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EFFECT OF CHLORIDE ION CONTENT IN UNSYMMETRICAL
DIMETHYLHYDRAZINE PROPELLANT ON FRACTURE PROPERTIES
OF STRUCTURAL ALLOYS

L. R. Toth, et al

Jet Propulsion Laboratory

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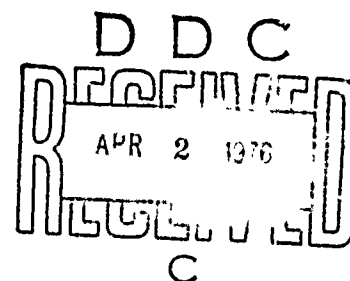
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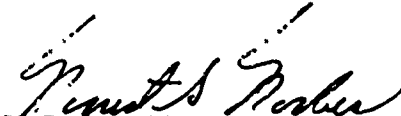
The work reported herein was sponsored by the Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, California under Military Interdepartmental Purchase Request, Project Order Number 0100-75-1. The program was administered under the Technical Direction of 1st Lt. E. C. Bishop (LKDP) and 2nd Lt. R. G. Butts (LKDP).

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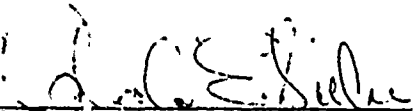
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This report has been reviewed by the Information Office/DOZ and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations. The report has been reviewed and is approved for publication.


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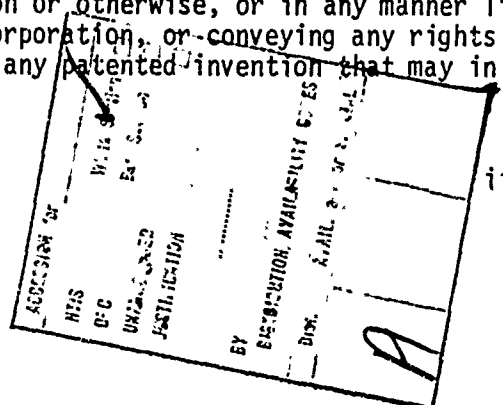

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not affect the sustained load stress corrosion crack growth properties of these alloys in the parent metal form specified for this program.

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I. INTRODUCTION

A. Background Information

This experimental investigation was undertaken to determine a realistic limit of the chloride ion (Cl^-) in unsymmetrical dimethylhydrazine (UDMH) for the purpose of establishing a specification limit (references 1 and 2). The contaminant can be introduced in the propellant during phases of production and/or handling as a salt (e.g. sodium chloride, NaCl) or as an acid (e.g. hydrochloric acid, HCl). Hence, it is also necessary to determine if the form of the chloride ion is a factor.

Three different types of materials of construction were utilized to provide a framework for understanding the interrelationship between the chloride ion (Cl^-) effect and stress corrosion crack growth properties. The alloys are:

Aluminum (Al), 2014-T6

Corrosion Resistant Steel (CRES), type 304L

Titanium (Ti), 6Al-4V.

These alloys are representative of weight sensitive, highly stressed structural materials used for critical feed system components in spacecraft or similar liquid chemical propulsion systems of operational Air Force vehicles.

The experimental testing involved the use of either a thin or thick gage tensile type specimen with a single, centrally located, semi-elliptical surface flaw, Figures 1 and 2. These specimens were subjected to sustained load testing with the flaws exposed to different variations of UDMH propellant listed in Table 1. Each specimen cross-section was examined after test using the Scanning Electron Microscope (SEM) method to evaluate the effects of testing.

B. Program Objective

The objective of this experimental program was to determine the form and limit of the chloride ion (Cl^-) content in UDMH propellant which can be tolerated by typical propulsion system materials.

C. Scope of Program

The scope of work was based upon utilization of standard stress corrosion crack test procedures to provide the necessary data. The test program for

propellant and material combinations was comprehensive, and included the following major categories:

- (1) Propellant assays
- (2) Material property determinations
- (3) Specimen pretest processing
- (4) Conducting twenty-four (24) and one thousand (1000) hour duration tests
- (5) Performing post test evaluations

D. Method of Qualification

The emergence of the fracture mechanics approach as a viable diagnostic method for generating reliable information is discussed in Appendix A. JPL has applied this procedure successfully to develop and qualify flight hardware for Mariner-class spacecraft for unmanned planetary missions. These previously developed analytical and operational procedures, test equipment, and facilities were used without modification in accomplishing the work for the AFRPL program.

The following sections present pertinent details of this work.

II. PROPELLANT AND MATERIALS

A. Propellant Procurement

The propellants shown in Table 1 were furnished by AFRPL.

B. Propellant Characterization

Pure unsymmetrical dimethylhydrazine (UDMH) is a colorless, hygroscopic liquid which freezes at -58°C and boils at 64°C . Its density at 25°C is 0.782 g/cm^3 .

The UDMH used for the baseline data in these tests was prepared from material of MIL-P-25604D quality which was distilled in vacuo. The specifications for MIL-P-25604D quality UDMH and the analysis of the distilled UDMH are shown in Table 2.

Chloride addition was accomplished in two ways. Chloride ion was introduced to the UDMH in a neutral salt, i.e., anhydrous lithium chloride. Originally it was planned to use sodium chloride, but the lithium salt is more

soluble in UDMH. Two levels of chloride ion concentration were tested: 36 ppm and 105 ppm. Analyses are shown in Table 2.

To determine if the form of the ion is a factor, an acid chloride--UDMH hydrochloride--was used as the source of chloride ion. Anhydrous UDMH hydrochloride, usually recrystallized from absolute ethanol, can be more conveniently and accurately measured than can gaseous HCl. Two levels of chloride ion concentration were prepared: 30 ppm and 108 ppm. These analyses are listed in Table 2.

C. Material Procurement

The materials shown in Table 1 were obtained from existing stock and used in the "as received" condition. Certifications were provided indicating compliance with appropriate specification requirements. The sources for the subject materials are listed below:

- (1) Titanium 6Al-4V, Sheet (MIL-T-9046) supplied by AFRPL in the heat treated condition.
- (2) Titanium, 6Al-4V, Forging (MIL-T-9047) supplied by JPL in the heat treated condition.
- (3) Aluminum, 2014-T6, Plate (QQ-A-250/3) supplied by NASA-Marshall Space Flight Center (MSFC) in the heat treated condition.
- (4) Corrosion Resistant Steel, type 304L, Plate (MIL-S-4043) supplied by NASA-MSFC in the annealed condition.

D. Material Characterization

1. Composition and Mechanical Properties

All test samples for this program were fabricated from the above stock. Specific units were subjected to chemical composition analyses and mechanical properties tests. These results, indicated in Tables 3 and 4, meet the applicable specification requirements without exception (see references 4 and 5). In three cases, the material used for the composition analysis was sectioned from a specimen which had already been subjected to a 1000 hour duration test (see Table 3, note b). This verified that the proper material was utilized in those critical tests.

2. Microstructure Examinations

Microstructure examinations were performed on cross-sections taken from each of the four types of materials. These cross-sections were approximately 1.27 cm from the surface flaw of specimens 18T, 68T, 48A, and 58S. The microstructure results are shown in Figures 3, 4, 5 and 6, and these microstructures are typical for the subject materials.

III. TEST PROGRAM

A. Discussion

1. Approach

Fracture mechanics test procedures were chosen to determine any detrimental effects of chloride in UDMH because such effects are magnified by the presence of a sharp crack. Also, a fracture mechanics approach permitted a quantitative determination of the maximum design stress for service in UDMH.

The part-through cracked (PTC) or surface cracked specimen loaded in uniaxial tension was used because this specimen represents real cracks in real structure. In addition, the PTC specimen is the only fracture mechanics specimen that can be used to test the thin materials specified for this program.

Three (3) specimens of the same alloy were tested simultaneously in tandem while exposed to the specific propellant (Figures 1 and 13 and Table 1). Different initially applied stress intensity levels, K_{Ii} , were obtained in each of the three specimens by varying the width of the specimens while the crack size was held constant. Basic configurations are illustrated in Figures 7, 8, 9, and 10.

Scanning electron microscopy (SEM) was used to examine the crack surfaces for evidence of crack growth caused by the UDMH. Because of the depth of field and the magnifications obtainable by SEM, growth which occurs during application of the load can be distinguished from growth caused by the UDMH.

The critical stress intensity for a steadily increasing load in an inert environment, K_Q , was measured for each specimen except for the corrosion resistant steel specimens. This parameter was measured because a plot of K_{Ii}/K_Q reduces the scatter in the data as compared to plotting K_{Ii} alone. Consequently, the tests were targeted to be loaded at various percentages of K_Q .

based on previous work with other propellants such as nitrogen tetroxide and hydrazine.

Annealed corrosion resistant steel is too crack tolerant to permit measurement of K_Q in these thin gages. When a steadily increasing load is applied to pre-cracked CRES specimens, the entire spectrum yields without crack growth occurring. For these specimens, an initial crack depth of approximately 0.076 cm (0.030 inch) was selected for the sustained load tests. This crack size was chosen because any cracks larger than this would be detectable in these gages by nondestructive inspection methods.

2. Test Matrix

The overall test plan is indicated in Table 1. The test matrix covering the program implementation is indicated in Table 5. The procedure followed was the same for all materials. Twenty-four (24) hour tests were conducted initially and results evaluated. Based upon these results, the most critical effect was determined. Using these conditions the one thousand (1000) hour tests were conducted.

B. Specimen Preparation

1. Pretest

The specimen configurations used are defined in Figures 7, 8, 9 and 10. These specimens were designed to comply with the guidelines of ASTM Committee E-24 for the surface-cracked specimen, References 6, 7, and 13, except for the thickness. None of the specified materials were thick enough to provide complete plane strain conditions at the tip of the crack. Therefore, thick titanium specimens were prepared from forging material so that the full plane strain condition could be tested (see Figure 8).

Test samples were machined to the configurations shown and a notch (see detail A of Figures 7, 8, 9 and 10) was put in the center of each specimen using electrical discharge machining (EDM). From this notch, an initial crack was started by cyclically stressing the notch in bending. This initial fatigue zone is clearly distinguishable by SEM. The design of the notch and the initial crack and the techniques for forming the initial crack were all in accordance with the guidelines of References 6, 7, and 13.

The specimens were degreased with isopropyl alcohol (IPA) after notching and before pre-cracking. Pre-cracking was done in air. No contaminant was allowed to get into the crack. Before testing, the completed initial crack was cleaned and dried to the requirements of the JPL cleaning specification for monopropellant hydrazine propulsion systems, Reference 8. It is noted that only IPA was used as a cleaning agent. Specimens were never exposed to any halogenated cleaning solvent (for example, Freon type) during processing, handling, or testing.

2. Post Test

Immediately after the specimens were removed from the test machine, the surfaces and cracks were decontaminated of UDMH using the procedures described in Appendix C. The cracks were then cyclically loaded by bending in air again to produce a post test "fatigue mark". Any crack growth which occurred during the test can be seen between the initial fatigue pre-crack and the fatigue mark because the two fatigue zones have a unique fractographic appearance when viewed in the SEM.

After marking, the titanium specimens were fractured in liquid nitrogen. K_Q at the liquid nitrogen temperature of -196°C (-320°F) was then calculated from the final "marked" crack size and the failure load. Liquid nitrogen was used to measure K_Q because the yield stress for titanium is much higher at -196°C (-320°F) than at ambient temperature. Therefore fracture occurred below the yield stress. K_Q at -196°C is about 80% of K_Q at 21°C (70°F) for this alloy. This technique was required because the thickness of the titanium specimen limited the depth of flaw that could be tested.

The aluminum specimens were fractured at ambient temperature for measurement of K_Q because the yield strength of aluminum alloys does not increase significantly at cryogenic temperatures. Also the aluminum specimens were twice as thick as the titanium specimens and deeper flaws were used.

The corrosion resistant steel specimens were merely broken apart at ambient temperature after marking so that the fracture surface could be analyzed. All the CRES steel specimens necked down in general yielding before fracture occurred.

C. Test Setup

The approach described below was used for testing the specimens during the program.

1. Fixtures

Each sustained load test specimen was tested with a fixture arrangement shown in Figure 11. UDMH propellant pressurized to 207.0 N/cm^2 (300 psig) was applied at all times to both sides of the test item to produce uniform load conditions. The seals were ultra-pure type Teflon, TFE, to eliminate the possibility of introducing any contamination. This TFE material was procured from the Fluorocarbon Corp, and is identified as FC-95.

A typical test specimen-fixture assembly arrangement is shown in Figure 12. The propellant cups (made of CRES type 321) are clamped onto each specimen using two 0.95 cm (0.375 inch) diameter high strength steel bolts with Belleville springs to maintain a constant preload. Corrosion resistant steel lines 0.32 cm (0.125 inch) diameter were used to interconnect each propellant container and the single source of UDMH.

2. Test Equipment and Facilities

The complete test setup with three specimens mounted in series is shown in Figures 13 and 14. The sustained load test specimens were loaded in either a 45.0 kN (10,000 lb) or 90.0 kN (20,000 lb) calibrated dead load creep machine. The room temperature was controlled at 49°C (120°F). All sustained load tests were conducted at the JPL-ETS facility.

The specimen fatigue marking and tensile fracture tests were performed in the Materials and Processes Laboratory located at JPL-Pasadena. Baldwin universal test machines were used for this phase.

IV. RESULTS

A. Summary

The results of all the tests are summarized in Tables 6-8. The complete data are reported in Appendix B.

None of the samples of UDMH caused any crack growth in any of the three alloys tested. For the titanium alloy, samples containing 100 ppm of chloride were loaded as high as 105 percent of K_Q at -196°C (-320°F) for 1000 hours

with no resultant crack growth (see 69-T of Table 6). The aluminum alloy was tested in UDMH of 108 ppm of chloride at 90 percent of K_Q at 21°C (70°F) for 24 hours and 69 percent of K_Q at 21°C (70°F) for 1000 hours with no crack growth occurring (see 35-A and 48-A of Table 7). The corrosion resistant steel alloy was loaded to its yield stress in UDMH of 104 ppm of chloride for 1000 hours with no growth occurring in a crack of 0.078 cm (0.031 inch) depth (see 58-S of Table 8). These results indicate that UDMH of 100 ppm of chloride content is essentially inert to the materials tested.

B. Dimpling

Upon application of the load during the sustained load tests, readily visible back-face dimpling occurred in most of the higher stressed titanium and aluminum specimens. Back-face dimpling is seen in thin materials when the plastic zone at the tip of the crack intersects the back-face free surface. The remaining elastic ligament is too thin to provide elastic constraint around the plastic zone. As a result, this thin ligament is deformed inward without rupturing to produce a depression in the back side of the specimen of about 0.33 cm (0.125 inch) diameter (see Figure 15). The depth of this dimple can be as much as 0.076 cm (0.030) inch or more. Such dimpling is currently being used as an inspection criterion at JPL for titanium tanks containing hydrazine propellant.

C. Scanning Electron Microscopy

Scanning electron micrographs (SEM) of some of the more highly loaded specimens of the three alloys are shown in Figures 16-22. None of the SEM fractographs show any crack growth between the initial fatigue precrack and the final fatigue mark. The borders between the initial fatigue zone and the final fatigue zone are visible and marked in each of the figures.

D. Comparison of UDMH with Other Environments

Figure 23 plots the data for UDMH and the titanium alloy from this program as K_{Ii}/K_Q versus exposure time which is the most commonly used graphical presentation method. Only the highest stress intensity ratio tested for each of the environment-material combinations is plotted because none of the specimens tested showed any growth. The threshold values are

ratios from no-growth ratios for the UDMH samples cannot be drawn because no growth was produced in any of the specimens.

As a means of comparison, data from environments which have produced growth (for example, reference 15) in the same titanium alloy in the same test equipment are superimposed on the graph and the threshold curves for two of these environment-material combinations are shown. Refined hydrazine is essentially inert to aged titanium forgings (upper curve) yet causes extensive growth in welds made from the same forgings but tested in the "as-welded" or unaged condition (lower curve of Figure 23). Also, isopropyl alcohol per Federal Specification TT-I-735 causes considerable growth in the aged forgings at a stress intensity ratio of approximately 0.5 (see Figure 23). In Figures 24 and 25 the growth which occurred in the sustained load exposure test is readily seen between the two fatigue zones.

V. CONCLUSIONS

The effect of different forms and levels of the chloride ion (Cl^-) content in unsymmetrical dimethylhydrazine (UDMH) on typical propulsion system materials has been investigated (see Table 5). Based upon these experimental results, the two conclusions are:

A. An allowable chloride ion (Cl^-) content in UDMH of thirty (30) parts per million (ppm) or less from either a neutral or acidic salt should be compatible with typical propulsion feed system materials of construction. Selection of this chloride ion level is consistent with the fact that the chloride ion levels of the UDMH samples tested did not affect the sustained load stress corrosion crack growth properties of these three typical propulsion system alloys. To add emphasis to the above conclusion, the titanium alloy tested is known to be so susceptible to cracking by free chloride ions that one (1) ppm is usually the maximum allowable free chloride ion level for any fluid contacting titanium. Yet one hundred (100) ppm of Cl^- in UDMH had no adverse effect whatever on the sustained load crack growth properties of the titanium alloy.

B. Therefore, based upon the above, a corollary conclusion may be drawn. This conclusion is that other alloys in existing systems should not be affected within the margin of safety provided by a thirty (30) ppm Cl^- limit.

VI. RECOMMENDATIONS

A limit of thirty (30) ppm chloride ion (Cl^-) content in specification grade UDMH propellant is recommended. However, before any new systems using UDMH are designed to minimum weight, fracture mechanics design data should be obtained in UDMH of thirty (30) ppm Cl^- content for each alloy, material form, and heat treat level planned for use in the new system. Both sustained load data and cyclic load data in the specific UDMH to be used should be obtained for the base metal, welds and weld heat-affected zones of each alloy used. If alloy thicknesses are used which are too thin to achieve a plane strain stress field at the tip of a crack, then each different thin gage thickness should be tested also.

REFERENCES

1. Specification MIL-P-25604 D, "Propellant, Uns-Dimethylhydrazine," May 1969.
2. Advance Procurement Supplemental Data Sheet, "Propellant, Uns-Dimethylhydrazine" Organization Code DL/AFRPL, dated 9 January 1975, applicable to MIL-P-25604 Revision D.
3. NASA Space Vehicle Design Criteria, SP-8040, "Fracture Control of Metallic Pressure Vessels," May 1970.
4. Military Standardization Handbook, MIL-HDBK-5B, "Metallic Materials and Elements for Aerospace Vehicle Structures," September 1971.
5. Aerospace Structural Metals Handbook, AFML-TR-68-115.
6. Tiffany, C. F. and Masters, J. N., "Applied Fracture Mechanics," Fracture Toughness Testing and Its Applications, ASTM Special Technical Publication No. 381, 1965, pp. 190, 249-277.
7. Anon; Damage Tolerant Design Handbook, HB-01, Sponsored by the AFML, December 1972.
8. JPL Specification (internal document) S504574, Revision C, "Cleaning Requirements for Spacecraft Propulsion Systems and Support Equipment," May 1974.
9. Masters, J. N., "Cyclic and Sustained Load Flaw Growth Characteristics of 6Al-4V Titanium," NASA CR 92231, 1968.
10. Inglis, C. E., "Stresses in a Plate Due to the Presence of Cracks and Sharp Corners," "Proceedings of the Institute for Naval Architects," Volume LV, 1913, pp. 219-230.
11. Griffith, A. A., "The Phenomenon of Rupture and Flow in Solids," "Philosophical Transactions of the Royal Society," London, Volume 221, 1921, pp. 163-198 (Paper Presented in 1920).
12. Irwin, G. R., "Crack-Extension Force for a Part-Through Crack in a Plate," Transactions of ASME, Journal of Applied Mechanics, Series E., Volume 29, No. 4, December 1962, pp. 651-654.
13. Orange, T. "Fracture Testing with Surface Cracked Specimens," Journal of Testing and Evaluation, Vol 3, No. 5, September 1975, pp 335-342.
14. Masters, J. N. et al; "Fracture Characteristics of Structural Aerospace Alloys Containing Deep Surface Flaws; NASA CR 134587, December 1973.
15. Bolstad, D. A. "Viking 75 Material Qualification Test Report of Fracture Mechanics Evaluation for the Viking Lander Pressure Vessels," Martin Marietta Corp (Denver Division) Report TR 3720358. February 1977

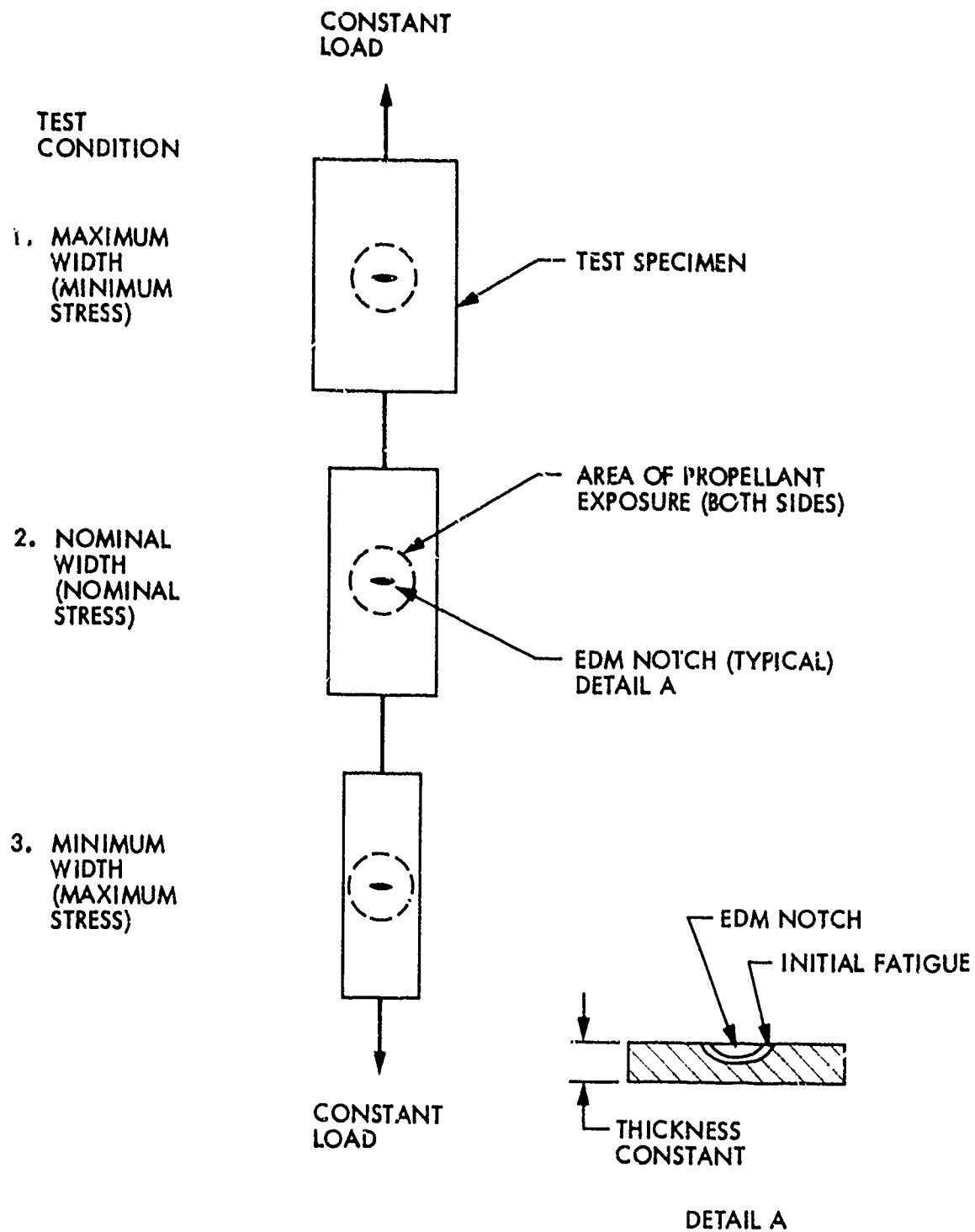


Figure 1. Typical Test Arrangement with Specimens in Series

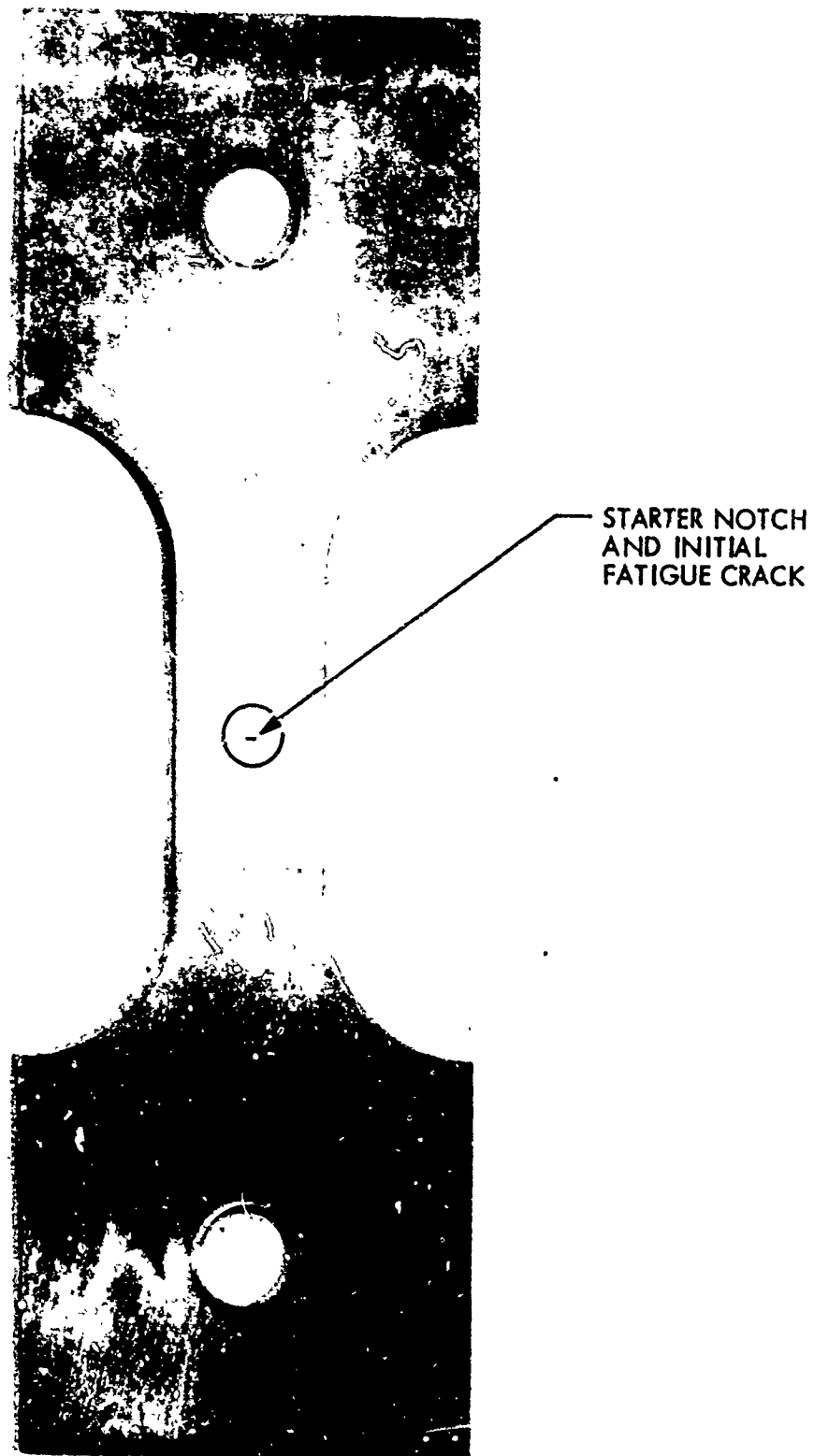


Figure 2. Typical Starter (EDM) Notch with Initial Fatigue Crack
(Titanium Specimen)



Figure 3. Microstructure (500X) - Titanium Sheet-Specimen 18T After 1000 Hour Test

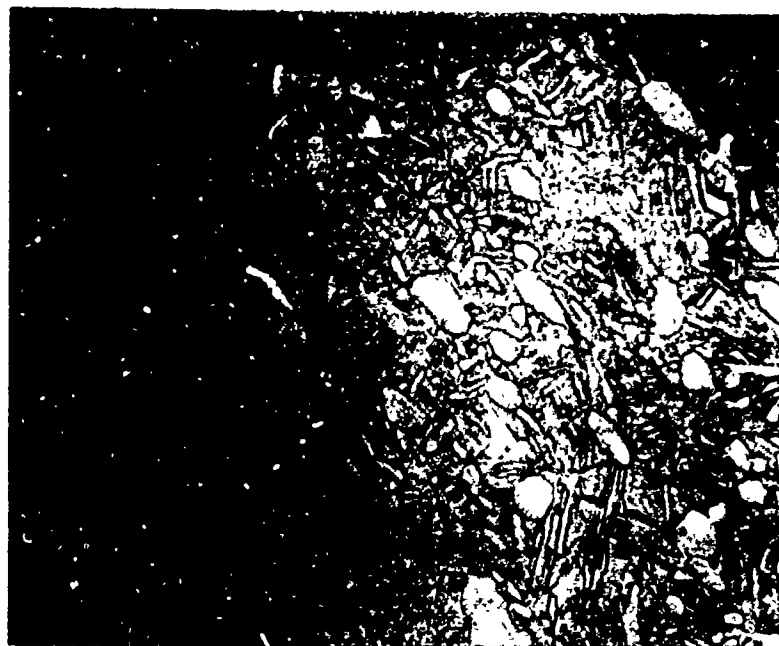


Figure 4. Microstructure (500X) - Titanium Forging-Specimen 68T After 1000 Hour Test



Figure 5. Microstructure (200X) Aluminum
Plate-Specimen 48A After 1000 Hour Test

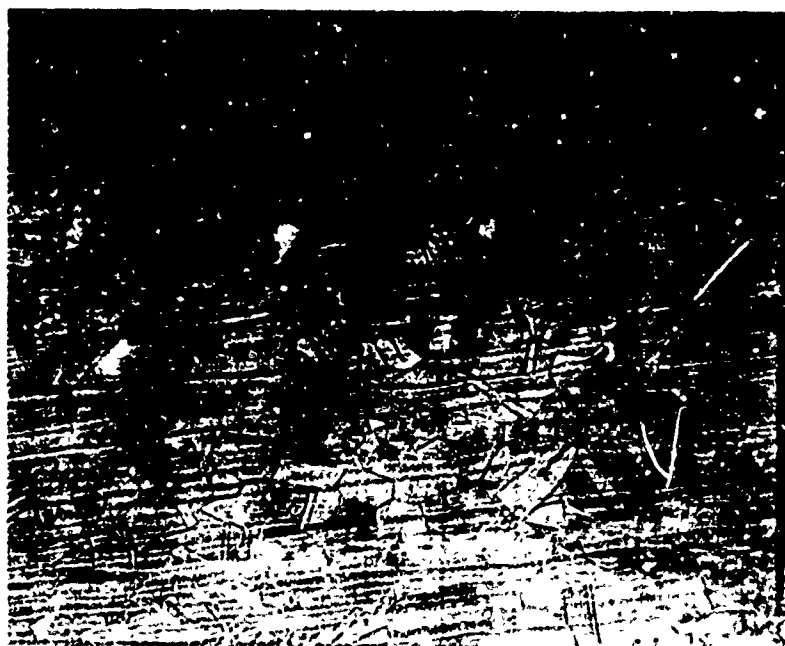
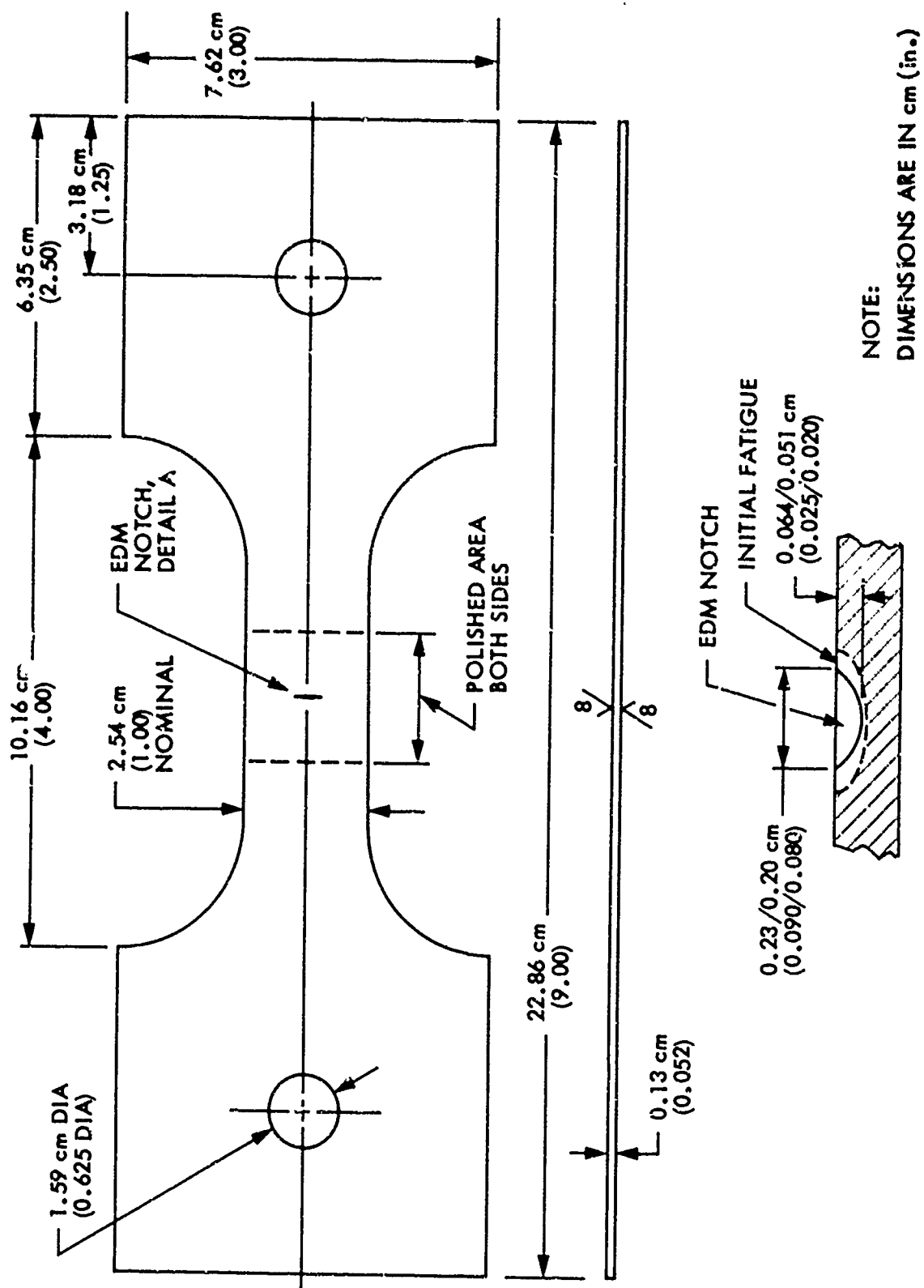


Figure 6. Microstructure (100X) Corrosion Resistant
Steel-Specimen 58S After 1000 Hour Test



DETAIL A

Figure 7. Titanium Sheet Basic Test Specimen Configuration

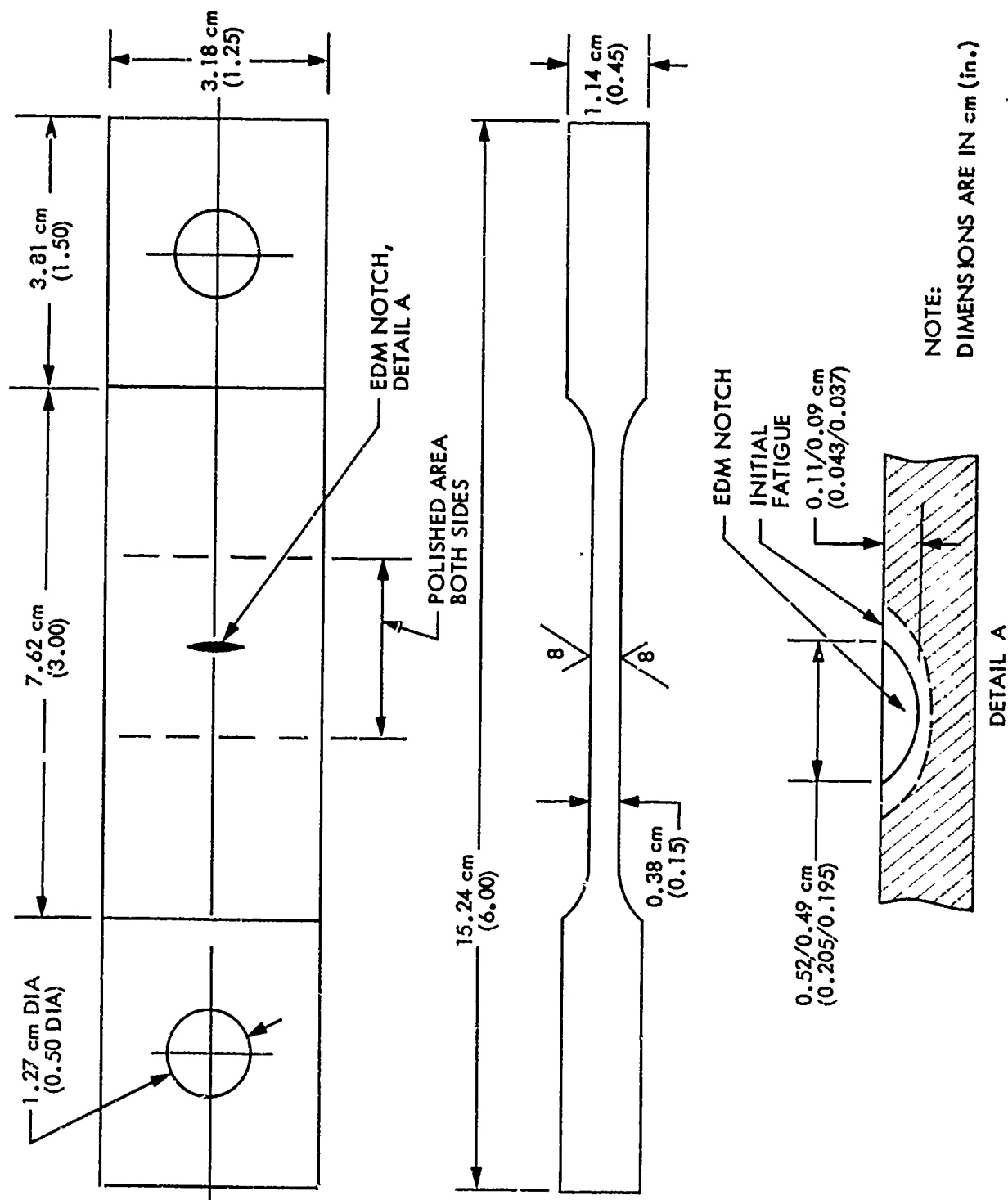


Figure - 8. Titanium Forging-Basic Test Specimen Configuration

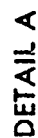


Figure 9. Aluminum Plate Basic Test Specimen Configuration

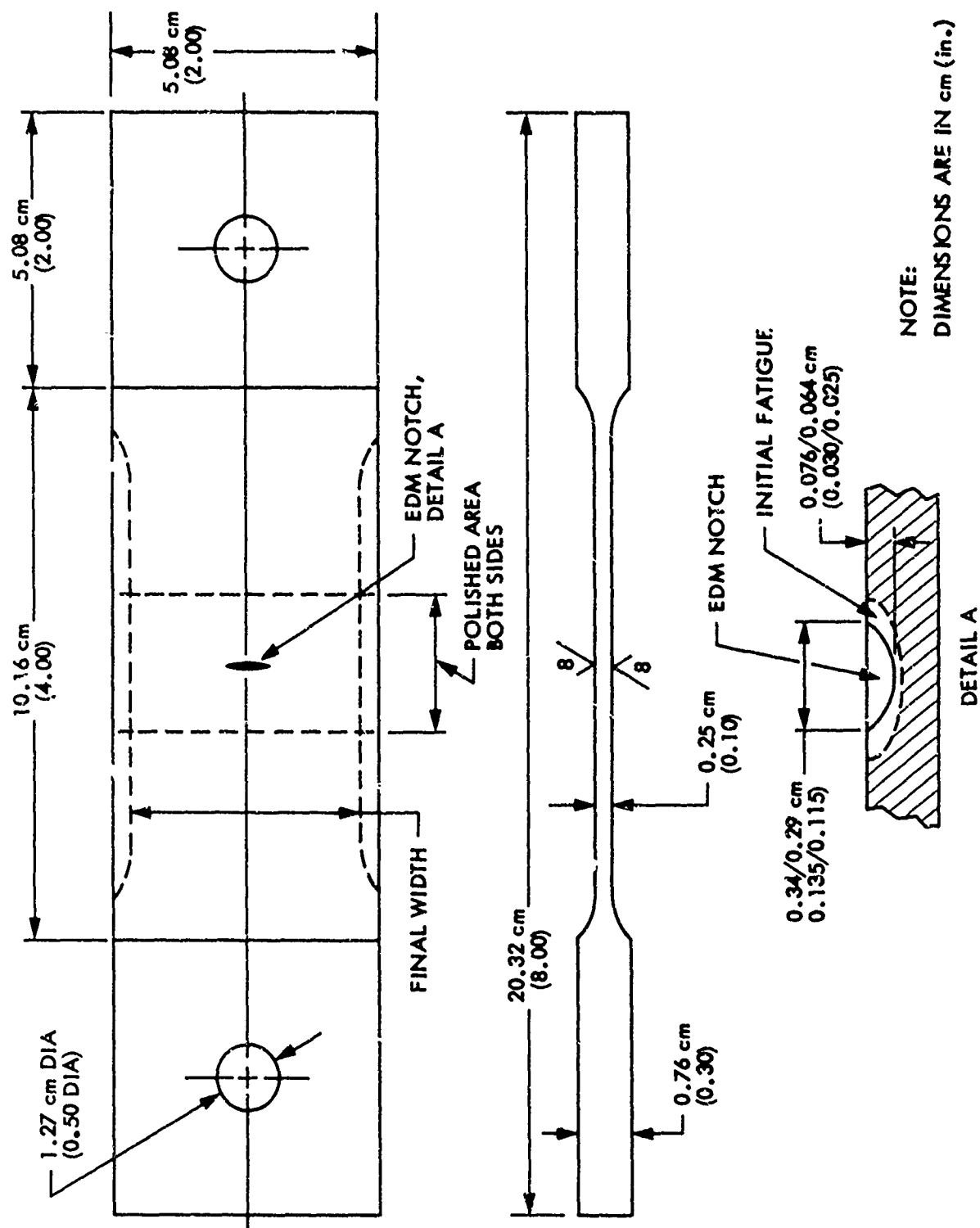
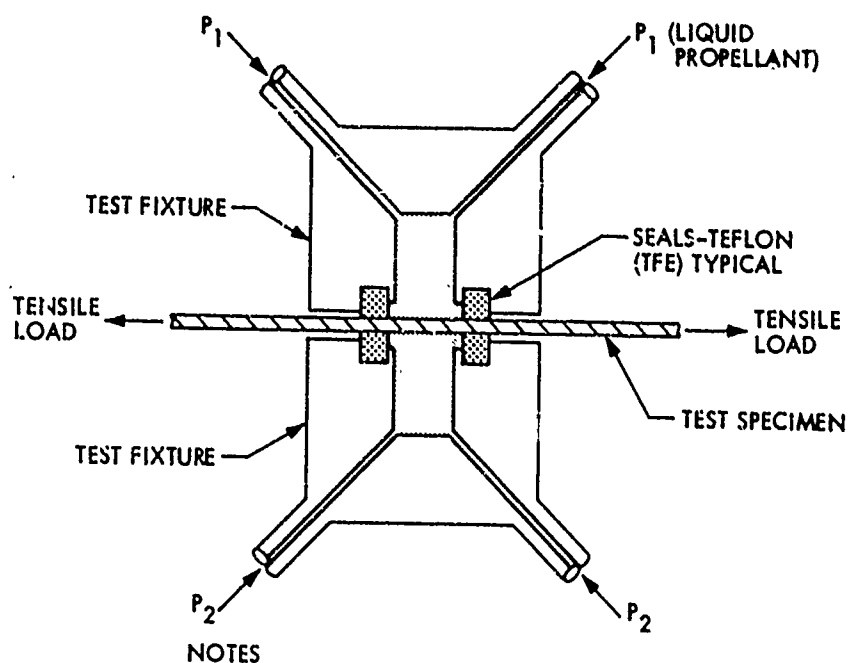


Figure 10. Corrosion Resistant Steel Plate Basic Test Specimen Configuration



NOTES

1. $P_1 = P_2$ PRESSURIZED PROPELLANT
2. TEST FIXTURES - SPRING LOADED

Figure 11. Test Specimen-Fixture Arrangement



Figure 12. Test Specimen-Fixture Assembly (Titanium Specimen)

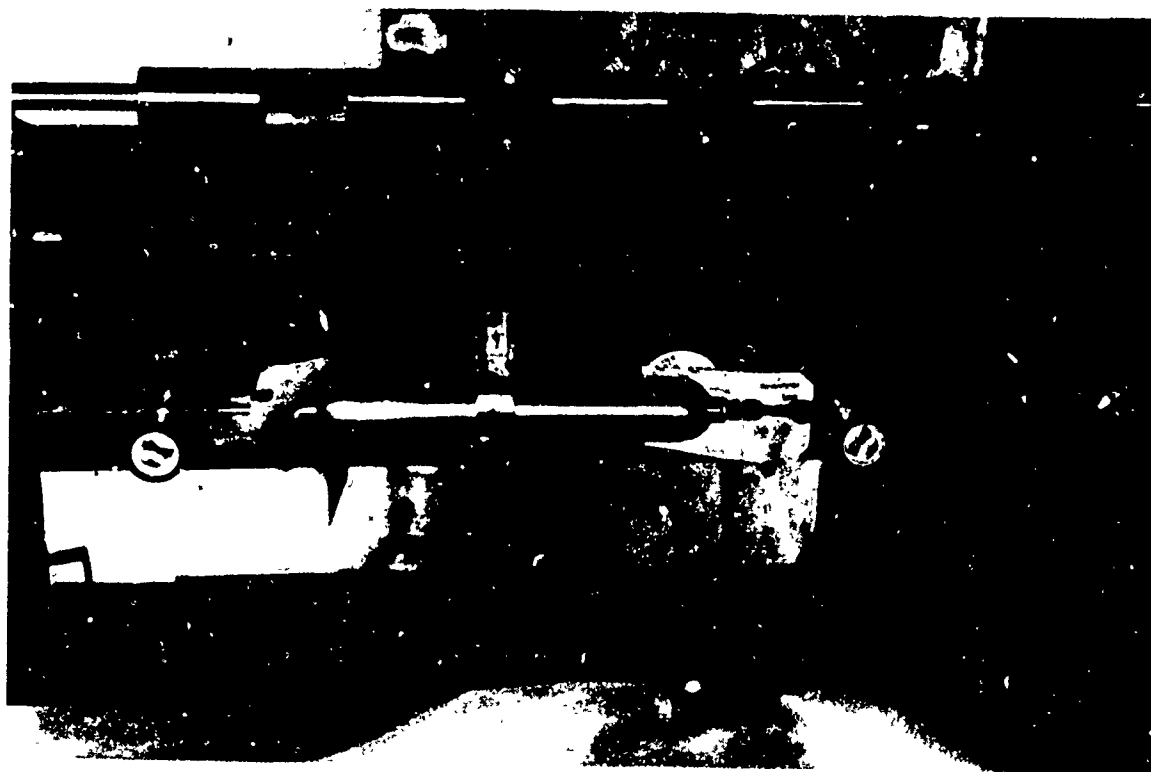


Figure 13. Test Setup-Three Specimens
and UDMH Liquid Propellant

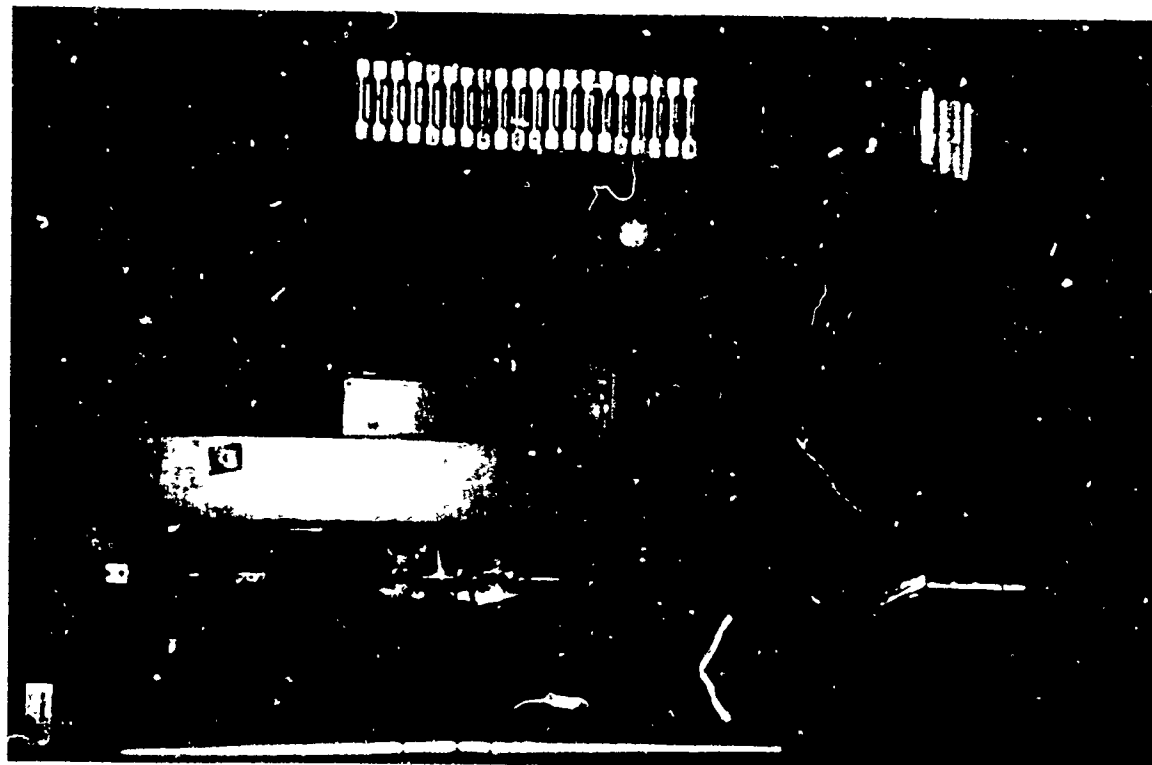


Figure 14. Actual Sustained Load Test-Specimens
Exposed to UDMH Liquid Propellant

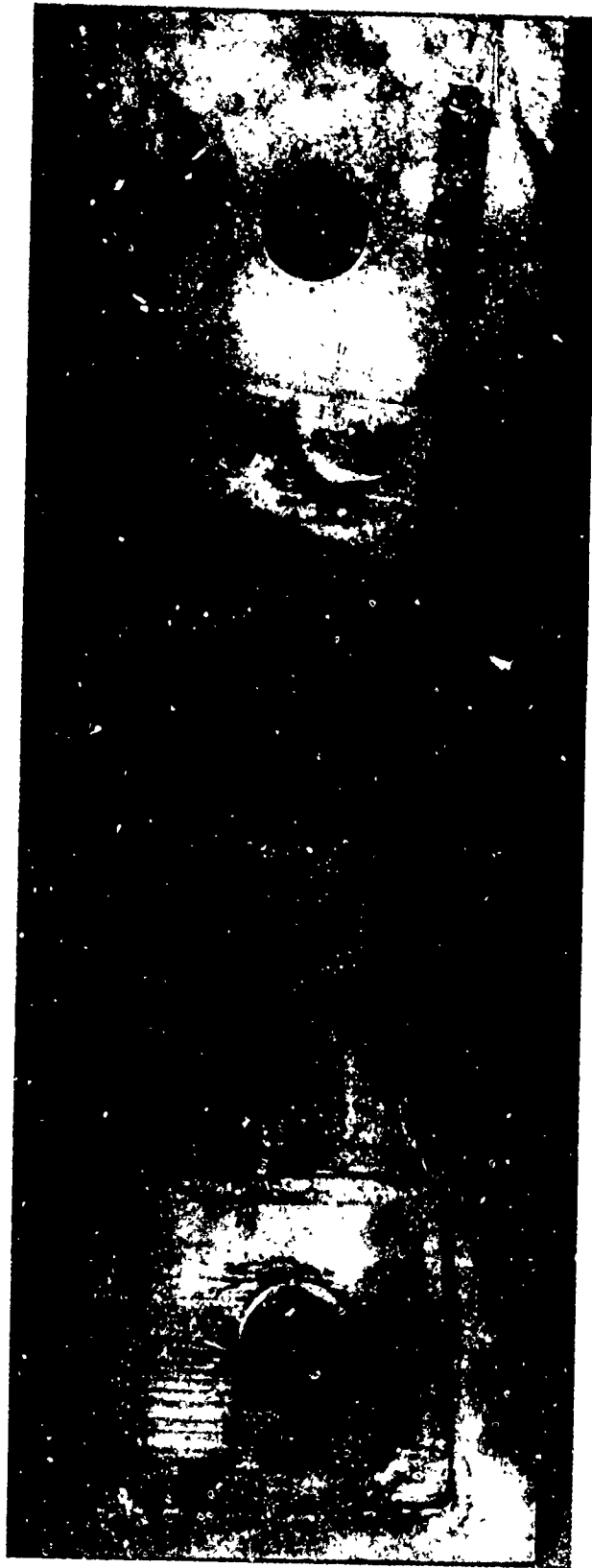


Figure 15. Typical Post-Test Dimple-Titanium Specimen 23T

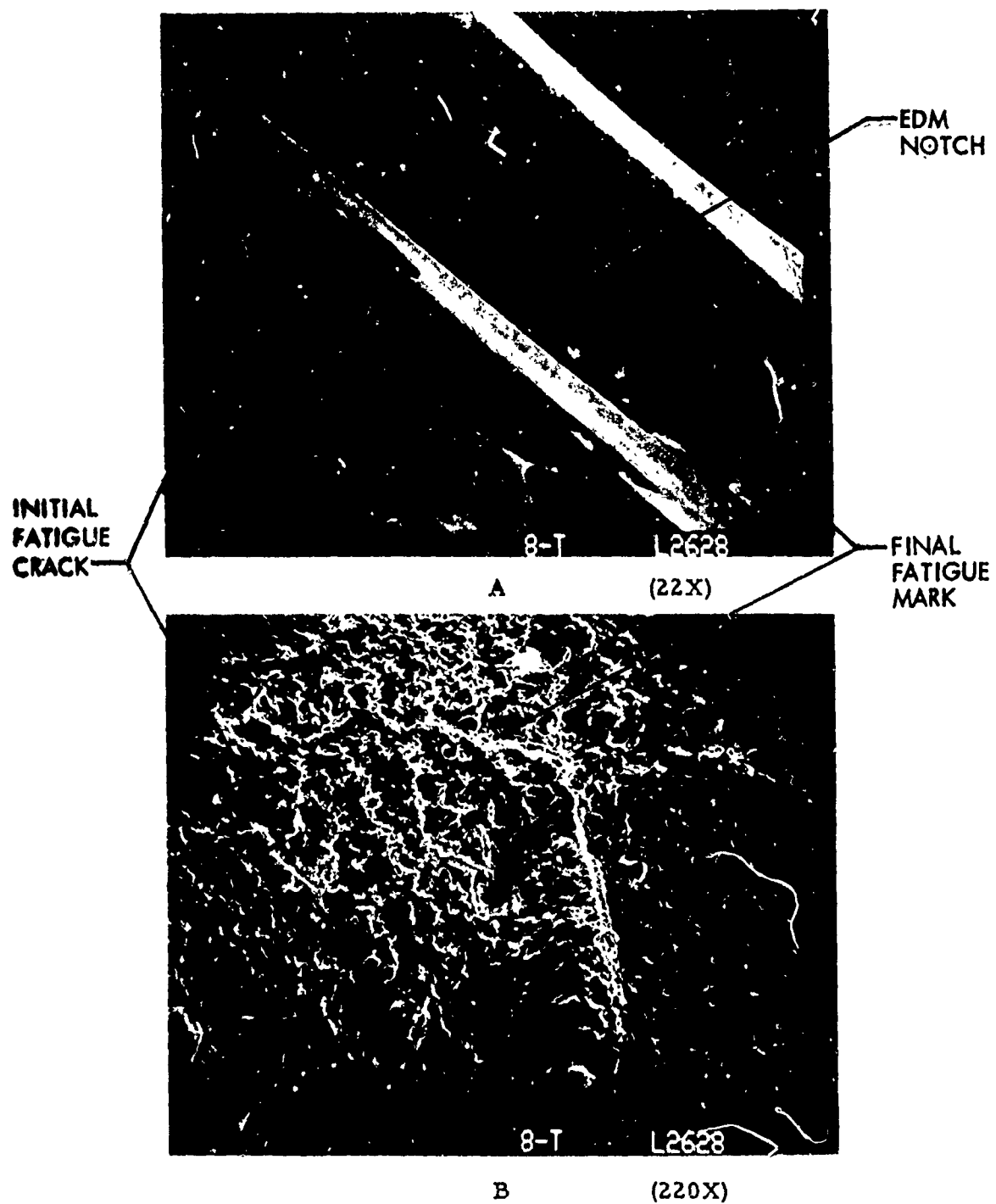


Figure 16. Scanning Electron Micrograph Titanium Specimen 8T After Exposure to Distilled UDMH

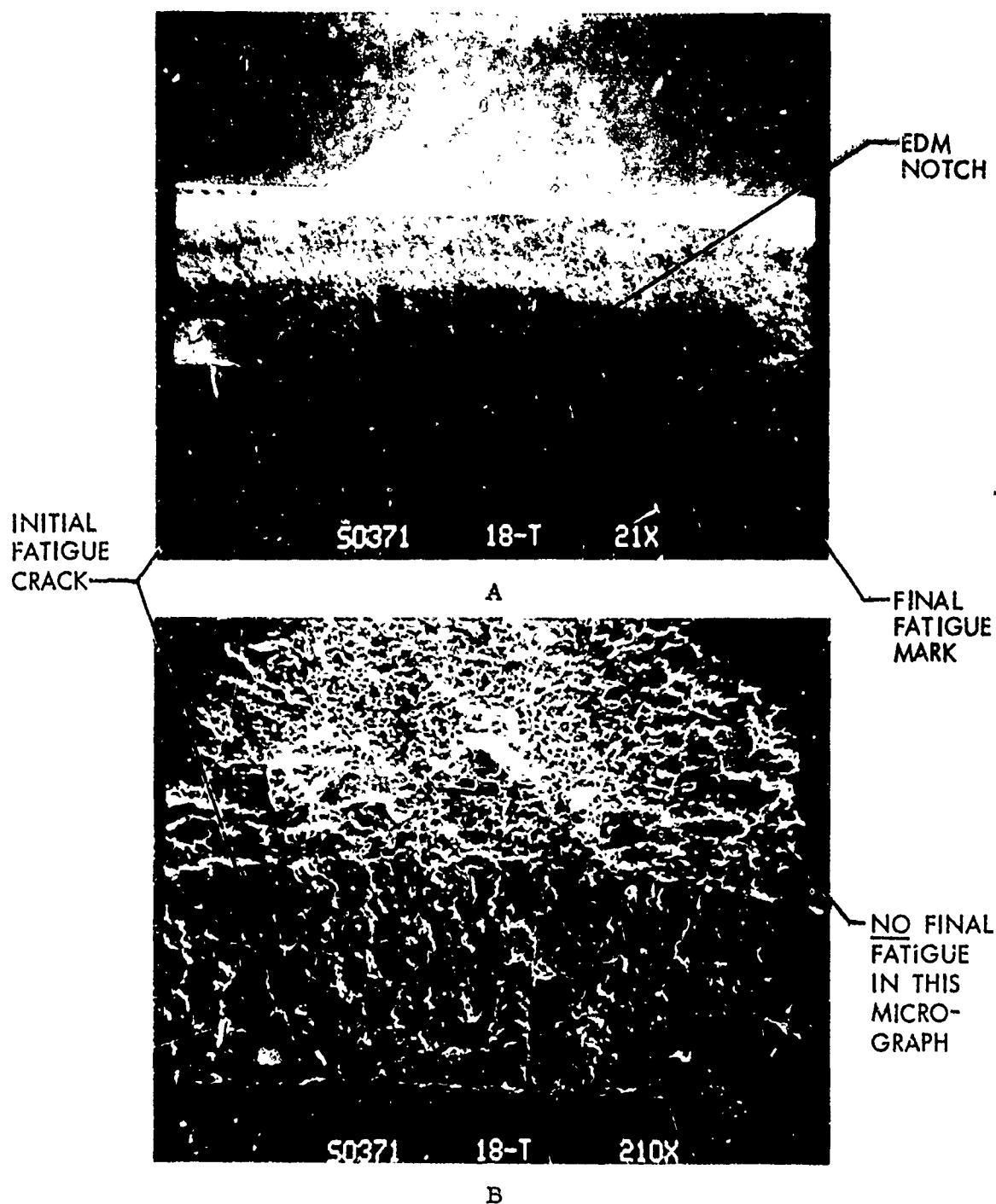


Figure 17. Scanning Electron Micrograph Titanium Specimen 18T After 1000 Hour Exposure to UDMH:HCl

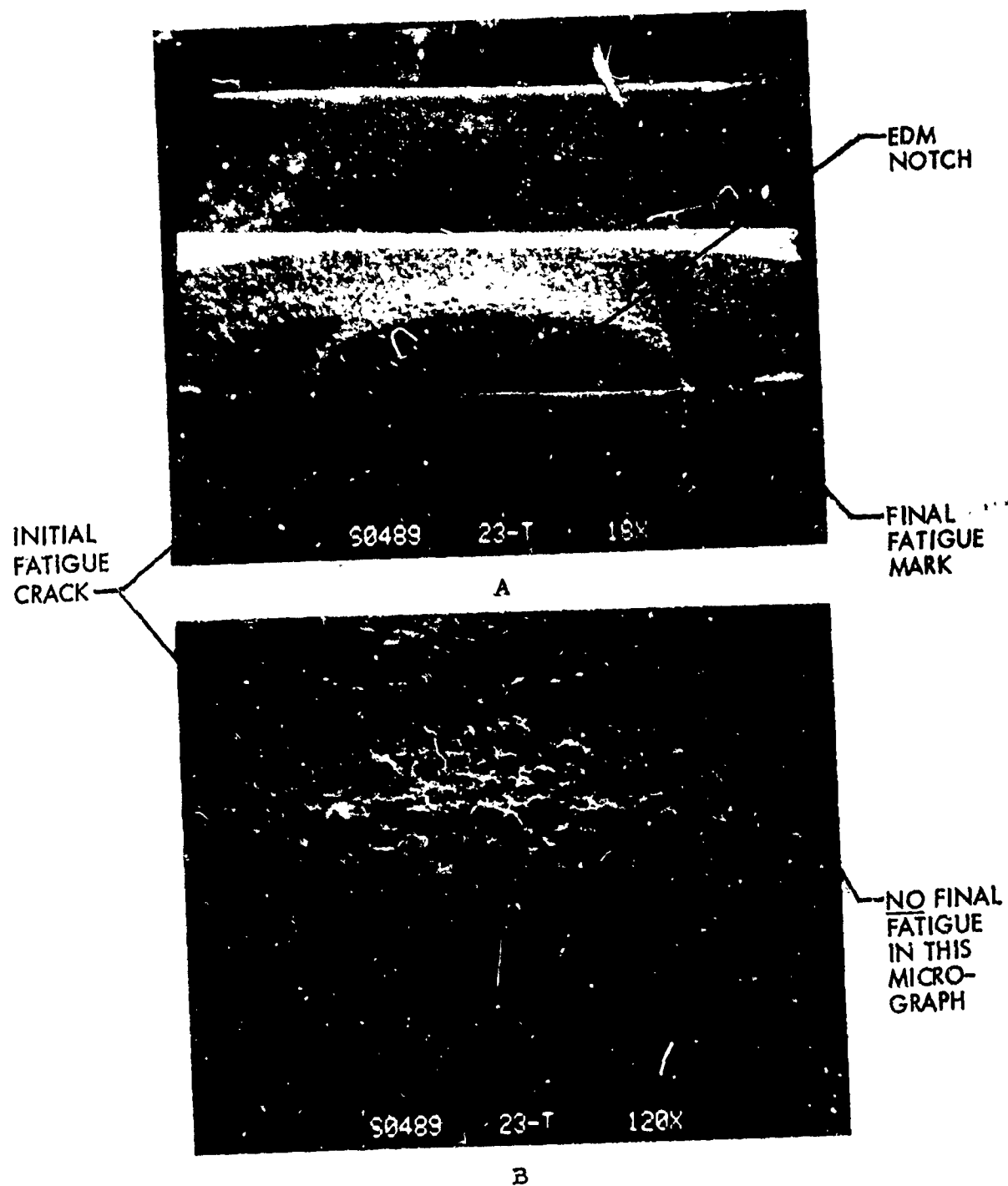


Figure 18. Scanning Electron Micrograph Titanium Specimen 23T After 1000 Hour Exposure to Specification Grade UDMH

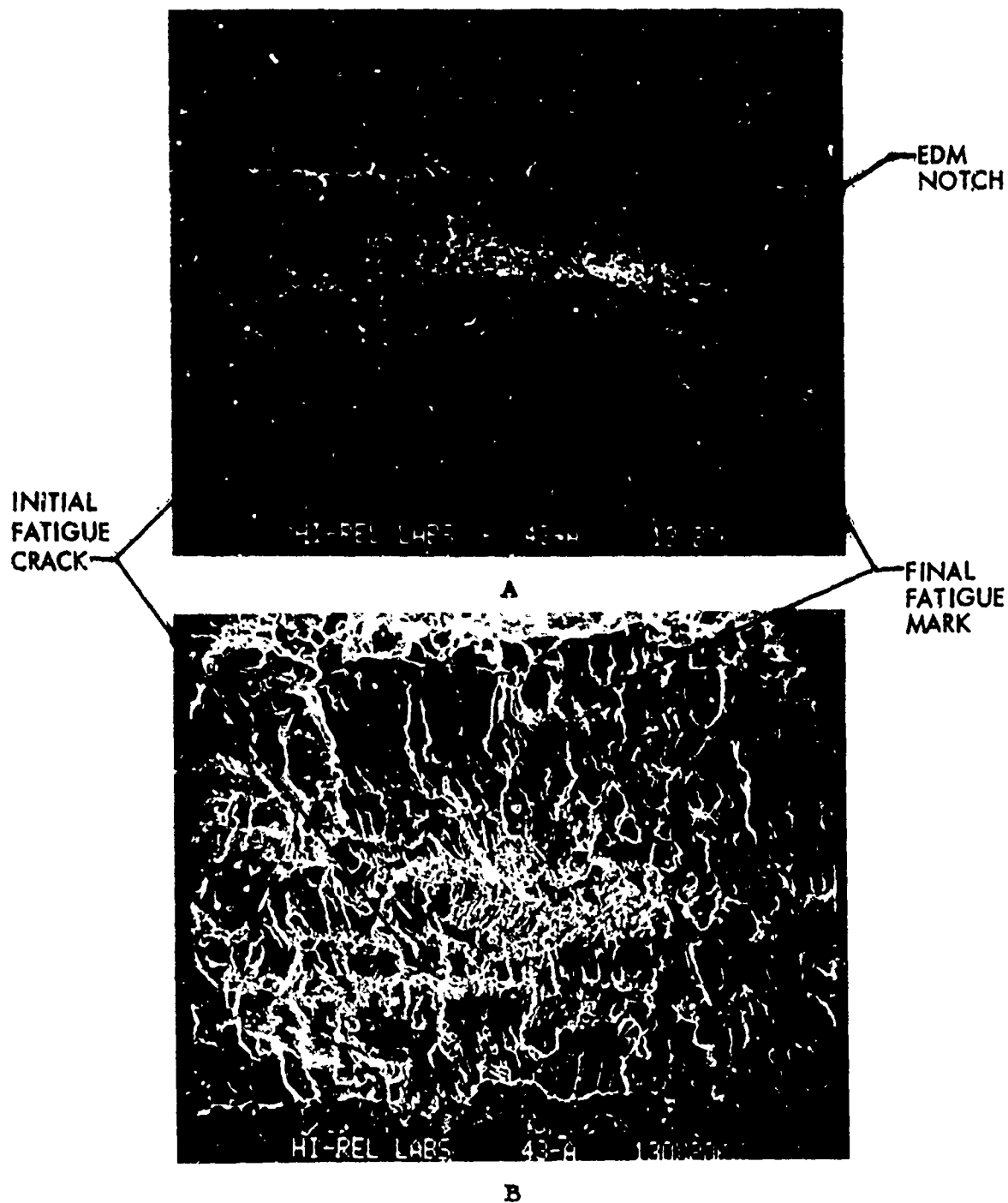


Figure 19. Scanning Electron Micrograph Aluminum Specimen 43A After Exposure to Distilled UDMH

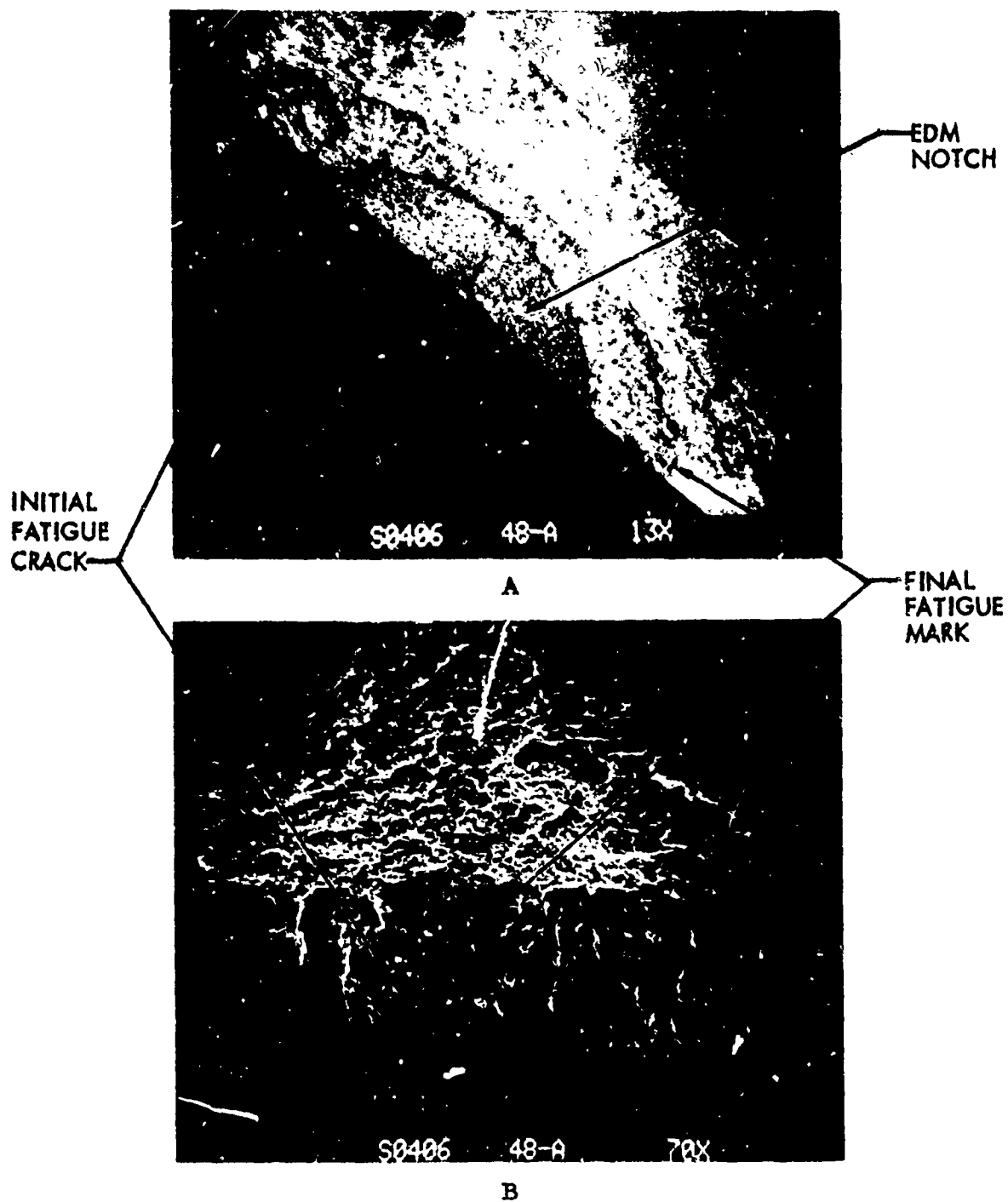


Figure 20. Scanning Electron Micrograph Aluminum Specimen 48A After 1000 Hour Exposure to UDMH:HCl

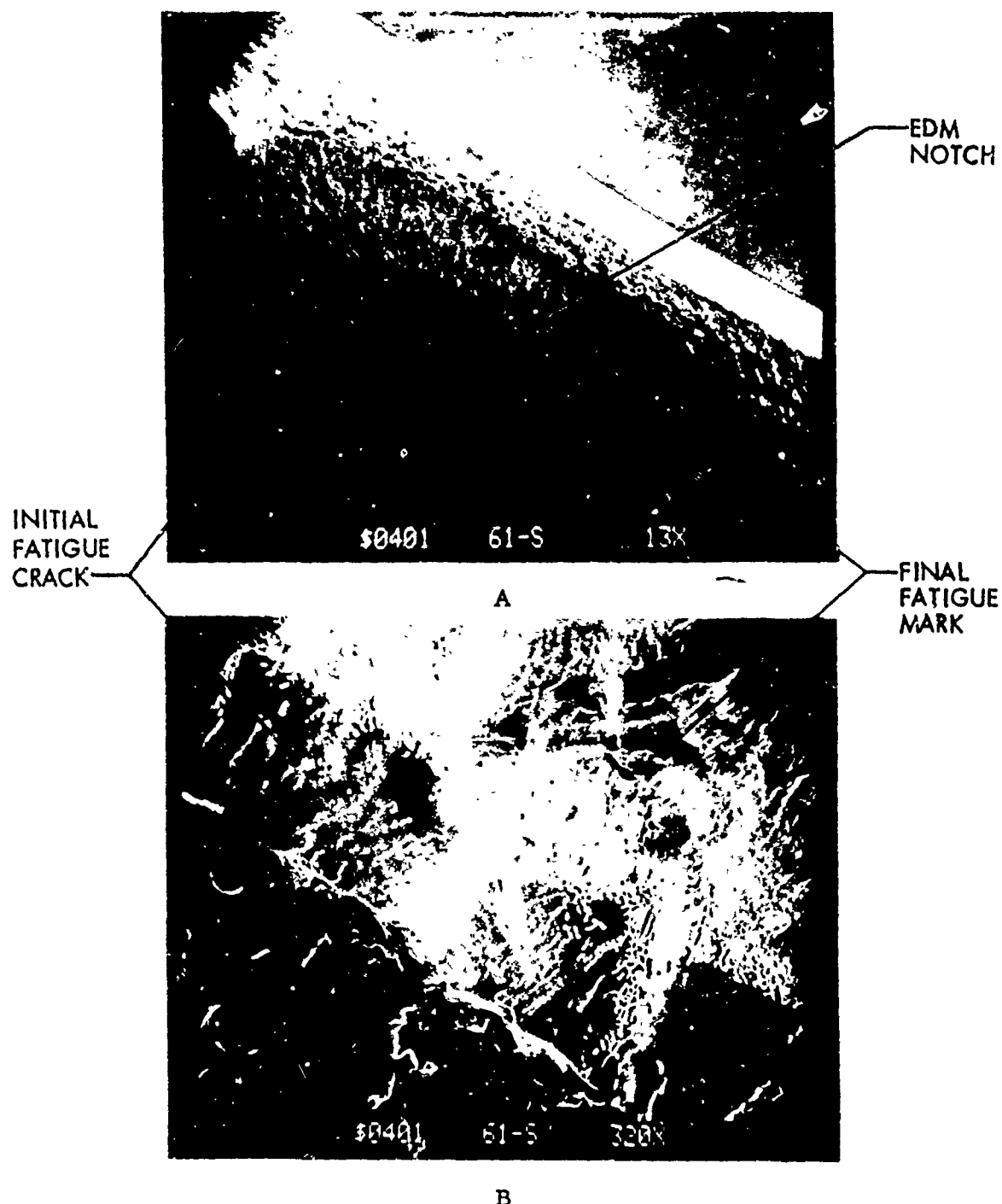


Figure 21. Scanning Electron Micrograph Corrosion Resistant Steel Specimen 61S After Exposure to Distilled UDMH

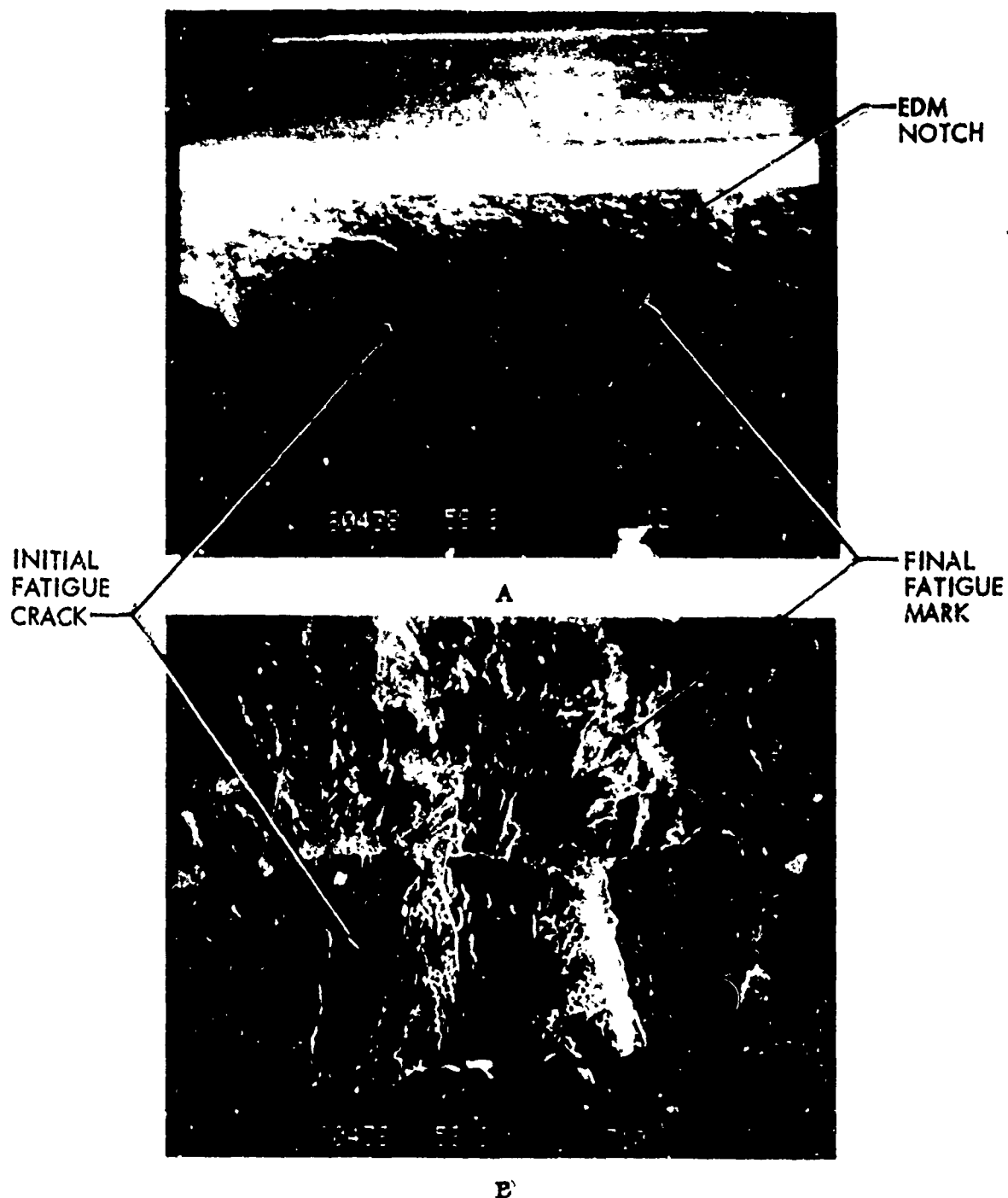
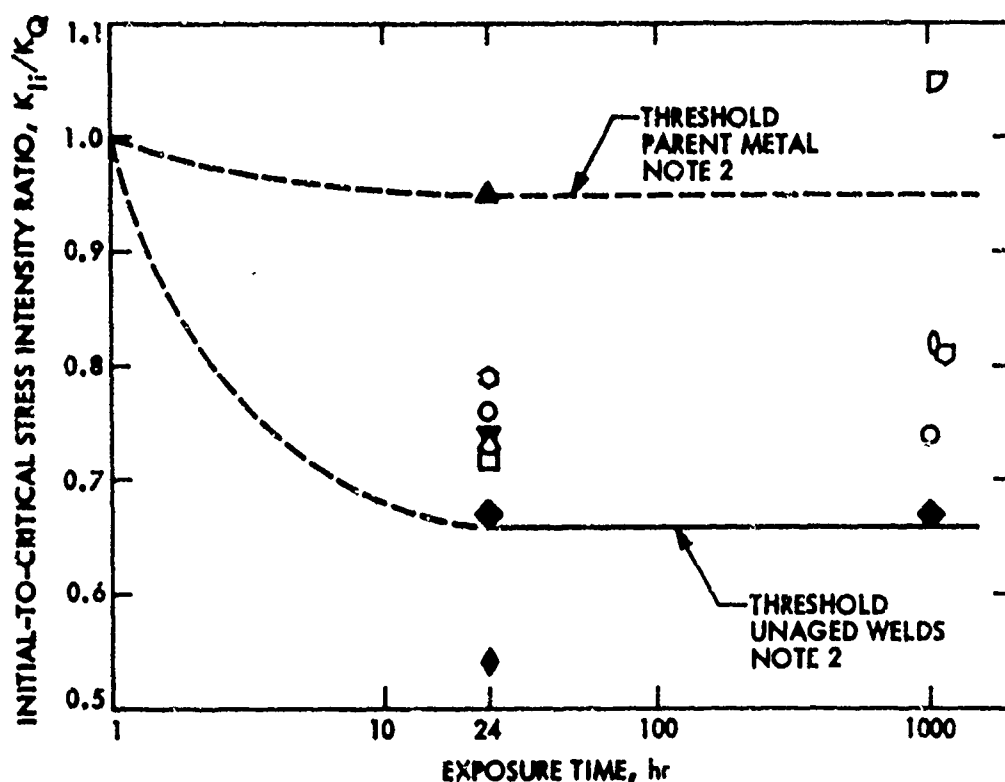


Figure 22. Scanning Electron Micrograph Corrosion Resistant Steel Specimen 58S After 1000 Hour Exposure to UDMH:HCl



NOTES:

1. LEGEND - UDMH WITH TITANIUM SHEET (OR FORGING) AT 49°C

HCl ppm	LiCl ppm	OTHER
○ 30	□ 36	▽ DISTILLED
△ 100	○ 100	○ SPECIFICATION GRADE FOOD MACHINERY
□ 100 + 1.5% H ₂ O		
◇ 100 (FORGINGS)		

2. LEGEND - HYDRAZINE REFINED GRADE AT 41°C (CHLORIDE < 1 ppm)

◇ UNAGED WELDS
△ FORGINGS

3. LEGEND - ISOPROPYL ALCOHOL AT 32°C

◇ FORGINGS

4. SYMBOLS - OPEN INDICATE NO GROWTH
CLOSED INDICATE GROWTH



Figure 23. Sustained Load Flaw Growth Data Comparison UDMH with Hydrazine-Titanium 6AL-4V Material

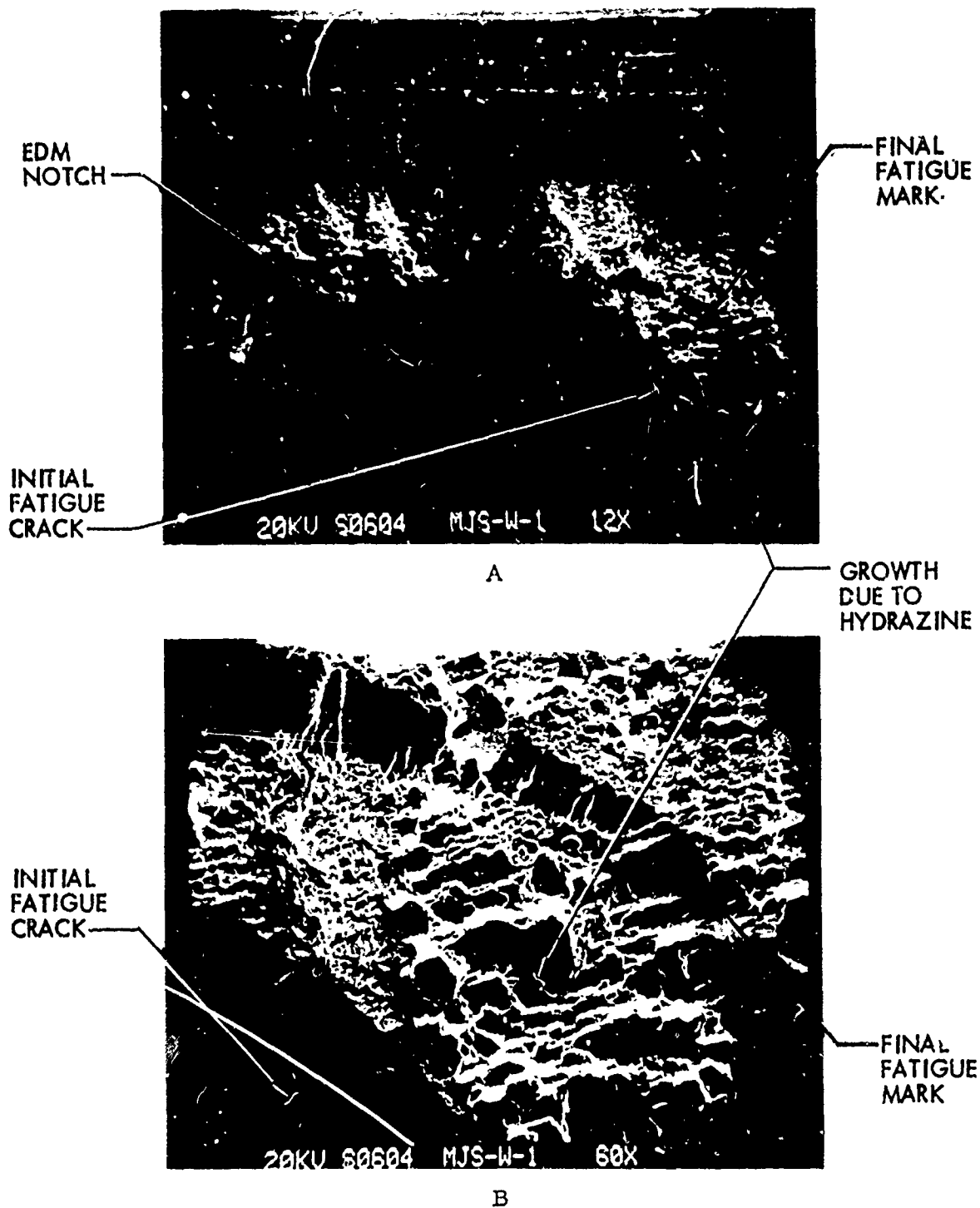
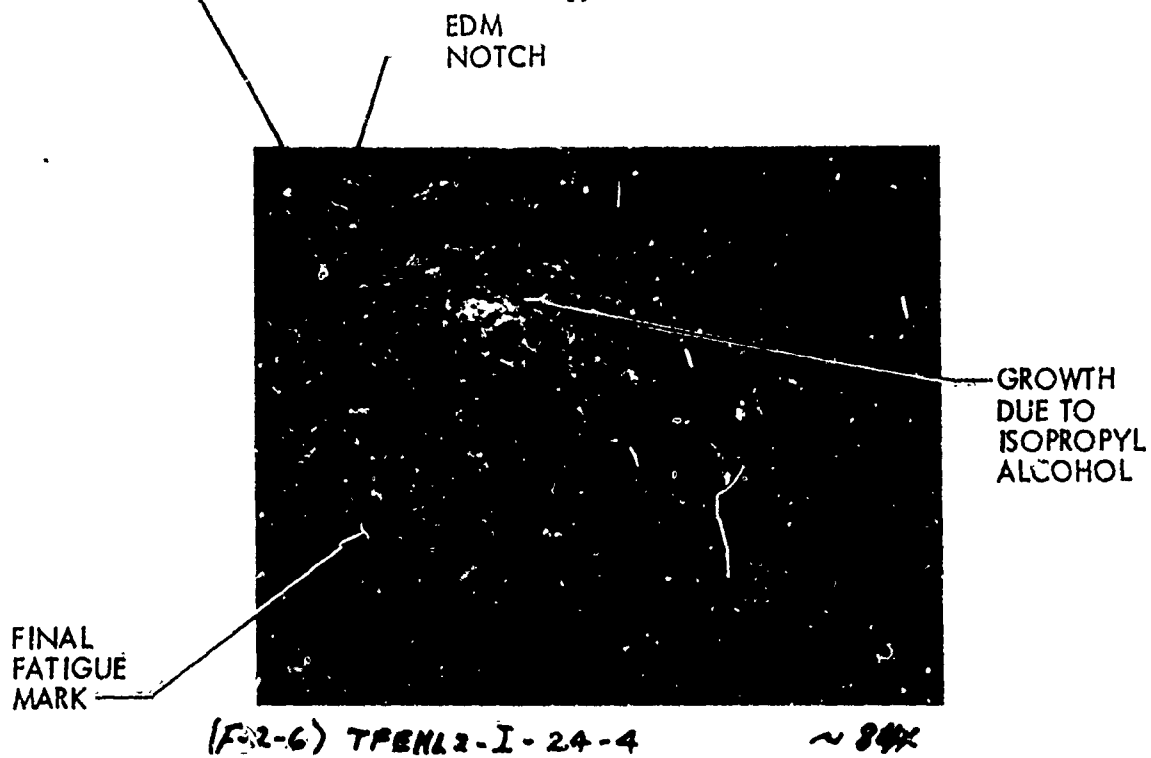


Figure 24. Scanning Electron Micrograph Titanium Forging Unaged Weld After Exposure to Hydrazine Showing Growth Region



A



B

Figure 25. Scanning Electron Micrograph Titanium Forging After Exposure to Isopropyl Alcohol Showing Growth Region

Table 1. Test Plan - Propellant Variations and Materials^a

PROPELLANT	MATERIALS		
UNS-DIMETHYL-HYDRAZINE, UDMH	TITANIUM, 6Al-4V, HEAT TREATED	ALUMINUM 2014-T6, HEAT TREATED	CORROSION RESISTANT STEEL, TYPE 304L, ANNEALED
DISTILLED	AS DISTILLED	AS DISTILLED	AS DISTILLED
DOPED, HCl, ppm ^b	25 AND 100	100	100
DOPED, LiCl, ppm ^b	25 AND 100	100	100
SPECIFICATION GRADE	AS RECEIVED	AS RECEIVED ^c	AS RECEIVED ^c

NOTES:

^aTESTS TO BE CONDUCTED IN ACCORDANCE WITH CONDITIONS INDICATED.
TEST TEMPERATURE 49°C (120°F)

^bCHLORIDE LEVEL, parts per million

^cTESTS TO BE ESTABLISHED BASED UPON PRECEDING RESULTS

Table 2. Assays of Uns-Dimethylhydrazine, (CH₃)₂NNH₂, Propellant

CONSTITUENT OR PROPERTY	SPECIFICATION LIMITS MIL-P-25604D	DISTILLED		DOMED ^c UDMH · HCl	DOMED ^d UDMH · LiCl
		AFRPL ^a	JPL ^b		
UDMH ASSAY, % BY WEIGHT	98.0 MIN	99.4	99.7	99.3	99.3
WATER, % BY WEIGHT	0.3 MAX	<0.1	<0.1	<0.1	<0.1
AMINES, % BY WEIGHT	1.5 MAX	0.7	0.19	0.7	0.7
N-NITROSODIMETHYLAMINE, % BY WEIGHT	0.01 MAX	—	—	—	—
CHLORIDE, % BY WEIGHT	0.0005 MAX	0.00004	0.00015	0.0030 ^b (0.0032) ^b	0.0036 ^b (0.0034) ^b
DENSITY, g/ml AT 25°C	0.783 TO 0.786	—	—	—	—
CARBON DIOXIDE, % BY WEIGHT	NOT REQUIRED	0.00008	0.0002	0.001 ^b (0.0004) ^b	0.0032 ^b (0.0004) ^b
PARTICULATE, mg/l	10.0 MAX	—	—	—	—
DISSOLVED METALS, PARTS PER MILLION% (ppm)	NOT REQUIRED	—	—	—	—
ALUMINUM		<0.5	<0.1	<0.5	<0.5
CHROMIUM		<0.1	—	<0.1	<0.1
COBALT		<0.1	<0.03	<0.1	<0.1
IRON		<0.1	<0.03	<0.1	<0.1
MANGANESE		<0.1	<0.02	<0.1	<0.1
NICKEL		<0.1	<0.03	<0.1	<0.1
TITANIUM		<0.5	<0.1	<0.5	<0.5
VANADIUM		<0.5	<0.1	<0.5	<0.5

NOTES:

^a ANALYSES PERFORMED BY AFRPL, LABORATORY TEST REPORT No. 75-75 (FEB 1975)

^b ANALYSES PERFORMED BY JPL, INTERNAL REPORT No. 382-215 (JULY 1975) AND 382-248 (AUG 1975)

^c UDMH WAS DOPED WITH HCl ALSO TO 0.0108% AND 0.0104%; SEE AFRPL REPORTS 143-75 (MAR 1975) AND 236-75 (APR 1975). BOTH CONTAMINATION LEVELS WERE USED

^d UDMH WAS DOPED WITH LiCl ALSO TO 0.0105 AND 0.0090%; SEE AFRPL REPORTS 143-75 (MAR 1975) AND 209-75 (APR 1975). BOTH CONTAMINATION LEVELS WERE USED

Table 3. Material Composition Analyses^a (% by Weight)

MATERIAL IDENTIFICATION	TITANIUM SHEET 6Al-4V	TITANIUM FORGING 6Al-4V	ALUMINUM PLATE 2014-T6	CORROSION RESISTANT STEEL, TYPE 304L
SPECIFICATION	MIL-T-9046	MIL-T-9047	QQ-A-250/3	MIL-S-4043
SPECIMEN NUMBER	21T	RMI HEAT NO. 301275	51A	605
ELEMENT				
ALUMINUM	6.02	6.3	BALANCE	0.034
CARBON	0.012	0.024		18.25
CHROMIUM			0.04	0.22
COPPER			4.15	BALANCE
IRON	0.08	0.14	0.52	
HYDROGEN	0.0056	0.116		
MAGNESIUM			0.53	1.39
MANGANESE			0.80	0.39
MOLYBDENUM				9.15
NICKEL				
NITROGEN	0.0082	0.008		0.030
OXYGEN	0.102	0.176		0.58
PHOSPHORUS				0.019
SILICON			0.84	
SULPHUR				
TITANIUM	BALANCE	BALANCE	0.02	
VANADIUM	4.00	4.3	0.08	
ZINC				
CERTIFICATION SOURCES	TITECH TEST LAB. REPORT 8C75-627 ^b	TITANIUM METALS CORPORATION OF AMERICA REPORT NO. R69-327	a) NASA-MSFC MATERIALS DIVISION REPORT EH32 (75-34) b) PEABODY TESTING MAGNAFLUX REPORT 74793-4-1 ^b	a) NASA-MSFC MATERIALS DIVISION REPORT EH32 (75-34) b) PEABODY TESTING MAGNAFLUX REPORT 74793-4-2 ^b

NOTES:

^a COMPLIES WITH SPECIFICATION REQUIREMENTS FOR CHEMICAL COMPOSITION

^b ANALYSIS CONDUCTED AFTER SPECIMEN SUBJECTED TO 1000 hr DURATION TEST

Table 4. Mechanical Properties^a

MATERIAL IDENTIFICATION	TITANIUM SHEET 6Al-4V		TITANIUM FORGING 6Al-4V		ALUMINUM PLATE 2014-T6		CORROSION RESISTANT STEEL, TYPE 304L	
SPECIFICATION	MIL-T-9046		MIL-T-9047		QQ-A-25013		MIL-S-4043	
CONDITION	HEAT TREATED		HEAT TREATED		HEAT TREATED		ANNEALED	
SPECIMEN NUMBER	71T	72T	HEAT NUMBER 301275/220 ^b	HEAT NUMBER 301275/231 ^b	41A-1	41A-2	655-1	655-2
ULTIMATE STRESS M Pa = MN/m ²	1186 (172000)	1200 (174000)	1234 (179000)	1269 (184000)	471 (68,300)	474 (68,700)	604 (87,600)	613 (88,900)
YIELD STRESS M Pa = MN/m ² (= psi)	1124 (163000)	1158 (168000)	1145 (166000)	1179 (171000)	440 (63,800)	443 (64,200)	291 (42,200)	276 (40,000)
ELONGATION, % ^c	7	9	13.7 ^d	12.9 ^d	9.0	10.0	54.0	56.0
CERTIFICATION	JPL MATERIALS LAB. REPORT 1175-25		MAGNAFLUX TEST LAB. REPORT 40515-6-1	MAGNAFLUX TEST LAB. REPORT 40515-6-2	JPL MATERIALS LAB. REPORT 1175-25		JPL MATERIALS LAB. REPORT 1175-25	

NOTES:

^aCOMPLIES WITH SPECIFICATION REQUIREMENTS FOR MECHANICAL PROPERTIES

^bREACTIVE METALS INCORPORATED HEAT NUMBER 301275, FORGINGS 220 AND 231

^cIN 5.08 cm (2.0 in.) EXCEPT AS NOTED

^dIN 2.54 cm (1.0 in.)

Table 5. Summary of Propellant Variations, Materials, and Specimens Used^a

PROPELLANT	MATERIAL			
	TITANIUM, 4AI-4V	ALUMINUM, 2014-T6	CORROSION RESISTANT STEEL, TYPE 304L	
JNS-DIMETHYL- HYDRAZINE, UDMH				
	NUMBER TEST SPECIMENS USED			
TEST DURATION HOURS	24	1000	24	1000
DISTILLED ^b	3 (0.4)		3 (0.4)	
DOPED, HCl ^b	3 (30) 3 (100)	3 (30) 3 (100) 3 (100)	3 (108)	3 (104)
DOPED, LiCl ^b	3 (36) 3 (100)		3 (105) 3 (90)	3 (100)
SPECIFICATION GRADE ^c		3		
TOTAL	15	12	12	3

NOTES:

^aTEST TEMPERATURE 49°C (120°F)

^b() CHLORIDE CONTENT, parts per million

^cUDMH IN "AS RECEIVED CONDITION"; SUPPLIER: FOOD MACHINERY CORP.

Table 6. Results of Sustained Load Tests of Titanium Alloy, 6Al-4V, in UDMH With Various Chloride Contents

SPECIMEN	CHLORIDE CONTENT	TEST LENGTH hr	INITIAL CRACK DEPTH		INITIAL CRACK LENGTH		APPLIED STRESS		APPLIED STRESS INTENSITY K_{II}		K_{II}/K_{IQ} -198°C (-320°F)	AMOUNT OF CRACK GROWTH m (in.)	REMARKS
			m	in.	m	in.	MN/m ²	ksi	MM/m ² √m	ksi√in.			
6-T	30 ppm HCl	24	6.604 × 10 ⁻⁴	0.026	3.251 × 10 ⁻³	0.128	926	134.3	48.2	43.9	0.77	NONE	DIMPLED
5-T			6.350	0.025	3.023	0.119	801	116.2	39.4	35.9	0.62		
4-T			6.350	0.025	3.200	0.126	518	75.1	25.4	23.1	0.38		
7-T	36 ppm LiCl		6.350	0.025	2.896	0.114	911	132.2	45.0	41.0	0.72		DIMPLED
11-T			5.842	0.023	2.921	0.115	769	111.6	35.8	32.6	0.53		
9-T			6.604	0.026	2.972	0.117	498	72.3	23.9	21.8	0.46		
8-T	0.4 ppm DISTILLED		6.604	0.026	2.921	0.115	904	131.1	44.9	40.9	0.75		DIMPLED
10-T			6.096	0.024	3.098	0.122	788	114.3	36.3	34.8	0.62		
13-T			6.604	0.026	3.302	0.130	503	72.9	25.0	22.8	0.44		
12-T	100 ppm LiCl		7.366	0.029	3.073	0.121	914	132.6	49.2	44.8	0.80		DIMPLED
15-T			6.096	0.024	3.073	0.121	768	111.4	36.9	33.6	0.63		DIMPLED
14-T			6.604	0.026	3.505	0.138	500	72.5	25.0	22.8	0.47		
16-T	100 ppm HCl		6.350	0.025	3.124	0.123	915	132.8	46.3	42.2	0.75		DIMPLED
19-T			6.096	0.024	3.048	0.120	769	111.5	36.8	33.5	0.59		DIMPLED
17-T		24	5.842	0.023	2.997	0.118	528	76.6	24.6	22.4	0.46		
18-T	30 ppm HCl	1000	6.096	0.024	3.276	0.129	896	129.9	44.6	40.6	0.74		DIMPLED
20-T		1000	5.142	0.023	3.023	0.119	760	110.3	35.7	32.5	0.56		DIMPLED
21-T		1000	6.858	0.027	3.023	0.119	514	74.6	25.3	23.0	0.39		
68-T	100 ppm HCl	1010	15.49	0.061	6.400	0.252	749	108.6	52.4	47.7	0.90		FORGING
70-T		1010	15.75	0.062	6.299	0.248	663	96.2	46.1	42.0	0.90		FORGING
69-T		1010	15.75	0.062	6.578	0.259	587	85.2	41.3	37.6	1.05		FORGING
27-T	100 ppm HCl + 1.5% H ₂ O	1124	7.112	0.028	2.972	0.117	930	134.9	48.4	44.1	0.81		DIMPLED
26-T		1124	6.858	0.027	3.124	0.123	817	118.5	42.2	38.4	0.63		DIMPLED
29-T		1124	5.842	0.023	3.124	0.123	696	100.0	32.7	29.8	0.53		
23-T	FOOD MACHINERY	1012	8.382	0.033	3.352	0.132	929	134.8	54.1	49.2	0.82		DIMPLED
22-T		1012	7.112	0.028	3.530	0.139	858	124.5	46.8	42.6	0.74		DIMPLED
28-T		1012	6.604 × 10 ⁻⁴	0.026	2.972 × 10 ⁻³	0.117	797	115.6	39.1	35.9	0.62	NONE	

NOTE: ALL TESTS WERE CONDUCTED AT 49°C (120°F) WITH 2.068 ± 0.34 MN/m² (300 ± 50 psi) APPLIED TO THE CRACK TIP

Table 7. Results of Sustained Load Tests of Aluminum Alloy, 2014-T6, in UDMH With Various Chloride Contents

SPECIMEN	CHLORIDE CONTENT	TEST LENGTH hr	INITIAL CRACK DEPTH		INITIAL CRACK LENGTH		APPLIED STRESS		APPLIED STRESS INTENSITY K_{II}		$\frac{K_{II}}{K_{IQ}}$ 21°C (70°F)	AMOUNT OF CRACK GROWTH m (in.)	REMARKS
			m	in.	m	in.	MPa/m ²	ksi	MPa/m ² √m	ksi√in.			
35-A	108 PPM	24	1.295 × 10 ⁻³	0.051	6.223 × 10 ⁻³	0.245	377	54.7	29.9	25.4	0.90	NONE	
37-A	HCl	↑	1.422	0.056	6.833	0.269	330	47.9	25.9	23.6	0.77		
39-A			1.422	0.056	6.985	0.273	235	34.1	17.9	16.3	0.52		
42-A	90 PPM		1.321	0.052	6.350	0.250	284	41.2	20.7	18.8	0.66		DIMPLED
44-A	LiCl		1.346	0.053	6.502	0.256	239	34.7	17.5	15.9	0.55		
49-A			1.346	0.053	6.553	0.258	200	29.0	14.5	13.2	0.43		
43-A	DISTILLED		1.321	0.052	6.071	0.239	285	41.3	20.4	18.6	0.65		DIMPLED
45-A	0.4 PPM	↓	1.270	0.050	5.994	0.236	243	35.2	16.7	15.2	0.53		
50-A		24	1.245	0.049	6.045	0.238	199	28.9	13.5	12.3	0.42		
48-A	108 PPM	1000	1.422	0.056	6.172	0.243	296	42.9	22.4	20.4	0.69		DIMPLED
46-A	HCl	1000	1.397	0.055	6.853	0.270	253	36.7	19.3	17.6	0.58		DIMPLED
51-A		1000	1.372 × 10 ⁻³	0.054	6.248 × 10 ⁻³	0.246	214	31.0	15.6	14.2	0.46	NONE	

NOTE: ALL TESTS WERE CONDUCTED AT 49°C (120°F) WITH 2.068 ± 0.34 MPa/m² (300 ± 50 psi) APPLIED TO THE CRACK TIP

Table 8. Results of Sustained Load Tests of Corrosion Resistant Steel, Type 304L, in UDMH With Various Chloride Contents

SPECIMEN	CHLORIDE CONTENT	TEST LENGTH hr	INITIAL CRACK DEPTH		INITIAL CRACK LENGTH		APPLIED STRESS		APPLIED STRESS INTENSITY K_{II}		AMOUNT OF CRACK GROWTH m (in.)	REMARKS
			m	in.	m	in.	MM/m^2	ksi	$MM/m^2\sqrt{m}$	$ksi\sqrt{in.}$		
54-S	104 PPM	24	8.636×10^{-4}	0.034	3.708×10^{-3}	0.146	177	25.7	9.6	8.7	NONE	NO DIMPLING
52-S	HCl	↑	7.620	0.030	3.378	0.133	156	22.7	7.8	7.1		
53-S			7.620	0.030	3.098	0.122	138	20.0	6.6	6.6		
57-S	100 PPM	↓	10.16	0.040	4.191	0.165	186	27.0	11.0	10.0	NONE	
58-S	LiCl		9.144	0.036	4.216	0.166	165	24.0	9.3	8.5		
59-S			8.636	0.034	3.607	0.142	145	21.0	7.6	6.9		
61-S	DISTILLED	↓	7.112	0.028	3.810	0.150	188	27.2	9.8	8.9	NONE	
62-S	0.4 PPM		7.620	0.030	3.912	0.154	164	23.8	8.6	7.8		
63-S			7.874	0.031	3.861	0.152	143	20.7	7.3	6.6		
55-S	104 PPM	1000	7.874	0.031	3.531	0.139	185	26.8	9.4	8.6	NONE	
56-S	HCl	1000	8.382	0.033	3.531	0.139	165	23.9	8.6	7.8		
60-S		1000	7.874×10^{-4}	0.031	3.683×10^{-3}	0.145	143	20.7	7.3	6.6		

NOTES:

THE YIELD STRENGTH OF THIS ALLOY IS 172-207 MN/m² (25-30 ksi)

ALL TESTS WERE CONDUCTED AT 49°C (120°F) WITH 2.068 ± 0.34 MN/m² (300 ± 50 psig) APPLIED TO THE CRACK TIP

APPENDIX A

The material in this appendix presents a discussion of "fracture mechanics as applied to environmental effects on metals".

Fracture mechanics is that sub-discipline of mechanics which treats the macroscopic, continuum behavior of a sharp crack as it interacts with any stress field. Fracture mechanics began with the solution of the stress field surrounding cracks in thin plates by Inglis in 1913, reference 10, followed by application of these solutions to fracture of glass by Griffith in 1920, reference 11. The next major step in the application of fracture mechanics to structural problems occurred in 1962 when Irwin, reference 12, applied the concepts to real cracks in metals. Tiffany and Masters, references 6, 9, and 14, then applied Irwin's solution to interactions between sharp cracks under stress and chemical environments.

Practical application of fracture mechanics increased when it became obvious that the smallest crack that was detectable by the best nondestructive inspection techniques was larger than that crack which would cause fracture below the yield stress of most high strength alloys. The status of this application was very adequately described by Tiffany in a NASA Space Vehicle Design Criteria document, reference 3. This document described in detail both the fracture mechanics theory and its application to environmental effects on metals, therefore these subjects will not be discussed herein (See references 7 and 13 also). The remainder of this appendix will consist of a discussion of those portions of (reference 3) which are necessary to reproduce the calculations of K_{Ii} and K_Q shown in Appendix B and Tables B1 - B5.

The equation which describes the relationship between a part-through surface crack and the applied remote gross stress is

$$K = 1.1 \sqrt{\pi(a/Q)} M_K \sigma \quad (1)$$

The flaw shape parameter, Q is obtained from Figure A1. The magnification factor, M_K , is obtained from Figure A2. For calculation of the applied initial sustained load stress intensity for a given environment, Equation (1) becomes

$$K_{Ii} = 1.1 \sqrt{\pi(a/Q)i} M_{Ki} \sigma_i \quad (2)$$

Calculation of K_Q is done by substituting the critical conditions into Equation (1) i.e.

$$K_Q = 1.1 \sqrt{\pi(a/Q)c} M_{Kc} \sigma_c \quad (3)$$

The parameter K_{Ii}/K_Q is calculated for each specimen because the material property for environmental effects appears to be this ratio rather than the absolute value of K_{Ii} . Since K_Q shows the same magnitude of scatter from specimen to specimen as other material properties such as the yield strength, it is important to measure K_Q on each specimen. The dimensionless plot of K_{Ii}/K_Q versus exposure time minimizes the scatter in the data and allows a clear crack growth threshold to be seen.

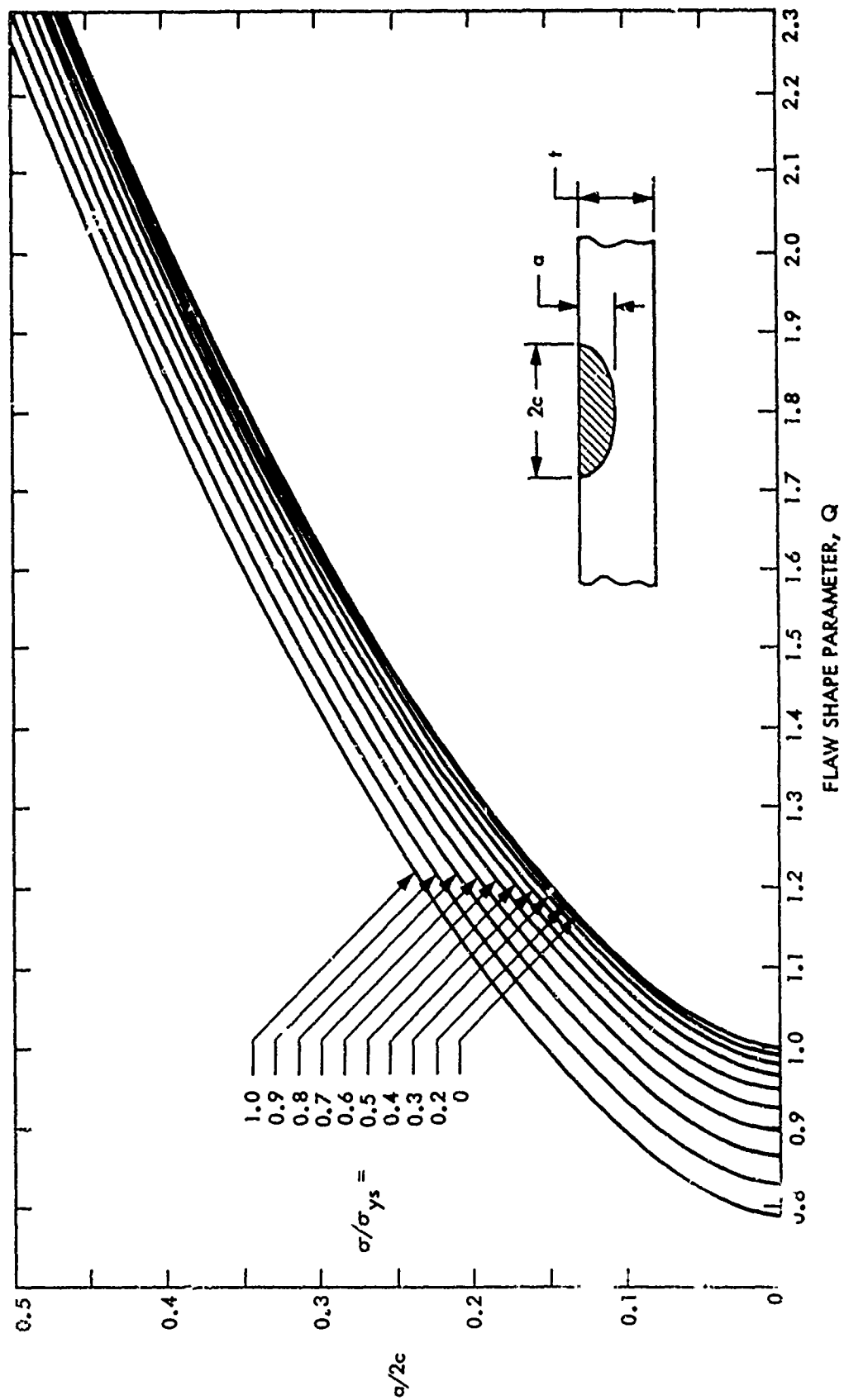


Figure A1. Shape Parameter Curves for Surface Flaws (Reference 3)

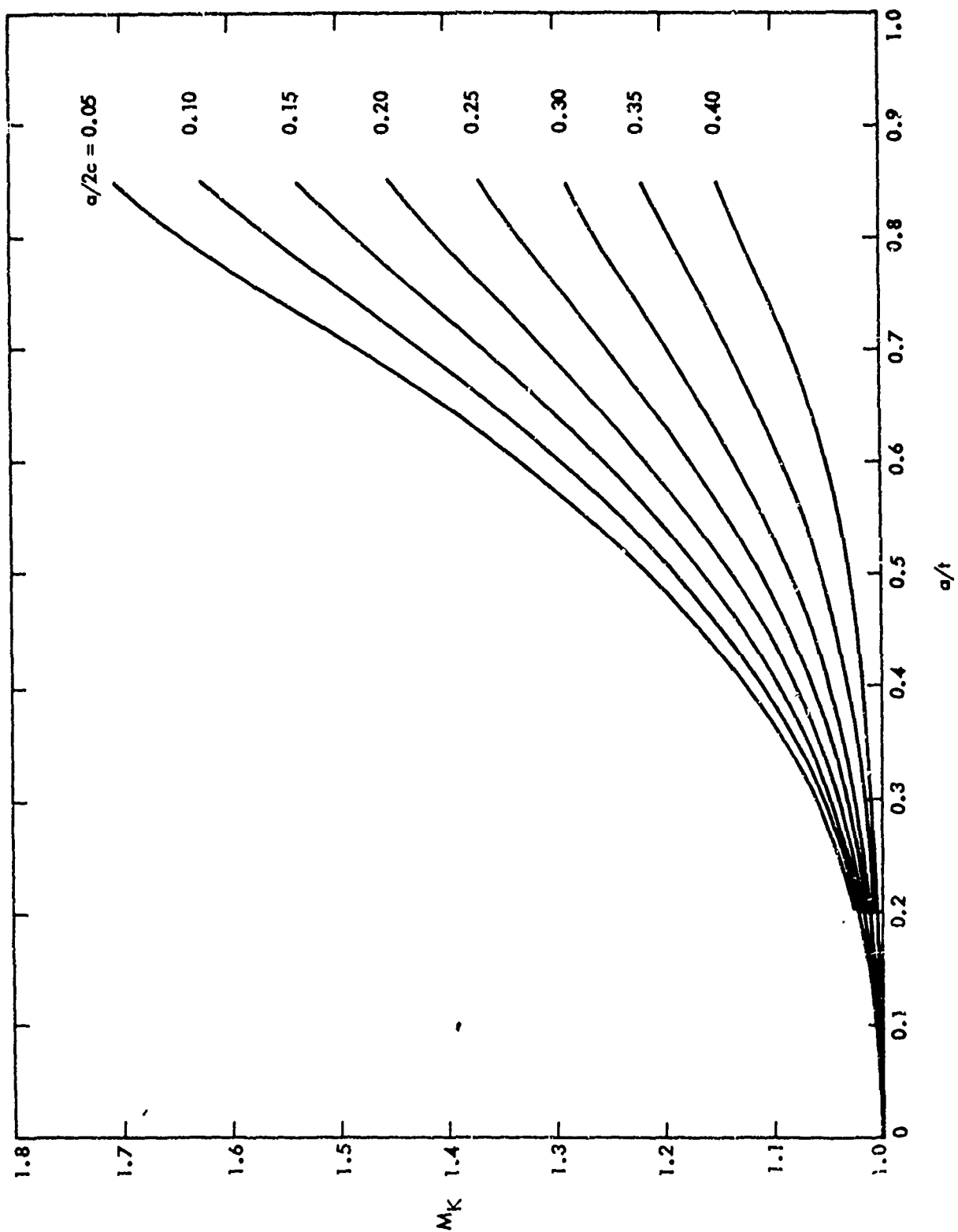


Figure A2. Magnification Curves (Reference 7)

APPENDIX B

The material presented in this appendix covers the detail results for the three subject materials tested with UDMH propellant.

Temperatures are defined as follows:

1. Sustained Load Tests conducted at 49°C (120°F).
2. Ambient or Room Temperature (R.T.) 21°C (70°F).
3. Liquid Nitrogen -196°C (-320°F).

Table B1. Sustained Load Flow Growth Data - Titanium Alloy

SPECIMEN IDENTIFICATION	THICKNESS (m)	WIDTH (m)	LOAD (kN)	STRESS σ_i (MN/m ²)	INITIAL CRACK DEPTH a_i (m)	INITIAL CRACK LENGTH $2C_i$ (m)	$\frac{a_i}{2C_i}$	$\frac{\sigma_i}{\sigma_{ys}}$	$\frac{a_i}{Y}$	$\frac{\sigma_i}{Q_i}$	$\frac{a_i}{Q_i}$ (m)	M_{ki}	$K_{Ii} \frac{MN \sqrt{m}}{2}$	$K_{II} \frac{K_{IC}}{-196} ^\circ C$	TEST LENGTH (mm)	REMARKS
30 ppm HCl																
6-T	1.366×10^{-3}	1.450×10^{-2}	18.3	926	6.60×10^{-4}	10^{-3}	0.203	0.84	0.483	1.18	5.588×10^{-4}	1.13	48.2	0.76	24	DIMPLED
5-T	1.351	1.694	18.3	802	6.35		0.210	0.73	0.470	1.23	5.080	1.11	39.4	0.61	24	
4-T	1.374	2.578	18.3	518	6.35	3.20	0.198	0.47	0.462	1.26	5.08	1.12	25.3	0.38	24	
36 ppm LiCl																
7-T	1.364	1.450	18.0	911	6.35	2.89	0.219	0.83	0.466	1.22	5.080	1.11	45.0	0.72	24	DIMPLED
11-T	1.384	1.694	18.0	769	5.84	2.92	0.200	0.70	0.422	1.22	4.826	1.09	35.8	0.57	24	
9-T	1.409	2.565	18.0	498	6.60	2.97	0.222	0.45	0.458	1.34	4.826	1.11	24.0	0.46	24	
DISTILLED																
8-T	1.371	1.455	18.0	904	6.60	2.92	0.226	0.82	0.481	1.25	5.334	1.11	44.9	0.74	24	DIMPLED
10-T	1.348	1.697	18.0	788	6.09	3.10	0.197	0.71	0.432	1.21	5.080	1.11	38.2	0.61	24	
13-T	1.400	2.565	18.0	502	6.60	3.30	0.200	0.46	0.472	1.27	5.080	1.12	25.0	0.44	24	
100 ppm LiCl																
12-T	1.346	1.466	18.0	914	7.36	3.07	0.240	0.80	0.547	1.28	5.842	1.15	49.2	0.79	24	DIMPLED
15-T	1.374	1.709	18.0	768	6.09	3.07	0.198	0.70	0.444	1.21	5.080	1.10	36.9	0.63	24	DIMPLED
14-T	1.402	2.575	18.0	500	6.60	3.50	0.186	0.45	0.471	1.25	5.334	1.12	25.0	0.47	24	
100 ppm HCl																
16-T	1.351	1.458	18.0	915	6.35	2.12	0.203	0.83	0.470	1.18	5.334	1.12	46.3	0.74	24	DIMPLED
19-T	1.379	1.702	18.0	769	6.09	3.05	0.200	0.70	0.442	1.22	5.080	1.10	36.8	0.59	24	DIMPLED
17-T	1.331	2.565	18.0	528	5.84	2.99	0.195	0.48	0.439	1.26	4.572	1.10	24.4	0.46	24	
30 ppm HCl																
18-T	1.377	1.455	17.9	895	6.09	3.27	0.186	0.81	0.443	1.15	5.33	1.11	44.6	0.74	1000	
20-T	1.384	1.704	17.9	760	5.84	3.02	0.193	0.69	0.422	1.20	4.826	1.09	35.7	0.56	1000	
21-T	1.369×10^{-3}	2.550×10^{-2}	17.9	514	6.36×10^{-4}	3.02×10^{-3}	0.227	0.47	0.501	1.35	5.080×10^{-4}	1.12	25.3	0.39	1000	

Table Bi. Sustained Load Flaw Growth Data - Titanium Alloy (Contd)

SPECIMEN IDENTIFICATION	THICKNESS (m)	WIDTH (m)	LOAD (kN)	STRESS σ_i (MN/m ²)	INITIAL CRACK DEPTH a_i (m)	INITIAL CRACK LENGTH $2C_i$ (m)	$\frac{\sigma_i}{2C_i}$	$\frac{\sigma_i}{\sigma_{ys}}$	$\frac{\sigma_i}{r}$	Q_i	$\frac{a_i}{Q_i}$ (m)	M_{ki}	$\frac{K_{II}}{MN \sqrt{m}}$	$\frac{K_{II}}{K_Q -1\% ^\circ C}$	TEST LENGTH (mm)	REMARKS
100 ppm HCl																
68-T	3.932×10^{-3}	2.537×10^{-2}	74.7	749	15.49×10^{-4}	6.40×10^{-3}	0.242	0.679	0.394	1.35	11.43×10^{-4}	1.06	52.4	0.90	1010	FORGING
	3.917	2.878	74.7	663	15.74	6.30	0.256	0.601	0.402	1.39	11.43	1.06	46.1	0.90	1010	FORGING
69-T	3.995	3.182	74.7	587	15.74	6.58	0.239	0.532	0.394	1.36	11.68	1.06	41.3	1.05	1010	FORGING
100 ppm HCl (1 1/2% H ₂ O)																
27-T	1.368	1.910	24.3	930	7.11	2.97	0.239	0.843	0.519	1.27	5.59	1.13	48.5	0.81	1124	
" "	1.364	2.179	24.3	817	6.86	3.12	0.220	0.741	0.503	1.25	5.59	1.13	42.2	0.63	1124	
27-T	1.366	2.555	24.3	696	5.84	3.12	0.187	0.631	0.428	1.21	4.83	1.10	32.7	0.53	1124	
FOOD MACHINERY																
23-T	1.372	2.222	28.3	929	8.38	3.35	0.250	0.842	0.611	1.31	6.35	1.18	54.1	0.82	1012	
" "	1.339	2.375	28.3	858	7.11	3.53	0.201	0.778	0.512	1.20	5.84	1.15	46.8	0.74	1012	
" "	1.394 $\times 10^{-3}$	2.550×10^{-2}	28.3	797	6.60×10^{-4}	2.97×10^{-3}	0.222	0.722	0.474	1.26	5.33×10^{-4}	1.11	39.4	0.62	1012	

Table B2. Plane Strain Fracture Toughness - Titanium Alloy

SPECIMEN IDENTIFICATION	WIDTH (m)	THICKNESS (m)	AREA (m ²)	FAILURE LOAD (kN)	FAILURE STRESS (NM/m ²)	CRACK DEPTH (m)	CRACK LENGTH 2C (m)	$\frac{a}{2C}$	$\frac{\sigma}{\sigma_{ys}}$	$\frac{a}{t}$	Q	$\frac{a}{Q}$ (m)	M_K	K_{IC} $\frac{MN}{m^{3/2}}$	REMARKS
G-T	2.57×10^{-2}	1.38×10^{-3}	3.55×10^{-5}	50.5	1422	5.08×10^{-4}	2.77×10^{-3}	0.163	0.86	0.368	1.13	4.57×10^{-4}	1.068	62.8	LN ₂ ↓
1-T	NO FATIGUE CRACK			51.2											
2-T	2.578	1.372	3.535	44.9	1270	6.350	3.277	0.194	0.77	0.463	1.18	5.334	1.198	65.8	
3-T	2.578	1.374	3.541	40.0	1130	5.842	3.200	0.183	0.58	0.425	1.18	4.826	1.098	53.8	
4-T	2.578	1.374	3.541	46.7	1318	6.350	3.404	0.187	0.80	0.462	1.15	5.588	1.118	67.6	
5-T	1.694	1.351	2.290	79.1	1270	6.604	3.098	0.213	0.77	0.488	1.23	5.334	1.124	64.5	
	1.450	1.366	1.980	22.1	1116	7.112	4.191	0.170	0.67	0.520	1.15	6.095	1.169	63.3	
7-T	1.450	1.364	1.980	24.6	1241	6.604	3.124	0.211	0.75	0.484	1.23	5.334	1.121	62.8	
8-T	1.455	1.371	1.993	23.6	1182	6.858	3.149	0.218	0.71	0.500	1.26	5.334	1.129	60.7	
9-T	2.565	1.410	3.619	38.3	1057	6.604	3.124	0.211	0.64	0.468	1.26	5.334	1.111	52.4	
10-T	1.697	1.348	2.290	27.9	1222	6.350	3.404	0.187	0.74	0.471	1.17	5.334	1.124	62.4	
11-T	1.694	1.384	2.348	29.6	1259	6.350	3.073	0.207	0.76	0.459	1.21	5.334	1.109	62.4	
12-T	1.465	1.346	1.974	22.3	1132	7.366	3.454	0.213	0.68	0.547	1.26	5.842	1.164	62.1	
13-T	2.565	1.399	3.593	39.1	1087	6.858	3.479	0.197	0.66	0.490	1.22	5.588	1.133	56.9	
	575	1.402	3.612	36.8	1019	6.858	3.658	0.188	0.62	0.489	1.21	5.588	1.136	53.7	
14-T	1.709	1.374	2.348	27.6	1174	6.350	3.302	0.192	0.71	0.462	1.19	5.334	1.116	59.0	
15-T	1.458	1.351	1.967	23.3	1181	6.858	3.531	0.194	0.71	0.508	1.20	5.842	1.146	63.1	
16-T	2.565	1.331	3.412	36.7	1076	6.096	3.231	0.188	0.65	0.453	1.20	5.060	1.116	52.8	
17-T	1.455	1.376	2.006	22.8	1137	6.604	3.810	0.173	0.61	0.490	1.15	5.842	1.140	60.5	
18-T	1.702	1.379	2.348	28.9	1234	6.350	3.378	0.188	0.75	0.460	1.16	5.588	1.117	62.9	
19-T	1.704	1.384	2.361	30.5	1291	6.096	3.251	0.186	0.78	0.440	1.16	5.334	1.100	63.5	
20-T	2.55×10^{-2}	1.37×10^{-3}	3.489×10^{-5}	42.9	1229	6.858×10^{-4}	3.353×10^{-3}	0.205	0.74	0.501	1.21	5.588×10^{-4}	1.140	65.0	

Table B2. Plane Strain Fracture Toughness - Ti-6Al-4V Alloy (Contd)

SPECIMEN IDENTIFICATION	WIDTH (m)	THICKNESS (m)	AREA (m ²)	FAILURE LOAD (kN)	FAILURE STRESS (MN/m ²)	CRACK DEPTH (m)	CRACK LENGTH (m)	$\frac{a}{2C}$	$\frac{\sigma}{\sigma_{ys}}$	$\frac{a}{r}$	Q	$\frac{a}{Q}$ (m)	M _K	K_{ISCC} (MN/m ^{3/2})	REMARKS
22-T	2.375 x 10 ⁻²	1.385 x 10 ⁻³	3.298 x 10 ⁻⁵	40.9	7240	0.686 x 10 ⁻³	3.175 x 10 ⁻³	0.216	0.750	0.494	1.25	0.548 x 10 ⁻³	1.12	63.4	LN ₂
23-T	2.222	1.372	3.048	30.7	1006	0.838	4.953	0.149	0.606	0.611	1.16	0.711	1.25	65.9	
26-T	2.179	1.364	2.972	36.0	1212	0.711	3.734	0.190	0.732	0.521	1.18	0.610	1.16	67.2	
27-T	1.097	1.369	2.610	27.6	1055	0.736	3.962	0.186	0.638	0.529	1.20	0.610	1.17	59.7	
28-T	2.550	1.245	3.175	40.5	1138	0.711	4.013	0.177	0.608	0.510	1.17	0.609	1.16	63.5	
29-T	2.555	1.366	3.490	42.7	1223	0.635	3.454	0.184	0.737	0.415	1.17	0.534	1.12	62.2	
68-T	2.537	3.93	9.97	79.2	793	1.53	7.54	0.205	0.400	0.394	1.29	1.19	1.08	57.9	
69-T	3.183	3.995	12.72	55.6	437	2.184	10.06	0.217	0.264	0.547	1.27	1.600	1.16	39.4	
70-T	2.878 x 10 ⁻²	3.916 x 10 ⁻³	11.27 x 10 ⁻⁵	83.2	736	1.575 x 10 ⁻³	7.569 x 10 ⁻³	0.206	0.445	0.402	1.44	1.072 x 10 ⁻³	1.08	51.4	LN ₂

Table B3. Sustained Load Flaw Growth Data - Aluminum Alloy

SPECIMEN IDENTIFICATION	THICKNESS (m)	WIDTH (m)	LOAD (kN)	STRESS σ_i (MN/m ²)	INITIAL CRACK DEPTH a_i (m)	INITIAL CRACK LENGTH $2C_i$ (m)	$\frac{a_i}{2C_i}$	$\frac{\sigma_i}{\sigma_s}$	$\frac{\sigma_i}{i}$	Q_i	$\frac{a_i}{Q_i}$	M_{K_I}	K_{II} $\frac{MN}{\sqrt{m}}$	$\frac{K_{II}}{K_Q}$ 21°C	TEST LENGTH (mm)	REMARKS
108 ppm HCl																
35-A	2.575 x 10 ⁻³	3.183 x 10 ⁻²	30.9	377	1.295 x 10 ⁻³	6.223 x 10 ⁻³	0.208	0.90	0.503	1.16	1.117 x 10 ⁻³	1.136	27.9	0.90	24	
37-A	2.570	3.640	30.9	330	1.422	6.832	0.208	0.79	0.533	1.21	1.168	1.172	25.3	0.77	24	
39-A	2.575	5.105	30.9	235	1.422	6.985	0.204	0.56	0.552	1.27	1.117	1.170	17.9	0.52	24	
105 ppm LiCl																
36-A	2.565	3.162	30.9	381	1.397	6.502	0.211	0.91	0.535	1.18	1.168	1.155	29.2		7 PLUS	FAILED (OVER STRESSED)
38-A	2.573	3.639	30.9	330	1.473	7.010	0.210	0.78	0.573	1.22	1.219	1.186			7 PLUS	
40-A	2.573	5.105	30.9	235	1.397	6.731	0.206	0.56	0.543	1.28	1.092	1.164			7 PLUS	
90 ppm LiCl																
42-A	2.535	3.630	26.1	264	1.321	6.350	0.208	0.68	0.521	1.25	1.067	1.148	20.6	0.66	24	DIMPLED
44-A	2.575	4.239	26.1	239	1.346	6.502	0.207	0.57	0.523	1.27	1.067	1.150	17.5	0.55	24	
49-A	2.558	5.105	26.1	200	1.346	6.553	0.205	0.48	0.526	1.29	1.042	1.153	14.5	0.43	24	
DISTILLED																
43-A	2.530	3.627	26.1	285	1.321	6.071	0.218	0.68	0.522	1.27	1.042	1.143	20.4	0.65	24	DIMPLED
45-A	2.540	4.239	26.1	243	1.270	5.994	0.212	0.58	0.500	1.30	0.965	1.131	16.7	0.53	24	
50-A	2.565	5.108	26.1	199	1.245	6.045	0.206	0.47	0.485	1.30	0.965	1.125	13.5	0.42	24	
108 ppm HCl																
48-A	2.568	3.632	27.6	296	1.422	6.172	0.230	0.70	0.554	1.27	1.117	1.146	22.4	0.69	1000	DIMPLED
46-A	2.575	4.232	27.6	253	1.377	6.858	0.204	0.60	0.542	1.25	1.117	1.170	19.3	0.58	1000	DIMPLED
51-A	2.530 x 10 ⁻³	5.105 x 10 ⁻²	27.6	214	1.372 x 10 ⁻³	6.248 x 10 ⁻³	0.219	0.51	0.542	1.37	1.041 x 10 ⁻³	1.160	15.6	0.46	1000	

Table B4. Plane Strain Fracture Toughness - Aluminum Alloy

SPECIMEN IDENTIFICATION	WIDTH (m)	THICKNESS (m)	AREA (m ²)	FAILURE LOAD (kN)	FAILURE STRESS (MN/m ²)	CRACK DEPTH (m)	CRACK LENGTH 2C (m)	$\frac{a}{2C}$	$\frac{C}{\sigma_{ys}}$	$\frac{a}{t}$	Q	$\frac{Q}{(m)}$	M _K	K _Q $\frac{MN}{m^{3/2}\sqrt{m}}$	REMARKS
22-A	5.100 x 10 ⁻²	2.51 x 10 ⁻³	12.8 x 10 ⁻⁵	61.4	478	1.42 x 10 ⁻³	6.65 x 10 ⁻³	0.214	0.99	0.566	1.15	1.24 x 10 ⁻³	1.18	38.6	LN ₂
33-A	5.100	2.573	13.09	62.8	480	1.422	6.604	0.215	0.99	0.533	1.15	1.241	1.17	38.3	LN ₂
34-A	5.100	2.573	13.09	63.1	482	1.498	6.502	0.230	1.00	0.582	1.19	1.270	1.18	39.3	LN ₂
35-A	2.027	2.575	8.192	32.9	401	1.372	6.477	0.212	0.94	0.533	1.17	1.168	1.15	30.9	R.T.
36-A	3.162	2.565	8.128												FAILED
37-A	3.639	2.570	9.354	37.8	402	1.473	7.290	0.202	0.95	0.573	1.14	1.295	1.19	33.8	R.T.
38-A	3.639	2.573	9.354	37.6	401	1.549	7.544	0.205	0.94	0.602	1.15	1.346	1.22	35.0	
39-A	5.105	2.575	13.16	53.6	408	1.473	7.442	0.198	0.95	0.572	1.13	1.295	1.19	34.2	
40-A	5.105	2.575	13.16	53.9	410	1.448	7.035	0.206	0.96	0.562	1.14	1.270	1.18	33.6	
41-A	3.629	2.535	9.225	36.7	399	1.346	6.985	0.193	0.93	0.531	1.12	1.194	1.16	31.3	
42-A	3.627	2.530	9.160	36.7	400	1.372	6.705	0.205	0.94	0.542	1.15	1.194	1.17	31.4	
43-A	4.239	2.575	10.90	44.5	407	1.372	6.823	0.201	0.95	0.533	1.14	1.194	1.16	32.0	
44-A	4.239	2.540	10.77	43.8	407	1.321	6.731	0.196	0.95	0.520	1.12	1.168	1.15	31.3	
45-A	4.232	2.575	10.90	43.8	402	1.448	7.518	0.193	0.94	0.562	1.12	1.295	1.19	33.5	
46-A	3.632	2.568	9.354	37.6	403	1.422	6.807	0.209	0.94	0.554	1.15	1.244	1.18	32.5	
47-A	5.105	2.558	13.03	53.2	407	1.448	7.188	0.201	0.95	0.566	1.14	1.270	1.19	33.6	
48-A	5.108	2.565	13.09	59.9	404	1.397	7.112	0.195	0.95	0.545	1.12	1.244	1.17	32.5	
49-A	5.105 x 10 ⁻²	2.559 x 10 ⁻³	12.90 x 10 ⁻⁵	52.3	405	1.397 x 10 ⁻³	7.645 x 10 ⁻³	0.183	0.95	0.552	1.09	1.270 x 10 ⁻³	1.19	33.6	R.T.

Table B5. Sustained Load Flaw Growth Data - Corrosion Resistant Steel

SPECIMEN IDENTIFICATION	THICKNESS (m)	WIDTH (m)	LOAD (kN)	STRESS σ_i (MN/m ²)	INITIAL CRACK DEPTH a_i (m)	INITIAL CRACK LENGTH $2C_i$ (m)	$\frac{\sigma_i}{2C_i}$	$\frac{\sigma_i}{\sigma_{ys}}$	$\frac{a_i}{t}$	Q_i	$\frac{a_i}{Q_i}$ (m)	M_{KI}	$\frac{K_{II}}{MN \sqrt{m}}$	$\frac{K_{II}}{K_Q}$	TEST LENGTH (mm)	REMARKS
104 ppm HCl																
54-S	2.54 x 10 ⁻³	3.95 x 10 ⁻²	17.8	177	8.64 x 10 ⁻⁴	3.71 x 10 ⁻³	0.233	0.86	0.340	1.25	6.86 x 10 ⁻⁴	1.05	9.6		24	
52-S	2.540	4.445	17.8	156	7.62	3.378	0.226	0.76	0.300	1.27	6.096	1.04	7.8		24	
53-S	2.540	5.080	17.8	138	7.62	3.099	0.246	0.66	0.300	1.35	5.588	1.05	6.6		24	
100 ppm LiCl																
57-S	2.540	3.945	18.7	186	10.16	4.191	0.242	0.90	0.400	1.26	8.128	1.07	11.0		24	
55-S	2.540	4.427	18.7	166	9.14	4.216	0.217	0.80	0.360	1.23	7.366	1.06	9.3		24	
59-S	2.540	5.080	18.7	145	8.64	3.607	0.239	0.70	0.340	1.33	6.604	1.05	7.6		24	
DISTILLED																
61-S	2.520	3.947	18.7	188	7.11	3.810	0.187	0.91	0.282	1.08	6.583	1.04	9.8		24	
62-S	2.557	4.452	18.7	164	7.62	3.911	0.195	0.79	0.298	1.16	6.568	1.04	8.6		24	
63-S	2.568	5.105	18.7	143	7.87	3.861	0.204	0.69	0.307	1.24	6.347	1.04	7.3		24	
104 ppm HCl																
58-S	2.565	3.934	18.7	185	7.87	3.531	0.223	0.89	0.307	1.22	6.350	1.04	9.4		1000	
56-S	2.553	4.440	18.7	165	8.38	3.531	0.237	0.80	0.328	1.29	6.604	1.04	8.6		1000	
60-S	2.560 x 10 ⁻³	5.103 x 10 ⁻²	18.7	143	7.87 x 10 ⁻⁴	3.683 x 10 ⁻³	0.214	0.69	0.308	1.26	6.350 x 10 ⁻⁴	1.04	7.3		1000	

APPENDIX C

The material presented in this appendix covers the test procedure used by JPL in conducting the UDMH fracture mechanics tests at the JPL-Edwards Test Station. This procedure is internal document number 384E-75-1221-TLN, titled, "E-75 Room 3 Fracture Mechanics Test Check List," and dated February 1975.

ETS SECTION 384 PROCEDURE
TITLE SHEET

Reference: 384E-75-1221-TLN

Date: Feb. 5, 1975

Revised: PCDR 1012 3-24-75

TITLE: "E-75" ROOM 3 FRACTURE MECHANICS
TEST CHECK LIST

COGNIZANT TEST ENGINEER: T. L. Nielsen

APPROVALS:

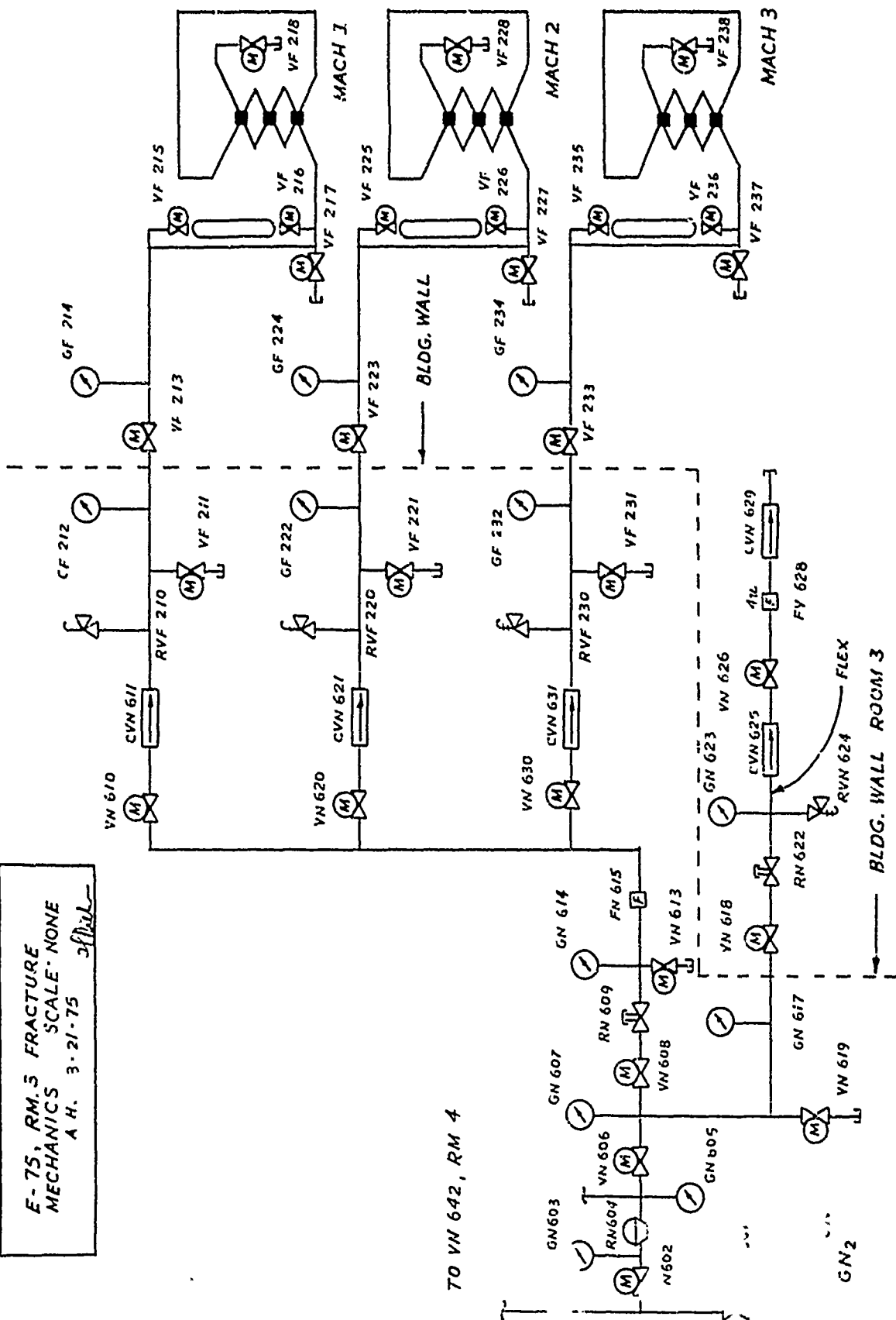
Merle E. Guenther 2/20/75
Liquid Operations Supervisor

L. Toth
Cognizant Engineer - JPL
Pasadena

PRELIMINARY INSTRUCTIONS

1. Normally all sections of this procedure, unless stated, are to be run consecutively for each test. Deletions (NR) or changes of order to this procedure known in advance will be listed on the "Tech" column of this Test Procedure Checklist and initialed by the Cognizant Test Engineer.
2. Any deviations or changes during the performance of this procedure require the Cognizant Engineer's initialed approval and date on a PCDR form prior to the deviation or change. Any PCDR changes will be approved by the L.P.O.S. prior to the next usage of this procedure.
3. All stand operations such as operating hand-valves will be performed by the Stand Mechanic and verified by a second person.
4. All steps in this procedure will be initialed as accomplished by the Stand Mechanic and second person. Each step will be dated and the time of accomplishment noted, except where a section or page is completed in a short time, then the beginning and end of the section or page will be dated and time noted.
5. Care should be taken to assure that contamination or moisture does not enter the system after cleaning, during, and in between tests, by maintaining a positive nitrogen pressure within the system.
6. It is the intent of this procedure to always have a positive flow of Gll_2 from any disconnected line or cup separation after the first test, to exclude the possibility of foreign material entering the propellant system.
7. Aluminum 37° conical seals will be used to seal tube flare fittings unless excepted.

E-75, RM. 3 FRACTURE
MECHANICS SCALE-NONE
A.H. 3-21-75



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DATE & TIME _____

TECH. _____

I. FLUID SYSTEM PRE-CLEANING

1. For new system plumbing, and new cups, clean as follows:
 - a. De-grease in M-17. _____
 - b. Soak in nitric acid solution for one-half (1/2) hour. _____
 - c. Rinse with distilled water. _____
 - d. Soak in hot detergent water for two (2) hours. _____
 - e. Rinse with distilled filtered water. _____
 - f. Rinse with filtered iso-propyl alcohol. _____
 - g. Vacuum dry at 160°F for one (1) hour minimum. _____
2. After new system is installed and leak checked, passivate, flush, and dry as follows. Also perform prior to any basic fluid change.
 - a. Confirm the new fluid is compatible with the prior history of the system, if not, do not proceed. _____
 - b. Passivate per Section III and IV of this procedure using simulated specimens with a 10 lb. weight (200 lb. load) and soak for a duration of three (3) hours. _____
 - c. After the final iso-propyl alcohol flush of this passivation procedure, a fifteen (15) minute GN₂ purge followed by vacuum drying the system to 100 micron for one (1) hour is required. _____
 - d. Upon completion of this initial system preparation a positive pressure, or positive purge, of the system will be practiced. _____

II. MACHINE AND ROOM PRE-TEST CHECKS

1. Install the load cell and confirm the machine arm is balanced with one clevis below load cell.
Mach. 1 _____ Mach. 2 _____ Mach. 3 _____
2. Connect clevis train with solid rod and lower weights until they swing free and check once with SR -4 Box.
Mach. 1 _____ Mach. 2 _____ Mach. 3 _____

DATE & TIME _____

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DATE & TIME _____

TECH.

3. Adjust weights until the SR-4 Box is nulled indicating the requested load. Check counterbalance weights to confirm a ratio of 20.

Mach. 1 _____ Mach. 2 _____ Mach. 3 _____

4. Lift weights an- disconnect train at center and confirm zero.

5. Remove load cell and replace with rod which has the same weight as the the load cell used for the above steps.

Mach. 1 _____ Mach. 2 _____ Mach. 3 _____

DATE & TIME _____

DATE & TIME _____

TECH.

III. INSTALLATION AND LEAK TEST OF SPECIMENS

A. Machine No. 1

1. Install the specimens for Machine No. 1 per the test record. For specimens with 5/8" holes install shims and bushings. _____
2. Verify weights for Machine No. 1 are per test record. _____
3. Lower weight-platform on Machine No. 1 until there is a $1/2" \pm 3/8"$ gap. _____
4. Confirm the testing machine arm is level, if not estimate amount long arm is from level _____ inches. _____
5. Lift weights, make adjustment of train bottom nut to make bar level. 1/2 turn of nut equals approximately 1" of height change of long arm. _____
6. Lower weights to preload specimens to approximately 100 ± 50 lbs. _____
7. Open isolation valve VN618. _____
8. Adjust flex line purge inside cell to 20 ± 10 psi on Gauge GN623. _____
9. Open purge valve VN626. _____
10. Open purge Valve VN627 and connect purge line to Valves VF217 and VF218 on Machine No. 1. _____
11. Adjust hand loader RN609 to 20 ± 10 psi on Gauge GN614 and open Valves VN610 and VF213 to read pressure on Gauge GF214. _____
12. Weigh Propellant Cylinder No. _____ on the scale in E-8 shop _____ lbs. _____
13. Instr'l propellant cylinder in Machine No. 1 confirm the 0 ± 10 psi at both ends to exclude any air from system. _____
14. Install cups over the test specimens one set at a time. The 20 ± 10 psi purge will be flowing on both halves. _____

DATE & TIME _____

DATE & TIME _____

TECH.

15. Center the cups over crack and torque the cup bolts to 40 ± 5 in-lbs. maintaining the gap between the two cups the same at both bolt locations. Use drill rod as go-no go gages to check this gap. _____
16. Increase Hand Loader RN609 to 325 ± 25 psi on Gauge GF212. _____
17. Increase Hand Loader RN622 to 325 ± 25 psi on Gauge GN623. _____
18. Soap leak check the fittings on Machine No. 1 and confirm no leaks. _____
19. Close Valves VF217 and VF218. _____
20. Reduce Hand Loader RN622 to 20 ± 10 psi on Gauge GN623. _____
21. Reduce Hand Loader RN609 to 20 ± 10 psi on Gauge GN610. _____
22. Disconnect and cap flex line at VF217 and VF218. _____

B. Machine No. 2

1. Install the specimens for Machine No. 2 per the test record. For specimens with $5/8$ " holes install shims and bushing. _____
2. Verify weights for Machine No. 2 are per test record. _____
3. Lower weight-platform on Machine No. 2 until there is a $1/2$ " $\pm$ $3/8$ " gap. _____
4. Confirm the testing machine arm is level, if not estimate amount long arm is from level _____ inches. _____
5. Lift weights, make adjustment of train bottom nut to make bar level. $1/2$ turn of nut equals approximately 1" of height change of long arm. _____
6. Lower weights to preload specimens to approximately 100 ± 50 lbs. _____

DATE & TIME _____

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DATE & TIME _____

TECH.

7. Open isolation valve VN618. _____
8. Adjust flex line purge inside cell to 20 ± 10 psi on Gauge GN 623. _____
9. Oper: purge Valve VN 626. _____
10. Open Purge Valve VN 627 and then connect purge line to Valves VF227 and VF228 on Machine No. 2. _____
11. Adjust hand loader RN609 to 20 ± 10 psi on Gauge GN614 and open Valves VN620 and VF223 to read pressure on Gauge GF224. _____
12. Weigh Propellant Cylinder No. _____ on the scale in E-8 shop. _____
13. Install propellant cylinder in Machine No. 2. Confirm the 20 ± 10 psi at both ends to exclude any air from system. _____
14. Install cups over the test specimens one set at a time. The 20 ± 10 psi purge will be flowing on both halves. _____
15. Center the cups over crack and torque the cup bolts to 40 ± 5 in-lbs. maintaining the gap between the two cups the same at both bolt locations. Use drill rod as go-no go gages to check this gap. _____
16. Increase Hand Loader RN609 to 325 ± 25 psi on Gauge GF222. _____
17. Increase Hand Loader RN622 to 325 ± 25 psi on Gauge GN623. _____
18. Soap leak check the fittings on Machine No. 2 and confirm no leaks. _____
19. Close Valves VF227 and VF228. _____
20. Reduce Hand Loader RN622 to 20 ± 10 psi on Gauge GN623. _____
21. Reduce Hand Loader RN609 to 20 ± 10 psi on Gauge GN610. _____
22. Disconnect and cap flex line at VF227 and VF228. _____

DATE & TIME _____

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DATE & TIME _____

TECH.

C. Machine No. 3

1. Install the specimens for Machine No. 3 per the test record. For specimens with 5/8" holes install shims and bushings. _____
2. Verify weights for Machine No. 3 are per test record. _____
3. Lower weight-platform on Machine No. 3 until there is a 1/2" $\pm$ 3/8" gap. _____
4. Confirm the testing machine arm is level, if not estimate amount long arm is from level _____ inches. _____
5. Lift weights, make adjustment of train bottom nut to make bar level. 1/2 turn of nut equals approximately 1" of height change of long arm. _____
- 6.1 Lower weights to preload specimens to approximately 100 $\pm$ 50 lbs. _____
7. Open isolation Valve VN618. _____
8. Adjust flex line purge inside cell to 20 $\pm$ 10 psi on Gauge GN623. _____
9. Open Purge Valve VN626. _____
10. Open Purge Valve VN627 and then connect purge line to Valves VF237 and VF238, Machine No. 3. _____
11. Adjust purge to 20 $\pm$ 10 psi on Gauge GN614, and open Valves VN630 and VF233 to read pressure on Gauge GF234. _____
12. Weigh Propellant Cylinder No. _____ on the scale in E-8 shop _____ lbs. _____
13. Install propellant cylinder in Machine No. 3. Confirm the 20 $\pm$ 10 psi at both ends to exclude any air from system. _____
14. Install cups over the test specimens one set at a time. The 20 $\pm$ 10 psi purge will be flowing on both halves. _____

DATE & TIME _____

DATE & TIME _____

TECH.

15. Center the cups over crack and torque the cup bolts to 40 $\pm$ 5/-0 in-lbs. maintaining the gap between the two cups the same at both bolt locations. Use drill rod as go-no gages to check this gap. _____
16. Increase hand loader RN609 to 325 $\pm$ 25 psi on gauge GF232. _____
17. Increase hand loader RN622 to 325 $\pm$ 25 psi on gauge GN623. _____
18. Soap leak check the fittings on machine No. 3 and confirm no leaks. _____
19. Close valves VF237 and VF238. _____
20. Reduce hand loader RN622 to 20 $\pm$ 10 psi on gauge GN623. _____
21. Reduce hand loader RN609 to 20 $\pm$ 10 psi on gauge GN614. _____
22. Disconnect and cap flex line at valve VF237 and VF238. _____

DATE & TIME _____

DATE & TIME _____

TECH.

IV. PROPELLANT PRIME OF SPECIMENS

A. Machine No. 1

1. Wrap the specimens in machine No. 1 with plastic to eliminate splatter in event of a leak. _____
2. Set a catch pan with water beneath machine No. 1 to catch and dilute any leaks of propellant. _____
3. Confirm gauge GN614 is 20 ± 10 psi and valves VN610 and VF213 are open. _____
4. Connect the propellant prime drain line to valve VF218 and terminate it in a breaker outside of test cell. _____
5. Open VF218 one half turn off seat. _____
6. Open top cylinder valve on machine 1. (VF215). _____
- *7. Open bottom cylinder on machine 1. (VF216). _____
- *8. When propellant is flowing without gas at the prime drain line close VF218 & VF216. _____
- *9. Carefully disconnect the propellant line from valve VF218. _____
- *10. Cap lines. _____

*Face shields, whitesides and gloves shall be worn for minimum protection by both personnel.

B. Machine No. 2

1. Wrap the specimens in machine No. 2 with plastic to eliminate splatter in event of a leak. _____
2. Set a catch pan with water beneath machine No. 2 to catch and dilute any leaks of propellant. _____
3. Confirm gauge GN614 is 20 ± 10 psi and valves VN620 and VF223 are open. _____

DATE & TIME _____

DATE & TIME _____

TECH.

4. Connect the propellant prime drain line to valve VF228 and terminate it in a beaker outside of test cell. _____
5. Open VF228 one half turn off seat. _____
6. Open top cylinder valve on machine 2. (VF225). _____
- *7. Open bottom cylinder on machine 2. (VF226). _____
- *8. When propellant is flowing without gas at the prime drain line close VF228 & VF226. _____
- *9. Carefully disconnect the propellant line from valve VF228. _____
- *10. Cap lines. _____

*Face shields, whitesides and gloves shall be worn for a minimum protection by both personnel.

C. Machine No. 3

1. Wrap the specimens in machine No. 3 with plastic to eliminate splatter in event of a leak. _____
2. Set a catch pan with water beneath machine No. 3 to catch and dilute any leaks of propellant. _____
3. Confirm gauge GN614 is 20 ± 10 psi and valves VN630 and VF233 are open. _____
4. Connect the propellant prime drain line to valve VF238 and terminate it in a beaker outside of test cell. _____
5. Open VF238 one half turn off seat. _____
6. Open top cylinder valve on machine 3. (VF235). _____
- *7. Open bottom cylinder on machine 3. (VF236). _____
- *8. When propellant is flowing without gas at the prime drain line close VF238 & VF236. _____
- *9. Carefully disconnect the propellant line from valve VF238. _____

DATE & TIME _____

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DATE & TIME _____

TECH.

*10. Cap lines.

*Face shields, whitesides and gloves shall be worn
for minimum protection by both personnel.

DATE & TIME _____

DATE & TIME _____

TECH.

V. TEST

- *1. Lower weights on machine No. 3 until there is a $1/2 \pm 1/4$ " gap under weight platform. _____
 a. Load cell machine No. 3 _____
 b. Record time and date _____.
- *2. Lower weights on machine No. 2 until there is a $1/2 \pm 1/4$ " gap under weight platform. _____
 a. Read load cell machine No. 2 _____.
 b. Record time and date _____.
- *3. Lower weights on machine No. 1 until there is a $1/2 \pm 1/4$ " gap under weight platform. _____
 a. Read load cell machine no. 1 _____.
 b. Record time and date. _____.
- 4. Close door. _____
- 5. Confirm temperature is proper. _____
- *White sides, face shield, and gloves required for both personnel.
- 6. Increase regulator RN609 to test pressure of 300 ± 10 psig on gauge GN614 and confirm same pressure on gauges GF212, GF222 and GF232. _____
- 7. Close valves VN610, VN620 and VN630 each test cylinder. _____
- 8. Periodically check the specimen and record the following information:

DATE	TIME	<u>GAUGE PRESSURE (PSIG)</u>			<u>PLATFORM GAP INCHES</u>		<u>ROOM TEMPERATURE</u>
		F-212 1**	F-222 2**	F-232 3**	1	3	(°F)
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____

**Machine number

DATE & TIME _____

DATE & TIME _____

TECH.

VI. TEST CONCLUSION

1. Reduce handloader RN609 to 20 ± 10 psi on gauge GN614. _____
2. Reduce machine No. 1 pressure 20 ± 10 psi by opening and closing valve VF211. _____
3. Reduce machine No. 2 pressure to 20 ± 10 psi by opening and closing valve VF221. _____
4. Reduce machine No. 3 pressure to 20 ± 10 psi by opening and closing valve VF231. _____
5. Enter room and close VF215 and VF216 cylinder valves on machine No. 1. _____
6. Close VF225 and VF226 cylinder valves on machine No. 2. _____
7. Close VF235 and VF236 cylinder valves on machine No. 3. _____
8. Lift weights on machine No. 1 and record time and date _____. _____
9. Lift weights on machine No. 2 and record time and date _____. _____
10. Lift weights on machine No. 3 and record time and date _____. _____

DATE & TIME _____

DATE & TIME _____

TECH.

VII. DECONTAMINATION AND SECURING

A. Machine No. 1

1. Connect the propellant drain line to valve VF 217. _____
2. Connect the purge line and an isopropyl alcohol flush container to valve VF 218. _____
3. Open valve VF 217 and VF 218 and purge the liquid out the drain line while flooding the drain exhaust with tap water. Continue to flow until effluent appears dry. _____
4. Alternate slug the specimen cups with isopropyl alcohol and then purge dry until 6 cycles have been accomplished to fully decontaminate the specimens. _____
5. Disconnect the isopropyl alcohol flush container and drain line and connect the purge into valve VF210. _____
6. Remove the propellant cylinder and cap lines. _____
7. Install the cylinder for the following test If it is available at this time. _____
8. Remove the specimens one at a time while purging at both cups. _____
9. If new seals are required, install if new specimens are being installed. _____
10. If specimens are not being installed, clamp the cup valves together to 40 ± 5 in-lbs. to seal. _____
11. Wrap the specimens individually in micro wipes without touching the surface, and insert them in plastic bags. _____
12. Log all pertinent information on the test record. _____
13. Weigh the propellant cylinder with metal caps on fittings. _____

DATE & TIME _____

DATE & TIME _____

TECH.

14. Ship the propellant cylinders as requested. _____
15. Ship the specimens to JPL. _____
- B. Machine No. 2
 1. Connect the propellant drain line to valve VF227. _____
 2. Connect the purge line and an iso-propyl alcohol flush container to Valve VF228. _____
 3. Open valve VF227 and VF228 and purge the liquid out the drain line while flooding the drain exhaust with tap water. Continue to flow until effluent appears dry. _____
 4. Alternately slug the specimen cups with iso-propyl alcohol and then purge dry until 6 cycles have been accomplished to fully decontaminate the specimens. _____
 5. Disconnect the iso-propyl alcohol flush container and drain line and connect the purge into valve VF228. _____
 6. Remove the propellant cylinder and cap lines. _____
 7. Install the cylinder for the following test if it is available at this time. _____
 8. Remove the specimens one at a time while purging at both cups. _____
 9. If new seals are required, install if new specimens are being installed. _____
 10. If specimens are not being installed, clamp the cup valves together to 40 ± 5 in-lbs. to seal. _____
 11. Wrap the specimens individually in micro wipes without touching the surface, and insert them in plastic bags. _____
 12. Log all pertinent information on the test record. _____
 13. Weigh the propellant cylinder with metal caps on fittings. _____

DATE & TIME _____

DATE & TIME _____

TECH.

14. Ship the propellant cylinder as requested. _____

15. Ship the specimens to JPL. _____

C. Machine No. 3

1. Connect the propellant drain line to valve VF237. _____

2. Connect the purge line and an iso-propyl alcohol flush container to Valve VF238. _____

3. Open valve VF237 and VF238 and purge the liquid out the drain line while flooding the drain exhaust with tap water. Continue to flow until effluent appears dry. _____

4. Alternately slug the specimen cups with iso-propyl alcohol and then purge dry until 6 cycles have been accomplished to fully decontaminate the specimens. _____

5. Disconnect the iso-propyl alcohol flush container and drain line and connect the purge into valve VF238. _____

6. Remove the propellant cylinder and cap lines. _____

7. Install the cylinder for the following test if it is available at this time. _____

8. Remove the specimens one at a time while purging at both cups. _____

9. If new seals are required, install if new specimens are being installed. _____

10. If specimens are not being installed, clamp the cup valves together to 40 ± 5 in-lbs. to seal. _____

11. Wrap the specimens individually in micro wipes without touching the surface, and insert them in plastic bags. _____

12. Log all pertinent information on the test record. _____

13. Weigh the propellant cylinder and fittings. _____

DATE & TIME _____

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DATE & TIME _____

TECH.

- 14. Ship the propellant cylinder as requested. _____
- 15. Ship the specimens to JPL. _____

DATE & TIME _____

GLOSSARY

<u>Term or Symbol</u>	<u>Definition or Identification</u>
a	semiminor axis of the ellipse $x^2/c^2 + y^2/a^2 = 1$ or crack depth of the semielliptical surface flaw, cm (1 cm = 0.394 in.)
2c	crack length of the semielliptical flaw, cm (in.)
Cl ⁻	chloride ion
HCl	hydrochloric acid
K _I	plane-strain stress-intensity factor, $\frac{\text{MN}}{\text{m}^2} \sqrt{\text{m}} \text{ (KSI } \sqrt{\text{in.}}),$ $\left[1.099 \frac{\text{MN}}{\text{m}^2} \sqrt{\text{m}} \approx 1 \text{ KSI } \sqrt{\text{in.}} \right]$
K _{Ii}	plane-strain stress-intensity factor at initial conditions, $\frac{\text{MN}}{\text{m}^2} \sqrt{\text{m}} \text{ (KSI } \sqrt{\text{in.}})$
K _Q	critical experimental stress-intensity factor or fracture toughness for a specified thickness of a given material, $\frac{\text{MN}}{\text{m}^2} \sqrt{\text{m}} \text{ (KSI } \sqrt{\text{in.}})$
M _K	stress-intensity magnification factor for deep surface flaws based upon Kobayashi's solution
Q	flaw-shape parameter $= \phi^2 - 0.212 (\sigma/\sigma_{ys})^2$
t	thickness of plate or specimen, cm (in.)

σ uniform gross stress applied remote from crack and perpendicular to plane of crack,

$$\frac{\text{MN}}{\text{m}^2} \text{ (KSI)},$$

$$\left[6.895 \text{ MN/m}^2 = 1 \text{ KSI} \right]$$

σ_{ult} or F_{t_u} ultimate strength of the material,

$$\frac{\text{MN}}{\text{m}^2} \text{ (KSI)}$$

σ_{y_s} or F_{t_y} uniaxial tensile yield strength of the material,

$$\frac{\text{MN}}{\text{m}^2} \text{ (KSI)}$$

ϕ complete elliptical integral of the second kind having modulus k defined as

$$k = (1 - a^2/c^2)^{1/2}$$

Subscripts

cr at critical conditions

i at initial condition

Abbreviation

AFRPL Air Force Rocket Propulsion Laboratory (United States)

ASTM American Society for Testing and Materials

Al aluminum

CRES corrosion resistant steel

EDM electrical discharge machining

ETS Edwards Test Station (JPL)

hr hour

Hz hydrazine

IPA	isopropyl alcohol
JPL	Jet Propulsion Laboratory (California Institute of Technology)
NASA-MSFC or MSFC	National Aeronautics and Space Administration , Marshall Space Flight Center
ppm	parts per million
PTC	part-through crack
SEM	scanning electron microscope
TFE	tetrafluoroethylene
Ti	titanium
UDMH	unsymmetrical dimethylhydrazine or uns-dimethylhydrazine