSIONS

An investigation was performed to determine if the presence of methanol was a factor in the failure of the Apollo spacecraft 017 and 101 pressurized fuel tanks. As a result of this investigation, the following conclusions were drawn.

(1) The tank failures were attributed to an incompatibility between methanol and the stressed fuel-tank material, Ti-6Al-4V-STA alloy.

(2) Evidence of a stress-corrosion cracking mechanism was indicated in the Ti-6Al-4V-STA alloy while simultaneously subjected to stress and methanol.

(3) Small amounts of moisture (1 percent or greater) contained in the methanol inhibited the adverse effect of the fluid on stressed Ti-6Al-4V-STA alloy.

(4) Cathodic protection could be used as a means of inhibiting the stress-corrosion effects of methanol on titanium.

Manned Spacecraft Center
National Aeronautics and Space Administration
Houston, Texas, December 16, 1966
914-50-20-06-72

TABLE I. - TENSILE PROPERTIES

Specimen origin	Number of specimens	Yield strength, ksi	Standard deviation, ksi	Ultimate tensile strength, ksi	Standard deviation, ksi	Test history at MSC
[a]SC 101	2	156	1	172	1	Remnant of SC 101, unexposed
SC 017	1	164	-	176	-	Remnant of SC 017, unexposed
Virgin	2	163	1	172	-	Virgin sheet, unexposed
Virgin	4	-	-	188	3	Notched, 48 hours at 120 ksi in water/sodium chromate solution and 5 minutes at 140 ksi in air
Virgin	3	-	-	171	22	Notched, 48 hours at 120 ksi in ethylene glycol/water solution and 5 minutes at 120 ksi in air
SC 101	1	-	-	143	-	Notched, 42.5 hours at 120 ksi in water and 42.3 hours at 120 ksi in methanol
SC 017	1	165	-	169	-	Double test section specimen remnant, 10 minutes at 120 ksi in methanol and 16.5 hours at 120 ksi in air
Virgin	5	-	-	178	6	Notched, 12 hours at 75 ksi in methanol
Virgin	8	-	-	188	7	Notched, no previous exposure
Virgin	1	-	-	182	-	Notched, 50.7 hours at 140 ksi in isopropyl alcohol
Virgin	2	-	-	178	5	Notched, 30 minutes age at 1000° F
Virgin	4	168	2	171	1	68 hours at 140 ksi in methanol
Virgin	5	-	-	184	7	Notched, 12 hours at 100 ksi in methanol
Virgin	5	-	-	188	2	Notched, 12 hours at 90 ksi in methanol
Virgin	8	-	-	187	6	Notched, 60 hours at 140 ksi in ethylene glycol/water solution

[a]Spacecraft.

TABLE II. - FATIGUE DATA

[All stressed at 7 to 140 ksi,[a] 6 cpm, unnotched specimens]

Specimen origin	Number of specimens	Test fluid	Cycles to failure, cycles	Standard deviation, cycles	Remarks
[b]SC 017	4	Methanol	86	23	Welded area[c]
SC 017	4	Air	1385	[d]100	Welded area, one specimen went to 1500 cycles without failure; two specimens failed in parent material
SC 017	4	Distilled H_2O	1269	333	Welded area, two went to 1500 cycles without failure
SC 101	4	Methanol	91	20	Welded area
SC 101	4	Ethylene glycol/H_2O	715	371	Welded area
SC 101	4	Air	537	142	Welded area
SC 101	5	Distilled H_2O	696	252	Welded area

[a]Stresses on welded samples calculated from thickness in specimen gage length; actual stresses in weld land are approximately 22 percent lower than the stresses indicated.

[b]Spacecraft.

[c]More than 3/4 of the specimens tested failed in the toe of the weld bead.

[d]In cases where one or more specimens survived 1500 cycles, "1500" was used in computing the standard deviation.

TABLE II. - FATIGUE DATA - Concluded

[All stressed at 7 to 140 ksi,[a] 6 cpm, unnotched specimens]

Specimen origin	Number of specimens	Test fluid	Cycles to failure, cycles	Standard deviation, cycles	Remarks
[b]SC 101	4	Freon MF	657	269	Welded area[c]
SC 101	4	Isopropyl alcohol	322	78	Welded area
SC 101	4	Aerozine-50	922	371	Welded area
SC 101	4	Ethylene glycol/H_2O	1098	[d]297	One specimen went to 1500 cycles without failure
SC 101	4	Air	1367	144	Two specimens went to 1500 cycles without failure
SC 101	4	Methanol	169	30	

[a]Stresses on welded samples calculated from thickness in specimen gage length; actual stresses in weld land are approximately 22 percent lower than the stresses indicated.

[b]Spacecraft.

[c]More than 3/4 of the specimens tested failed in the toe of the weld bead.

[d]In cases where one or more specimens survived 1500 cycles, "1500" was used in computing the standard deviation.

TABLE III - CONSTANT LOAD DATA[a]

Specimen origin	Number of specimens	Notched	Load, ksi	Test fluid	Time to failure, min	Standard deviation, min	Remarks
Virgin	1	No	140	Methanol	>300	-	
Virgin	2	No	140	Methanol	63	3	Aged additional 4 hours at 1000° F in air before testing
Virgin	4	Yes	120	Methanol	31	16	
Virgin	5	Yes	120	Ethylene glycol/H_2O	>3183	-	No failures
Virgin	4	Yes	120	H_2O/sodium chromate	>2880	-	No failures
Virgin	2	Yes	120	Methanol	44	28	Aged 30 minutes at 1000° F before testing
Virgin	1	Yes	130	Methanol	20	-	
Virgin	5	Yes	140	Methanol	19	5	
Virgin	8	Yes	140	Ethylene glycol/H_2O	>3588	-	No failures

[a]Several specimens were exposed to methanol without failure for extended periods of time at stresses below 100 ksi. However, the methanol, due to its hygroscopic properties, was diluted with water to various amounts which undoubtedly influenced the time-to-failure. These specimen results were not included in the above table.

TABLE III. - CONSTANT LOAD DATA[a] - Continued

Specimen origin	Number of specimens	Notched	Load, ksi	Test fluid	Time to failure, min	Standard deviation, min	Remarks
Virgin	1	Yes	140	Isopropyl alcohol	>3042	-	No failures
[b]SC 017	5	No	90	Methanol	28	10	
SC 017	4	No	100	Methanol	33	5	
SC 017	4	No	120	Methanol	12	1	
SC 017	1	No	120	H_2O/sodium chromate	>2880	-	No failures
SC 017	5	No	140	Methanol	7	1	
SC 101	5	No	90	Methanol	24	4	
SC 101	5	No	100	Methanol	24	10	
SC 101	10	No	120	Methanol	17	22	

[a]Several specimens were exposed to methanol without failure for extended periods of time at stresses below 100 ksi. However, the methanol, due to its hygroscopic properties, was diluted with water to various amounts which undoubtedly influenced the time-to-failure. These specimen results were not included in the above table.

[b]Spacecraft.

TABLE III. - CONSTANT LOAD DATA[a] - Concluded

Specimen origin	Number of specimens	Notched	Load, ksi	Test fluid	Time to failure, min	Standard deviation, min	Remarks
[b]SC 101	2	No	120	Air	>4463	-	No failures
SC 101	1	No	130	Methanol	2	-	
SC 101	5	No	140	Methanol	6	1	
SC 101	3	Yes	120	Methanol	9	2	
SC 101	3	Yes	120	Aerozine-50	-	-	Specimens loaded for over 2 weeks with no failure at this writing
SC 101	2	Yes	120	Distilled H_2O	>2565	-	No failures

[a]Several specimens were exposed to methanol without failure for extended periods of time at stresses below 100 ksi. However, the methanol, due to its hygroscopic properties, was diluted with water to various amounts which undoubtedly influenced the time-to-failure. These specimen results were not included in the above table.

[b]Spacecraft.

NASA-Langley, 1967 S-148

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