FR-8971
SEPTEMBER 1977

MECHANICAL PROPERTIES OF SEVERAL NICKEL ALLOYS IN HYDROGEN AT ELEVATED TEMPERATURES

FINAL REPORT

Contract NAS8-30744
Exhibit D
National Aeronautics and Space Administration
George C. Marshall Space Flight Center

(NASA-CR-150412) MECHANICAL PROPERTIES OF SEVERAL NICKEL ALLOYS IN HYDROGEN AT ELEVATED TEMPERATURES Final Report (Pratt and Whitney Aircraft Group) 66 p HC A04/MF A01 CSCL 11F G3/26
N77-33315
Unclas
49476

PRATT & WHITNEY AIRCRAFT GROUP
Government Products Division
P. O. Box 2691
West Palm Beach, Florida 33402

UNITED TECHNOLOGIES ®

30

Printed in the United States of America

FR-8971
SEPTEMBER 1977

MECHANICAL PROPERTIES OF SEVERAL NICKEL ALLOYS IN HYDROGEN AT ELEVATED TEMPERATURES

FINAL REPORT

Contract NAS8-30744
Exhibit D
National Aeronautics and Space Administration
George C. Marshall Space Flight Center

John R Warren
John R. Warren
Principal Investigator

John A. Harris Jr.
John A. Harris Jr.
Program Manager

M.C. VanWanderham
M. C. VanWanderham
Mgr Mechanics of
Materials and Structures

PRATT & WHITNEY AIRCRAFT GROUP
Government Products Division
P. O. Box 2691
West Palm Beach, Florida 33402

UNITED
TECHNOLOGIES ®

Printed in the United States of America

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

TABLES

SECTION I
INTRODUCTION

This report is submitted in accordance with the requirements of Contract NAS8-30744 and represents a final report covering work performed under Exhibit D — "Mechanical Properties of Several Nickel Alloys In Hydrogen at Elevated Temperatures." The Exhibit A effort of this contract, covering properties of Incoloy 903 in various heat-treat and welded conditions was documented in Pratt & Whitney Aircraft Report FR-7175, "Influence of Gaseous Hydrogen on the Mechanical Properties of Incoloy 903," September 1975. Exhibits B and C of this contract, covering various mechanical properties of high-temperature alloys in gaseous hydrogen at elevated temperatures, were documented in Pratt & Whitney Aircraft Report FR-7746, "Influence of Gaseous Hydrogen on the Mechanical Properties of High-Temperature Alloys," July 1976.

The objective of this phase of the program was to obtain low-cycle fatigue and crack propagation properties of Incoloy 903 and MAR-M-246. These alloys are proposed for use in space propulsion systems in pure or partial high-pressure hydrogen environments at different temperatures. The specific environments included air, hydrogen, and hydrogen and water vapor at 34.5 MN/m² (5,000 psig).

After the initiation of the Exhibit D test program, rocket propulsion system material problems developed. Consequently the test plan was modified to include the investigation of specific system operating conditions. In order to remain within the contract funding restraints other testing was abbreviated or eliminated. Therefore, some areas of investigation and analysis were limited, and all questions which arose could not be pursued completely.

The overall Exhibit D test program including types, conditions, and numbers of tests conducted, is outlined in table I-1. The primary goal of these tests was to determine the fatigue and crack growth rate properties of Incoloy 903 and MAR-M-246 (Hf modified) in hydrogen and hydrogen-rich steam at elevated temperatures.

All testing was conducted on solid specimens exposed to external gaseous pressure. Specific mechanical properties determined and the testing methods used are:

1. Low-Cycle Fatigue - Low-cycle fatigue life was established by constant total strain testing using smooth specimens and a closed-loop test machine.
2. Crack Growth - Crack growth rate properties were determined using specimen configuration and testing techniques, where applicable, according to ASTM E399-74.

This report is arranged in sections that cover the program conclusions, material tested, and results and conclusions of the individual property tests. It includes the Incoloy 903 and MAR-M-246 (Hf modified) information covered in the monthly progress reports previously issued under the contract.

The International System of Units (SI) is used as the primary system of units for reporting specimen and test parameters and results. Customary English units are included in parenthesis following the SI units, or in separate columns in data tables. The customary system of units was used for the principal measurements and calculations and results converted to SI units for reporting purposes.

Table I-1. Exhibit "D" Experimental Test Outline for Incoloy 903 and MAR-M-246 (Hf Modified)

			Test Conditions					Type and Number of Tests	
			Test Temperature		Pressure				
Material	Form	Condition[1]	°K	°F	MN/m²	psig	Test Environment[2]	Low-Cycle Fatigue (LCF)	Crack Growth Rate (CGR)
Incoloy 903	Forging	Wrought + STA-2	589	600	34.5	5000	$GH_2 + H_2O$	1	
			755	900	34.5	5000	$GH_2 + H_2O$	2	
			977	1300	34.5	5000	$GH_2 + H_2O$	3	
			1033	1400	34.5	5000	$GH_2 + H_2O$	12	
			1033	1400	One Atmosphere		Air	2	
Incoloy 903	Forging	TMP + STA-1	644	700	34.5	5000	$GH_2 + H_2O$	3	
			811	1000	31.0	4500	$GH_2 + H_2O$	3	
			922	1200	34.5	5000	$GH_2 + H_2O$	3	
Incoloy 903	Forging	As Welded	811	1000	22.4	3250	$GH_2 + H_2O$	1	
			977	1300	34.5	5000	$GH_2 + H_2O$	3	
Incoloy 903	Forging	Welded + 718 STA	644	700	22.4	3250	$GH_2 + H_2O$	1	
			977	1300	34.5	5000	$GH_2 + H_2O$	3	
Incoloy 903	Forging	EB Welded + STA-2	755	900	26.9	3900	$GH_2 + H_2O$	1	
Incoloy 903	Forging	STA-2	811	1000	34.5	5000	GH_2		4
			922	1200	34.5	5000	GH_2		4
			922	1200	34.5	5000	$GH_2 + H_2O$		4
MAR-M-246 (Hf Modified)	Conventionally Cast	CC	811	1000	34.5	5000	GH_2		4
			1144	1600	34.5	5000	GH_2		4
			1144	1600	34.5	5000	$GH_2 + H_2O$		4
MAR-M-246 (Hf Modified)	Directionally Solidified	DS	811	1000	34.5	5000	GH_2		2
			1144	1600	34.5	5000	GH_2		2
			1144	1600	34.5	5000	$GH_2 + H_2O$		3

---

[1] Condition:

STA-2 — 1227°K (1750°F) 1 hr, AC + 991°K (1325°F) 8 hr + 894°K (1150°F) 8 hr (Total Age Time = 18 hr), AC

STA-1 — 1130°K (1575°F) 1 hr, AC + 991°K (1325°F) 8 hr + 894°K (1150°F) 8 hr (Total Age Time = 18 Hr), AC

As welded — Preweld STA-2 HT per above + GTA (gas tungsten arc) weld

Welded + 718 STA — GTA weld + 1311°K (1900°F) 20 min, AC + 1033°K (1400°F) 10 hr + 922°K (1200°F) for Total Age Time = 20 hr, AC

EB Welded + STA-2 — EB (electron beam) weld + STA-2 HT per above

TMP + STA-1 — Finish Forging Operation (Final 40/50% reduction) performed below 1144°K (1600°F) — (Thermo-mechanical Processing) + STA-1 HT per above

CC — As cast

DS — 1494°K (2230°F) 2 hr, FC + 1144°K (1600°F) 24 hr, FC

[2] Environment:

$GH_2 + H_2O$ — 50% gaseous hydrogen + 50% water vapor by weight

Air — Air at atmospheric pressure

GH_2 — Gaseous hydrogen (with oxygen content less than 1 part per million)

This program was conducted using the Program Manager - Project Group System by the Pratt & Whitney Aircraft Group, Government Products Division, Materials and Mechanics Technology Laboratory, under the cognizance of Mr. W. B. McPherson, Metallurgy Branch, Materials & Process Laboratory, Marshall Space Flight Center. John A. Harris, Jr. was the Pratt & Whitney Program Manager for the effort.

Acknowledgement is given to the following personnel of the Project Group.

J. R. Warren — Principal Investigator
D. B. Granda — Crack Growth Rate Mechanical Tests
J. M. Lieske II — Low-Cycle Fatigue Mechanical Tests
D. L. Pearson — Test Stand Operations
M. W. Ridler — Proposal and Report Preparation
E. I. Veil — Metallurgical Investigations
M. L. Zaccagnino — Proposal and Report Preparation

SECTION II
RESULTS AND CONCLUSIONS

A. GENERAL

Efforts in this program consisted of testing to determine Low-Cycle Fatigue (LCF) and crack growth rate properties of one iron-base alloy and two forms of one cast nickel-base alloy. The alloys were tested in various forms and/or heat-treat conditions that are proposed for use in a high-pressure hydrogen or a hydrogen-water vapor environment. Environmental degradation of properties could not be established due to the absence of comparable testing in an inert atmosphere, however some general conclusions can be made comparing the results of tests in the hydrogen environment with those in the hydrogen-water vapor environment.

Detailed conclusions are presented in the various sections pertaining to types of tests. General results and conclusions are presented below.

B. LOW-CYCLE FATIGUE

Strain vs cyclic life for Incoloy 903 in various parent and welded conditions were generated at 644°K, 755°K, 811°K, 922°K, 977°K, and 1033°K (700°F, 900°F, 1000°F, 1200°F, 1300°F, and 1400°F respectively) for cyclic and/or cyclic dwell conditions.

Dwell (hold) time at the maximum compressive strain had a significant effect on the fatigue life of wrought Incoloy 903 at 1033°K (1400°F), with lowest life occurring at the longer dwell times. Investigation of the effect of dwell was limited and only conducted at 1033°K (1400°F).

Forging chemical composition had some effect on Incoloy 903 at 1033°K (1400°F). The forging having slightly lower carbon and nickel content had consistently greater LCF life. Additional testing however, may show this difference to be within normal data scatter.

The hydrogen-water vapor environment caused a 50% average reduction in fatigue life, indicating extreme degradation when compared with tests conducted in air, for Incoloy 903 at 1033°K (1400°F).

There was no significant difference between the LCF life of wrought Incoloy 903 and welded + 718 STA Incoloy 903 at 977°K (1300°F). However, the as-welded Incoloy 903 had slightly less LCF life in comparison with the wrought material at these conditions.

C. CRACK GROWTH

Crack growth rate curves (da/dn vs ΔK) were generated for Incoloy 903 at 811°K (1000°F) and 922°K (1200°F), and for MAR-M-246 (Hf modified) in the conventionally cast (CC) and directionally solidified (DS) forms at 811°K (1000°F) and 1144°K (1600°F).

Crack growth rates increased significantly for all materials with increasing test temperature. A very significant increase (three orders of magnitude) in crack growth rate occurred for Incoloy 903 tested in the hydrogen-water vapor environment when compared with testing done in hydrogen alone at 922°K (1200°F). The hydrogen-water vapor environment had less effect on MAR-M-246 DS material and almost no effect on MAR-M-246 CC material at 1144°K (1600°F), as compared to hydrogen alone.

SECTION III
MATERIALS AND SPECIMENS

A. TEST MATERIAL

The purpose of this program was to determine the low-cycle fatigue and crack growth properties of one iron-base alloy and two nickel-base alloys in high-pressure hydrogen environments. Testing evaluated these materials in various forms and heat-treat conditions. All test materials were supplied in the fully heat-treated condition by the Marshall Space Flight Center (MSFC). Table III-1 lists the various materials, their form and heat-treat conditions, and the types of tests performed. Chemical compositions are listed in table III-2, and typical microstructures and macrostructures are shown in figures III-1 through III-3.

B. TEST GASES

Hydrogen and hydrogen-water vapor were used during the testing of specimens, and nitrogen was used as a preliminary purge gas. Propellant grade hydrogen was provided under Military Specification P-27201, which requires the gas to have an oxygen content of less than 1 part per million. Analysis verified the gas to be of this purity. The hydrogen gas was used to provide the test environments. The hydrogen and water vapor environment was obtained by utilizing triple distilled water and a retort system so the water was vaporized by furnace heat while maintaining the specified pressure.

Gas handling systems supplying the test vessels were equipped to enable sampling before and after specimen tests. The hydrogen was sampled extensively, both dry and saturated with water vapor (wet hydrogen was dried prior to analysis). Samples were analyzed with a gas chromatograph with accuracy in the parts per billion range. No appreciable difference was noted between pretest and post-test samples, indicating no gas contamination by the test rig and/or test itself.

C. TEST SPECIMENS

All specimens were machined by the Pratt & Whitney Aircraft Group, Materials Control Laboratory Machine Shop and finished to an average roughness of 16 μ-in. rms, or less. LCF specimens were machined from supplied round specimen blanks. Crack growth specimens were machined from pancake forgings (Incoloy 903) and from cast or directionally solidified plate material (MAR-M-246). Specimens were oriented in the Incoloy 903 pancake forgings to determine crack growth properties in the radial direction. Specimens for the directionally solidified MAR-M-246 (Hf modified) material were oriented to determine crack growth properties parallel to the primary grain direction (see figure III-2).

A typical set of specimens is listed in table III-3 and shown in figure III-4. Specimen prints are presented in figures III-5 through III-7. (Note: All dimensions on specimen prints are in inches).

Table III-1. *Government Furnished Materials Used to Determine Low-Cycle Fatigue and Crack Growth Rate Properties of Various Alloys in High-Pressure Hydrogen Environments*

Material	*Form*	*Vendor*	*Heat No.*	*As Tested Condition (Heat Treatment)*	*Type Test*[1]
Incoloy 903	Forging — Flat Disk	Huntington Alloy	HH24A9UK	Wrought STA-2 1227°K (1750°F), 1 hr, AC + 991°K (1325°F), 8 hr + 894°K (1150°F) 8 hr (with Total Age = 18 hr) + AC.	LCF
Incoloy 903	Forging — Flat Disk	Huntington Alloy	HH22A9UY	Wrought STA-2 (same HT as above)	LCF
Incoloy 903	Forging — Ring	Huntington Alloy	HH36A5UK	TMP + STA-1 Finish Forging Operation (Final 40/50% reduction) performed below 1144°K (1600°F) + 1130°K (1575°F), 1 hr AC + 991°K (1325°F), 8 hr + 894°K (1150°F), 8 hr (with Total Age = 18 hr) + AC	LCF
Incoloy 903	Forging — Flat Disk	Huntington Alloy	HH22A9UY	As Welded[2] 1227°K (1750°F) 1 hr, AC + 991°K (1325°F), 8 hr + 894°K (1150°F), 8 hr, (with Total Age = 18 hr), + AC + GTA weld	LCF
Incoloy 903	Forging — Flat Disk	Huntington Alloy	HH36A2UK	Welded + 718 STA GTA welded + 1311°K (1900°F), 20 min. + AC, + 1033°K (1400°F), 10 hr, + 922°K (1200°F) for Total Age = 20 hr, + AC.	LCF
Incoloy 903	Forging — Flat Disk	Huntington Alloy	HH22A9UY	EB Welded + STA-2 EB welded + 1227°K (1750°F) 1 hr, AC + 991°K (1325°F) 8 hr, + 894°K (1150°F) 8 hr, (with Total Age = 18 hr) + AC	LCF
Incoloy 903	Forging — Flat Disk	Carpenter Technology	91178	STA-2 1227°K (1750°F) 1 hr, AC + 991°K (1325°F) 8 hr, + 894°K (1150°F) 8 hr, (with Total Age = 18 hr) + AC	CGR
MAR-M-246 (Hf Modified)	Conventionally Cast	Austenal	L99-HBE	As Cast	CGR
MAR-M-246 (Hf Modified)	Directionally Solidified	Austenal	DE-002	1494°K (2230°F) 2 hr, FC + 1144°K (1600°F) 24 hr, FC	CGR

---

[1] Types of Tests:
LCF — Low-Cycle Fatigue
CGR — Crack Growth Rate

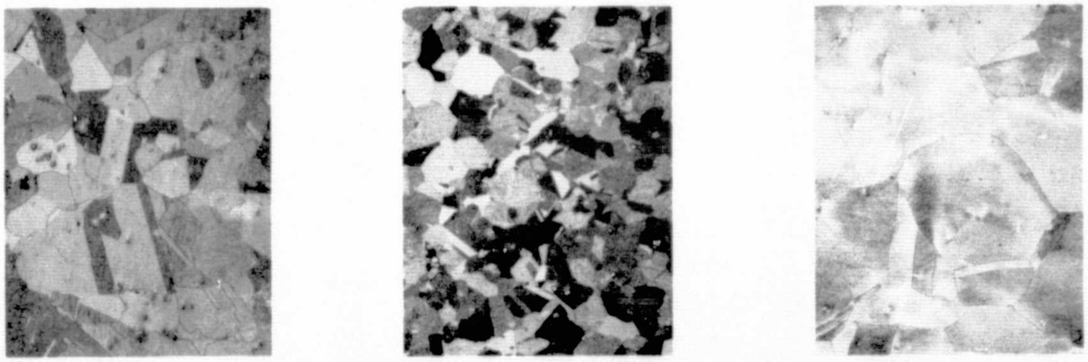
[2] Heat Treatment was prior to weld operation.

Table III-2. Chemical Composition of Materials Used to Determine Low-Cycle Fatigue and Crack Growth Rate Properties of Various Alloys High-Pressure Hydrogen Environments

			Elemental Composition — % By Weight																		
Material	*Form*	*Heat No.*	*C*	*Si*	*Mn*	*P*	*S*	*Cr*	*Ni*	*Mo*	*Al*	*Ti*	*Cu*	*Fe*	*Cb+Ta*	*Ta*	*Co*	*W*	*Zr*	*B*	*Hf*
Incoloy 903	Forging	HH24A9UK	0.03	0.12	0.05		0.004	0.10*	38.06		0.91	1.67	0.09*	40.76	2.86		15.42			0.007*	
		HH22A9UY	0.04	0.18	0.14		0.004	0.01*	38.44		0.94	1.37	0.02*	40.80	2.96		15.11			0.008*	
		HH36A5UK	0.03	0.15	0.17		0.002	0.09	38.43		0.74	1.33	0.07	40.73	3.04		15.38			0.007	
		HH36A2UK	0.03	0.16	0.17		0.044	<0.13	38.38		0.86	1.29	<0.15	40.83	3.10		15.18			<0.066	
		91178	0.025	0.03	0.05	0.002	0.005	0.15	37.82		1.01	1.77	0.16	39.93	3.00		16.04			0.007	
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	0.16	<0.1	<0.1		0.0018	8.90	Bal.	2.60	5.50	1.68	<0.1	0.18		1.40	10.0	9.20	0.07	0.016	1.90
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	0.14	<0.05	0.01		0.002	8.91	Bal.	2.27	5.74	1.61	<0.05	0.07		1.70	10.88	9.50	0.06	0.014	1.60

---

*Included in % Fe reported.



Wrought STA-2, S/N HH24A9UK

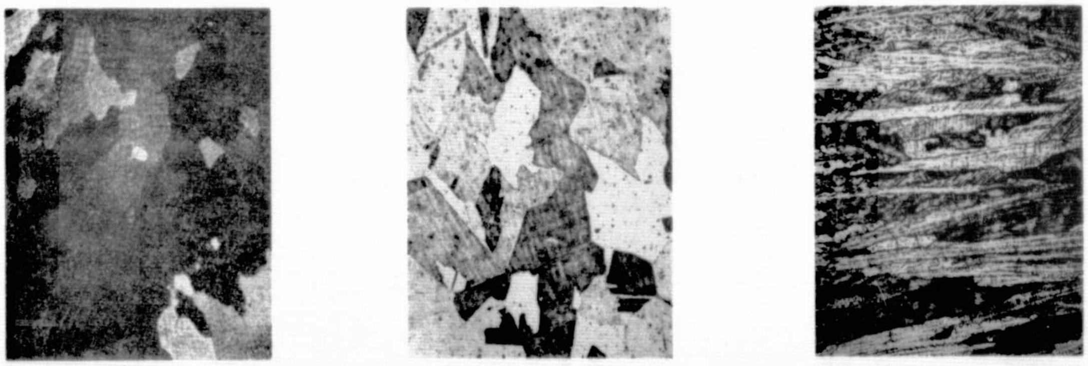
Wrought STA-2, S/N HH22A9UY

TMP STA-1, S/N HH36A5UK

As Welded (Preweld STA-2) Parent Metal Zone, S/N HH22A9UY

Welded + 718 STA Parent Metal Zone, S/N HH36A2UK

EB Welded + STA-2 Parent Metal Zone, S/N HH22A9UY



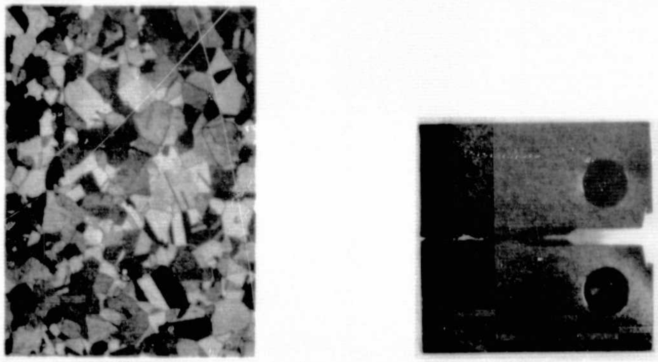
As Welded (Preweld STA-2) Weld Zone, S/N HH22A9UY

Welded + 718 STA Weld Zone, S/N HH36A2UK

EB Welded + STA-2 Weld Zone, S/N HH22A9UY

FD 122661

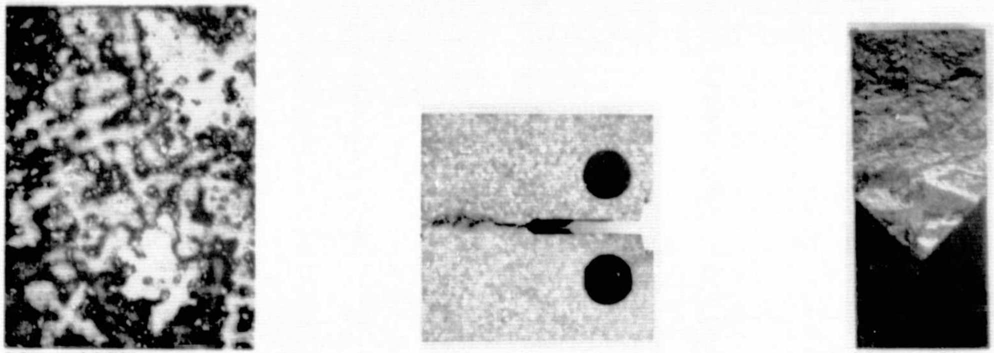
Figure III-1. Typical Microstructures of Incoloy 903 Material Used for Low-Cycle Fatigue Testing. Magnification = 100X (Prior to a 50% Reduction for Printing Purposes)



Mag: 100X
INCOLOY 903 STA-2

Mag: 2X
INCOLOY 903 STA-2
Specimen 1-11

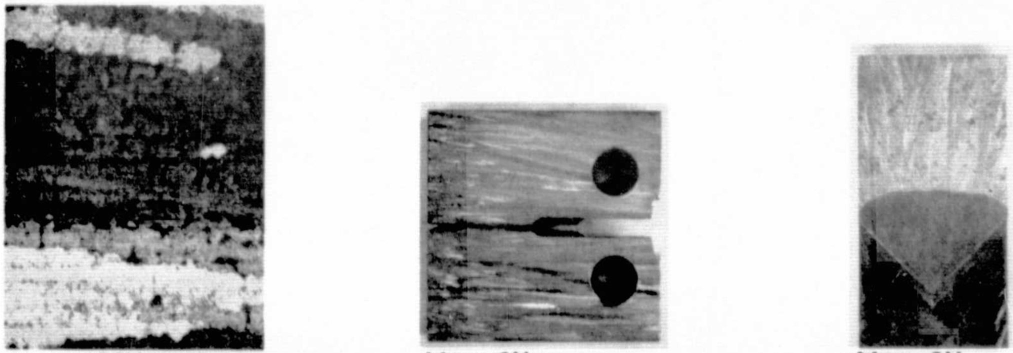
Mag: 3X
INCOLOY 903 STA-2
Specimen 1-11



Mag: 100X
Conventionally Cast
MAR-M-246 (Hf Modified)

Mag: 2X
Conventionally Cast
MAR-M-246 (Hf Modified)
Specimen 1-A

Mag: 3X
Conventionally Cast
MAR-M-246 (Hf Modified)
Specimen 1-A



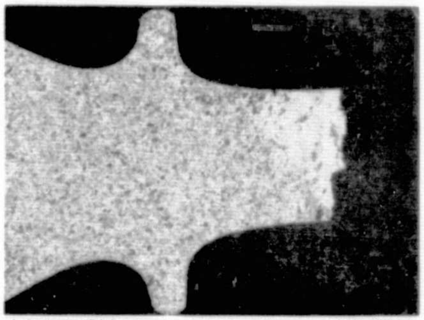
Mag: 100X
Directionally Solidified
MAR-M-246 (Hf Modified)

Mag: 2X
Directionally Solidified
MAR-M-246 (Hf Modified)
Specimen 1-H1

Mag: 3X
Directionally Solidified
MAR-M-246 (Hf Modified)
Specimen 1-H1

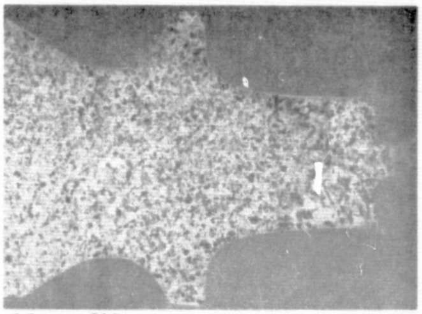
FD 122662

Figure III-2. Typical Microstructures, Macrostructures, and Fracture Faces of Materials Used for Crack Growth Rate Testing (Magnifications Indicated are Prior to a 50% Reduction for Printing Purposes)



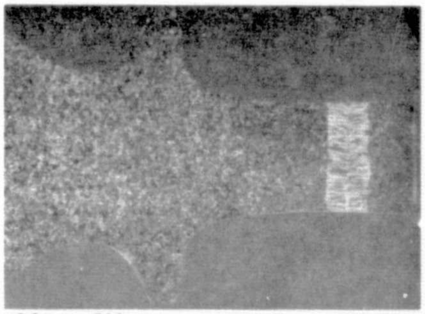
Mag: 6X FAL 43980

Incoloy 903 As Welded
(Specimen AW-10)



Mag: 6X FAL 43979

Incoloy 903 Welded + 718 STA
(Specimen 1-92)



Mag: 6X FAL 43978

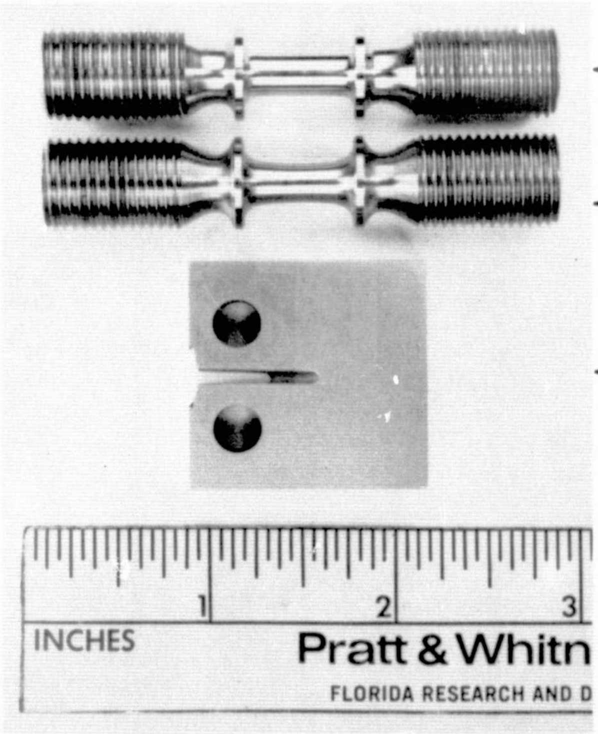
Incoloy 903 EB Welded + STA-2
(Specimen EB-1)

FD 122663

Figure III-3. Typical Macrostructures of Welded Incoloy 903 Low-Cycle Fatigue Test Specimens (Magnifications Indicated are Prior to Reduction for Printing Purposes)

Table III-3. Specimens Used to Determine Low-Cycle Fatigue and Crack Growth Rate Properties of Various Alloys in High-Pressure Hydrogen Environments

Specimen Name	*Print No.*	*Figure No.*
Constant Strain LCF	FML 95716C	III-5
Modified Constant Strain LCF (Cylindrical Gage Section)	FML 96655	III-6
Compact Tensile (Crack Growth Rate)	FML 95559-3	III-7



Modified Constant Strain LCF Specimen (Cylindrical Gage Section), FML 96655

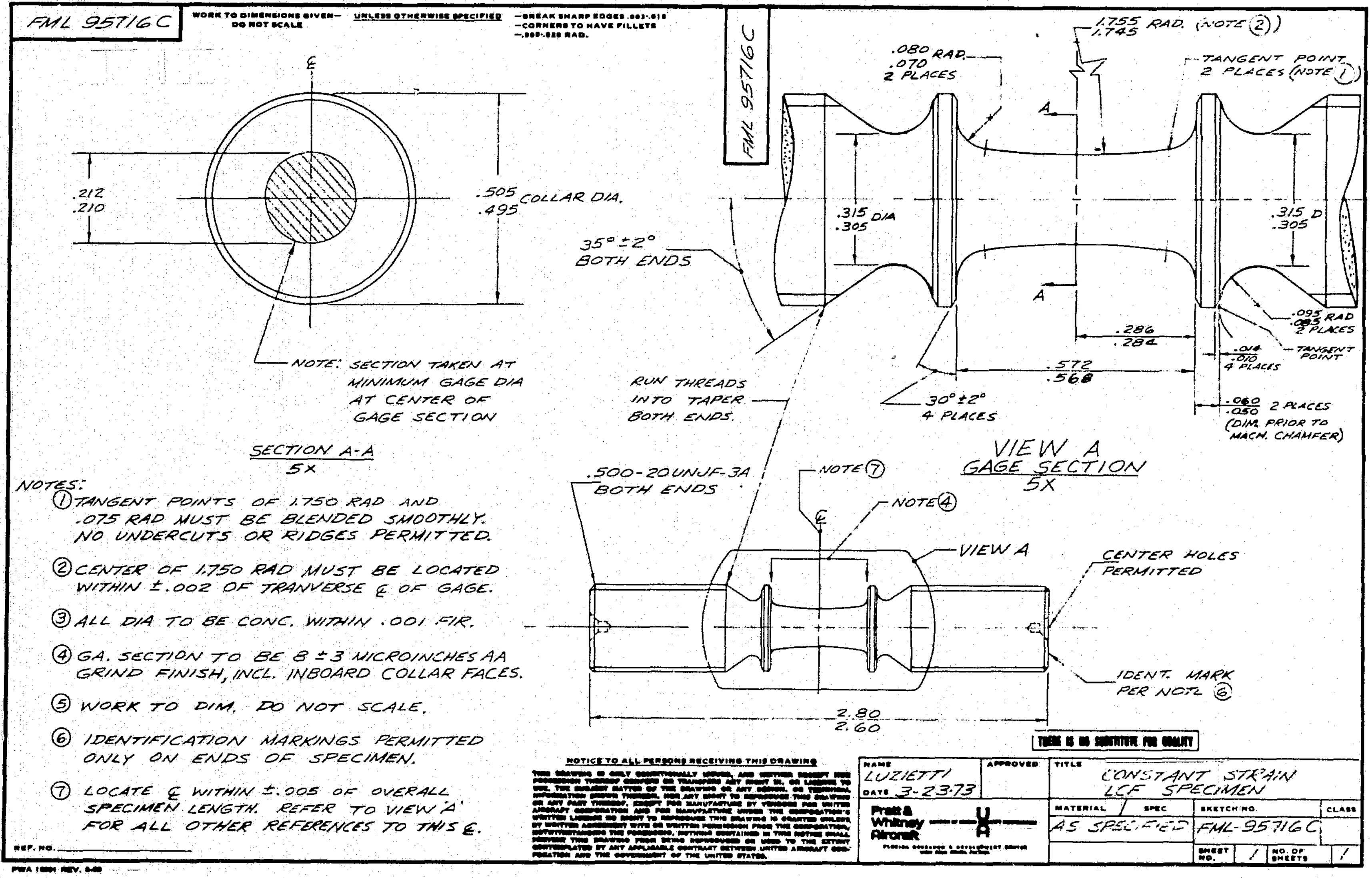
Constant Strain LCF Specimen, FML 95716C

Compact Tensile Specimen (Crack Growth) FML 95559-3

FAL 43661

FD 122664

Figure III-4. Typical Test Specimens Used to Determine the Effect of High-Pressure Gaseous Environments on Low-Cycle Fatigue and Crack Growth Rate Properties of Materials



NOTES:

① TANGENT POINTS OF 1.750 RAD AND .075 RAD MUST BE BLENDED SMOOTHLY. NO UNDERCUTS OR RIDGES PERMITTED.

② CENTER OF 1.750 RAD MUST BE LOCATED WITHIN ±.002 OF TRANVERSE ℄ OF GAGE.

③ ALL DIA TO BE CONC. WITHIN .001 FIR.

④ GA. SECTION TO BE 8 ±3 MICROINCHES AA GRIND FINISH, INCL. INBOARD COLLAR FACES.

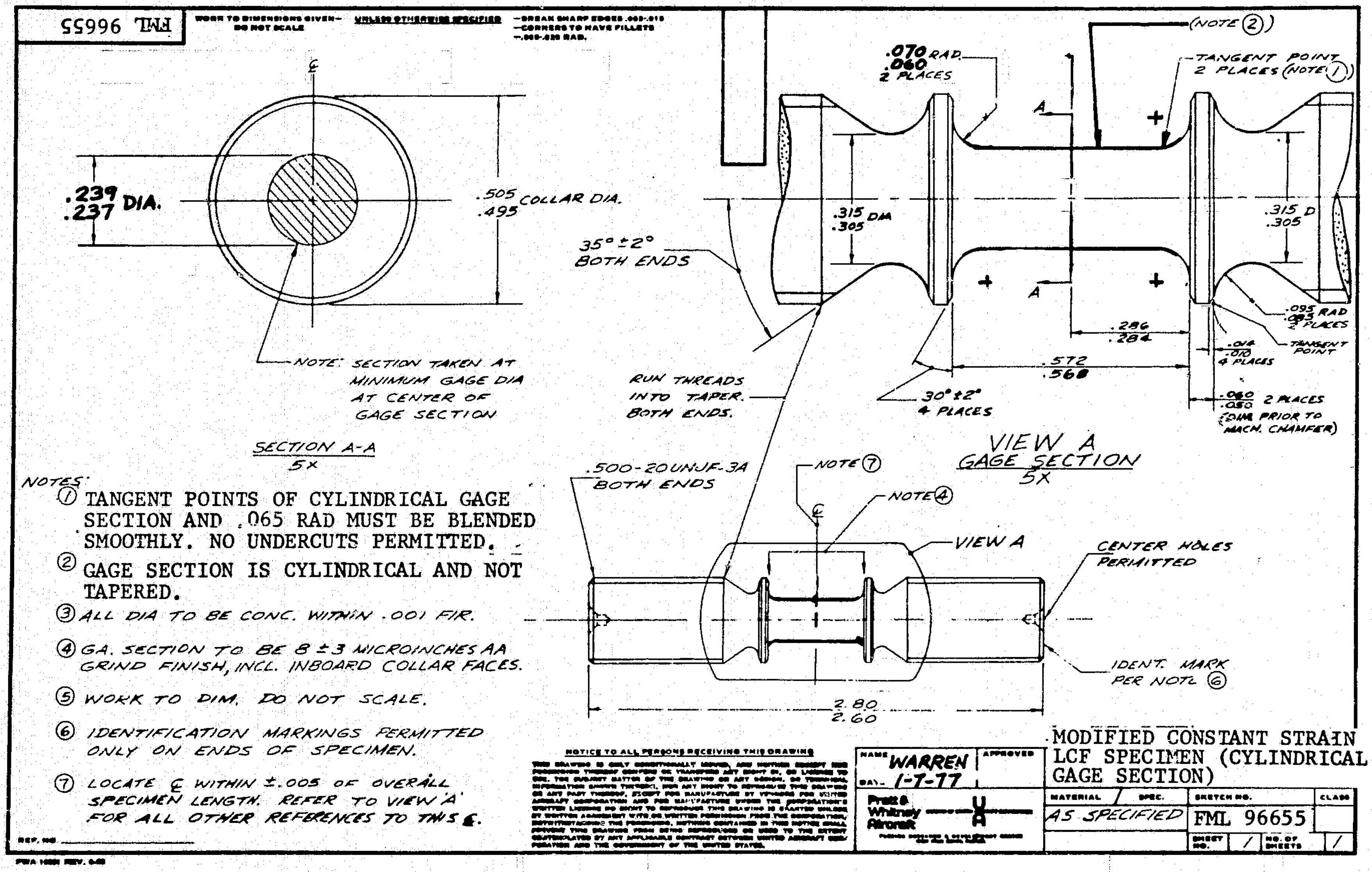
⑤ WORK TO DIM. DO NOT SCALE.

⑥ IDENTIFICATION MARKINGS PERMITTED ONLY ON ENDS OF SPECIMEN.

⑦ LOCATE ℄ WITHIN ±.005 OF OVERALL SPECIMEN LENGTH. REFER TO VIEW 'A' FOR ALL OTHER REFERENCES TO THIS ℄.

NAME LUZIETTI DATE 3-23-73	APPROVED	TITLE CONSTANT STRAIN LCF SPECIMEN
Pratt & Whitney Aircraft FLORIDA RESEARCH & DEVELOPMENT CENTER	MATERIAL / SPEC AS SPECIFIED	SKETCH NO. FML-95716C CLASS SHEET NO. 1 NO. OF SHEETS 1

Figure III-5. Low-Cycle Fatigue Specimen



NOTES:

① TANGENT POINTS OF CYLINDRICAL GAGE SECTION AND .065 RAD MUST BE BLENDED SMOOTHLY. NO UNDERCUTS PERMITTED.

② GAGE SECTION IS CYLINDRICAL AND NOT TAPERED.

③ ALL DIA TO BE CONC. WITHIN .001 FIR.

④ GA. SECTION TO BE 8 ± 3 MICROINCHES AA GRIND FINISH, INCL. INBOARD COLLAR FACES.

⑤ WORK TO DIM. DO NOT SCALE.

⑥ IDENTIFICATION MARKINGS PERMITTED ONLY ON ENDS OF SPECIMEN.

⑦ LOCATE ℄ WITHIN ±.005 OF OVERALL SPECIMEN LENGTH. REFER TO VIEW 'A' FOR ALL OTHER REFERENCES TO THIS ℄.

MODIFIED CONSTANT STRAIN LCF SPECIMEN (CYLINDRICAL GAGE SECTION)

Figure III-6. Low-Cycle Fatigue Specimen (Cylindrical Gage Section)

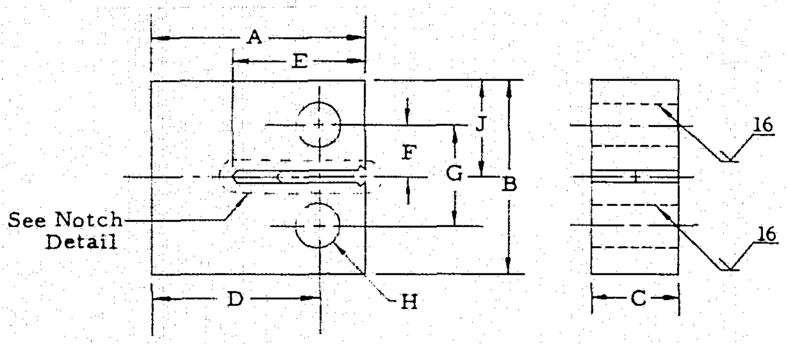
PWA [illegible] REV. 9-63

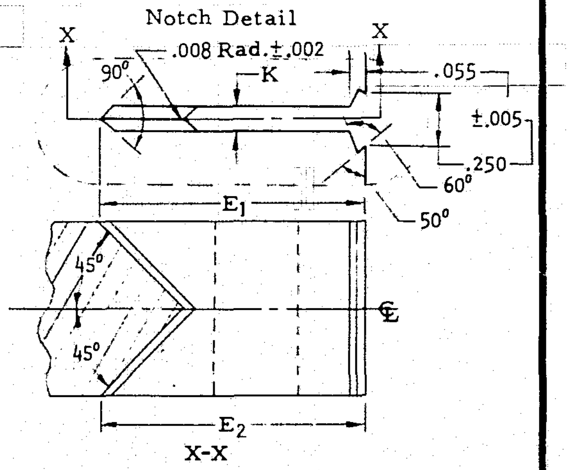
FML 95559

WORK TO DIMENSIONS GIVEN — DO NOT SCALE

UNLESS OTHERWISE SPECIFIED
— BREAK SHARP EDGES .003-.015
— CORNERS TO HAVE FILLETS
— .005-.020 RAD.

FML 95559





NOTES:
1- All dimensions in inches.
2- All external surfaces must be perpendicular and parallel to within .002.
3- Finish surfaces to within 32 r.m.s. or smoother per PWA 362 U.O.S.
4- $E_1 = E_2$ to within .0025.
5- Indicated ℄ must be within .0015 of true position.

Specimen Number	A ± .005	B ± .005	C ± .005	D ± .002	E ± .005	F ± .002	G ± .004	H + .001 - .000	J ± .002	K ± .005
1	0.625	0.600	0.250	0.500	0.325	0.137	0.275	0.125	0.300	0.031
2	0.937	0.900	0.375	0.750	0.512	0.206	0.412	0.187	0.450	0.046
3	1.250	1.200	0.500	1.000	0.700	0.275	0.550	0.250	0.600	0.062
4	1.875	1.800	0.750	1.500	1.075	0.412	0.825	0.375	0.900	0.094
5	2.500	2.400	1.000	2.000	1.450	0.550	1.100	0.500	1.200	0.125
6	3.125	3.000	1.250	2.500	1.825	0.687	1.375	0.625	1.500	0.156
7	3.750	3.600	1.500	3.000	2.200	0.825	1.650	0.750	1.800	0.187

THERE IS NO SUBSTITUTE FOR QUALITY

NAME J. Mucci
DATE 6-8- 6
APPROVED
TITLE Fracture Toughness Specimen
Pratt & Whitney Aircraft
MATERIAL / SPEC. As Supplied
SKETCH NO. FML 95559
CLASS
SHEET NO. 1
NO. OF SHEETS 1

Figure III-7. Crack Growth Rate Specimen

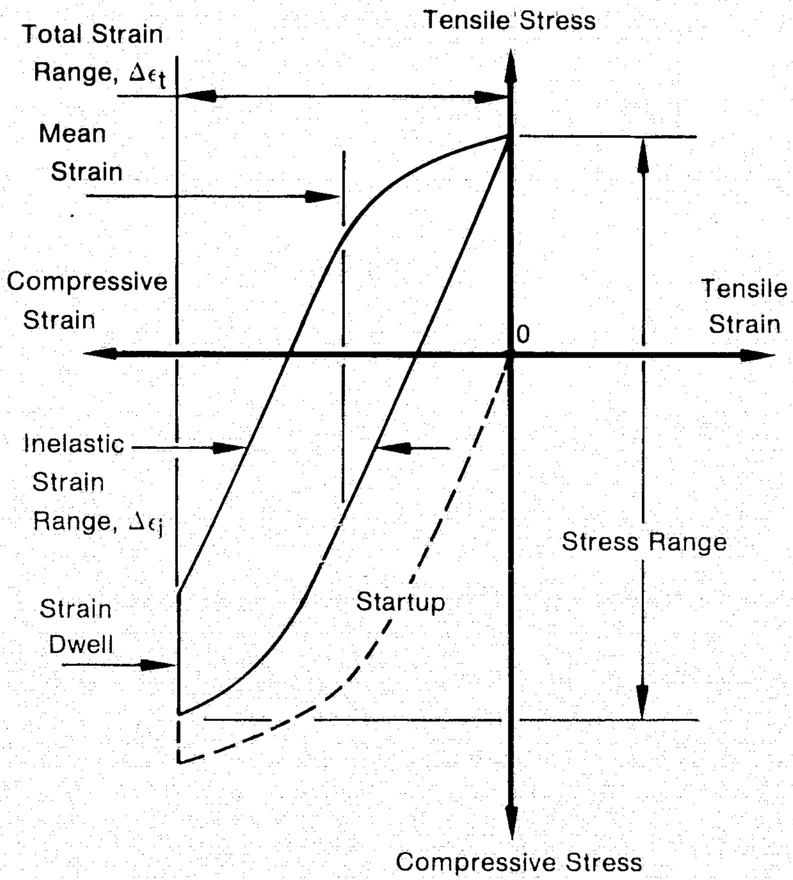
SECTION IV
LOW-CYCLE FATIGUE

A. INTRODUCTION

Strain controlled low-cycle fatigue (LCF) tests were conducted to establish cyclic life of Incoloy 903 in parent and welded forms in a high-pressure environment of hydrogen and water vapor (50% by weight). The majority of the test data was generated for limited design and engineering use. In some cases, comparable conditions were evaluated and the effects of heat-treatment and/or weld process could be noted.

The testing was very limited, consequently general observations could be made but could not be statistically substantiated.

The LCF tests were conducted in the strain-controlled mode. The test consisted of a compressive start with the material cycling through a constant total strain range (elastic plus inelastic) until specimen fracture. The strain cycle was totally compressive, with a dwell or hold time at the maximum compressive strain. This resulted in a mean strain equal to one-half the maximum compressive strain. This cycle is shown in figure IV-1. Test strain levels were initially selected by the MSFC contract monitor and occasionally adjusted as the test data became available.

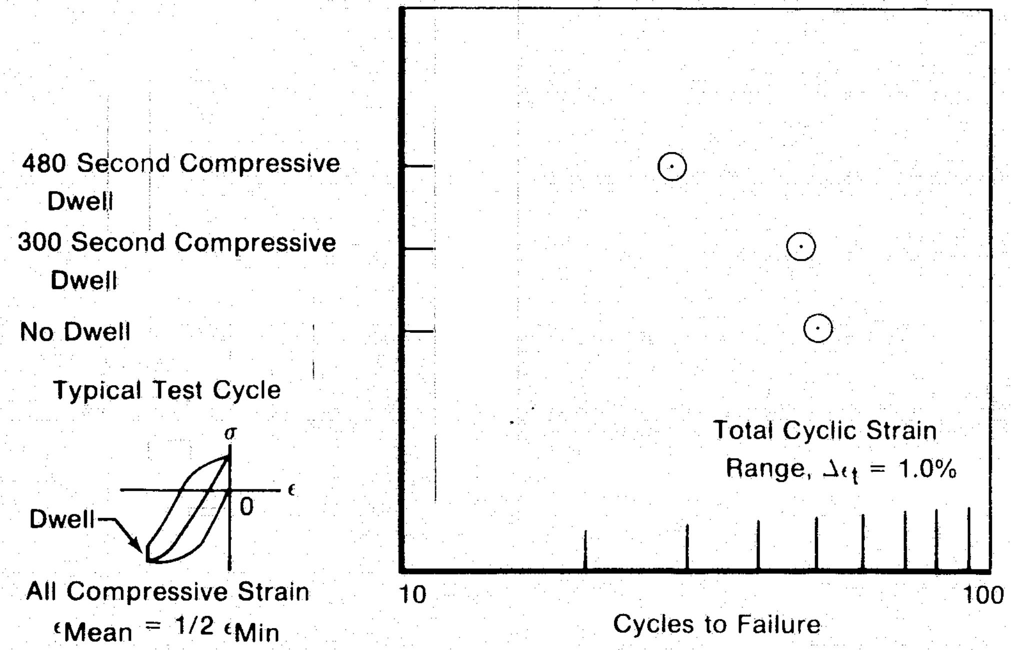


FD 122665

Figure IV-1. Typical Stress-Strain Hysteresis Curve Obtained During a Specimen Low-Cycle Fatigue Test

B. RESULTS AND CONCLUSIONS

Testing was initially conducted to establish the effect on cyclic life due to dwell time at the maximum compressive strain, and to determine the minimum dwell period which would produce the maximum life reduction consistent with proposed space propulsion mission duration. This dwell time would then be used for all subsequent tests. Incoloy 903 in the wrought STA-2 condition was tested in 34.5 MN/m² (5,000 psig) hydrogen and water vapor at 1033°K (1400°F), with dwell times of 480 seconds, 300 seconds, and with no dwell time (figure IV-2). The specimen tested with the 480-second dwell time had the lowest cyclic life; consequently this dwell time was used for all remaining tests.



FD 122666

Figure IV-2. Effect of Dwell (Hold Time) on Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 in 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 1033°K (1400°F)

Forging composition had some effect on LCF life for Incoloy 903 wrought STA-2 at 1033°K (1400°F), see figure IV-3. Specimens were machined from two different forged disks and were tested at similar strain ranges in 34.5 MN/m² (5,000 psig) hydrogen and water vapor. The difference noted however may be within normal data scatter.

Two specimens were machined to a cylindrical gage section geometry (figure III-6) for comparison with tapered gage LCF specimens (figure III-5) from the same forging. The data did show some difference, but the difference was not consistent at both strain ranges tested, and again the distinction may fall within data scatter at these conditions (figure IV-4).

Environmental degradation could be seen in LCF life due to the hydrogen and water vapor atmosphere when compared with tests conducted in air at 1033°K (1400°F) as shown in figure IV-5. A reduction in LCF life of 20% was observed at a total strain range of 1.5%, and a 70% reduction occurred at 0.7% total strain range for Incoloy 903 wrought STA-2 at 1033°K (1400°F). Figure IV-6 presents all test data for Incoloy 903 at 1033°K (1400°F).

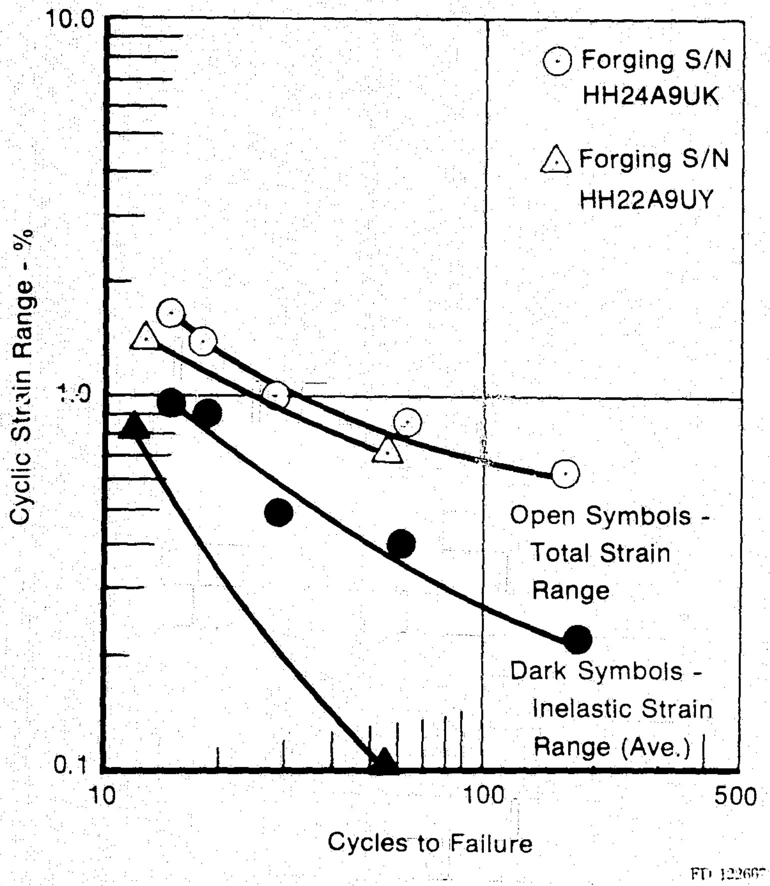


Figure IV-3. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 Comparing Forging Composition in 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 1033°K (1400°F)

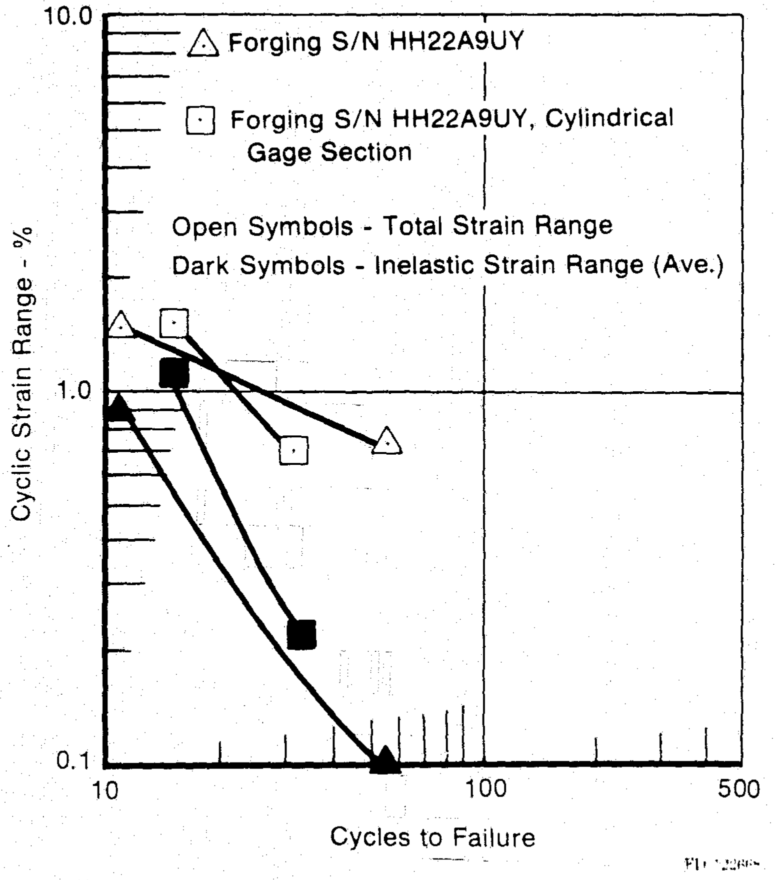


Figure IV-4. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 Comparing Specimen Gage Section Geometry in 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 1033°K (1400°F)

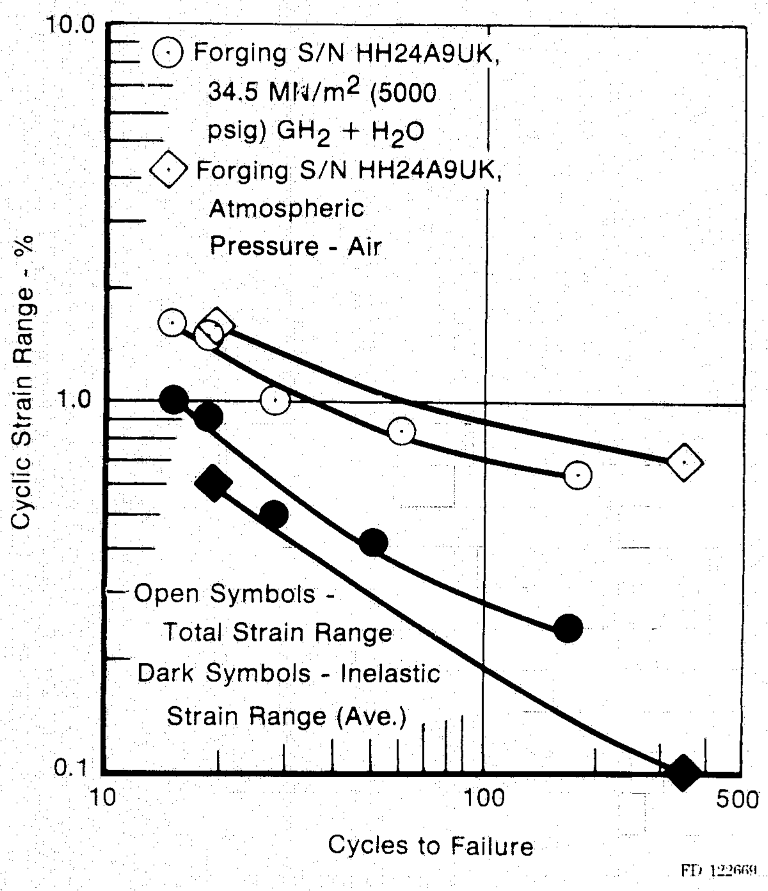


Figure IV-5. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 in Air and 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 1033°K (1400°F)

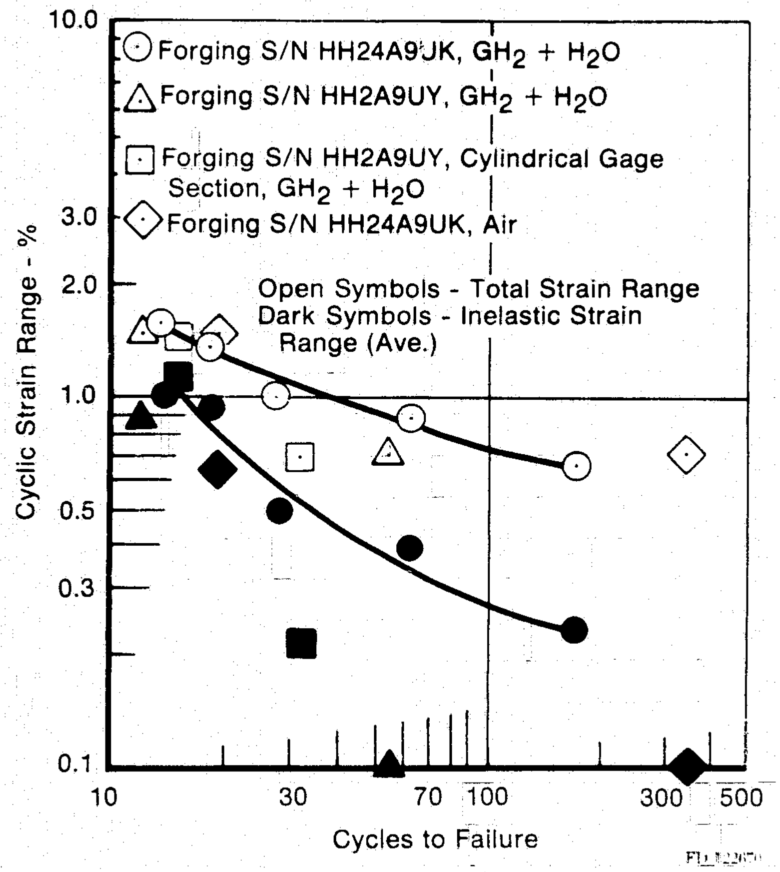


Figure IV-6. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 Comparing Forging Composition, Specimen Gage Section Geometry, and Gas Environment at 1033°K (1400°F)

Incoloy 903 material was tested in the wrought STA-2, as-welded, and welded + 718 STA forms at 977°K (1300°F). The as-welded material appeared equivalent to the wrought material at higher strain ranges, but was degraded at lower strain ranges (figure IV-7). The welded + 718 STA material was not significantly different from the wrought material at all strain ranges (figure IV-8). Figure IV-9 presents all test data for Incoloy 903 at 977°K (1300°F).

Cyclic strain range vs life for Incoloy 903, wrought STA-2 and EB welded, at 755°K (900°F) is shown in figure IV-10.

Incoloy 903 in the TMP + STA-1 form was tested at 644°K, 811°K, and 922°K (700°F, 1000°F, and 1200°F respectively). TMP represents thermomechanical processing, wherein the finish forging operation (final 40/50% reduction) is performed below 1144°K (1600°F). Cyclic strain range vs life for this material is presented in figures IV-11 through IV-13.

Some specific tests were conducted to screen for problems. Certain material/temperature/strain range conditions which engine hardware is expected to experience were investigated. A cyclic life requirement of 240 cycles was specified. If failures occurred prior to reaching 240 cycles, further tests were conducted. If a specimen completed 240 cycles without failure, the test was discontinued.

Complete LCF test results are listed in table IV-1.

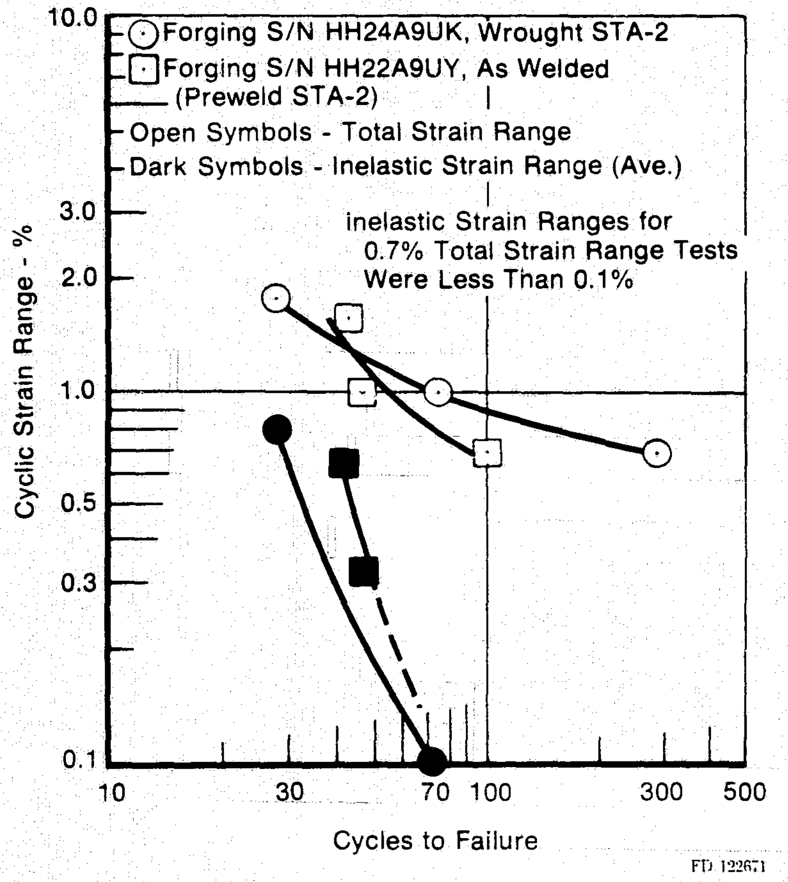


Figure IV-7. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 and as Welded in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 977°K (1300°F)

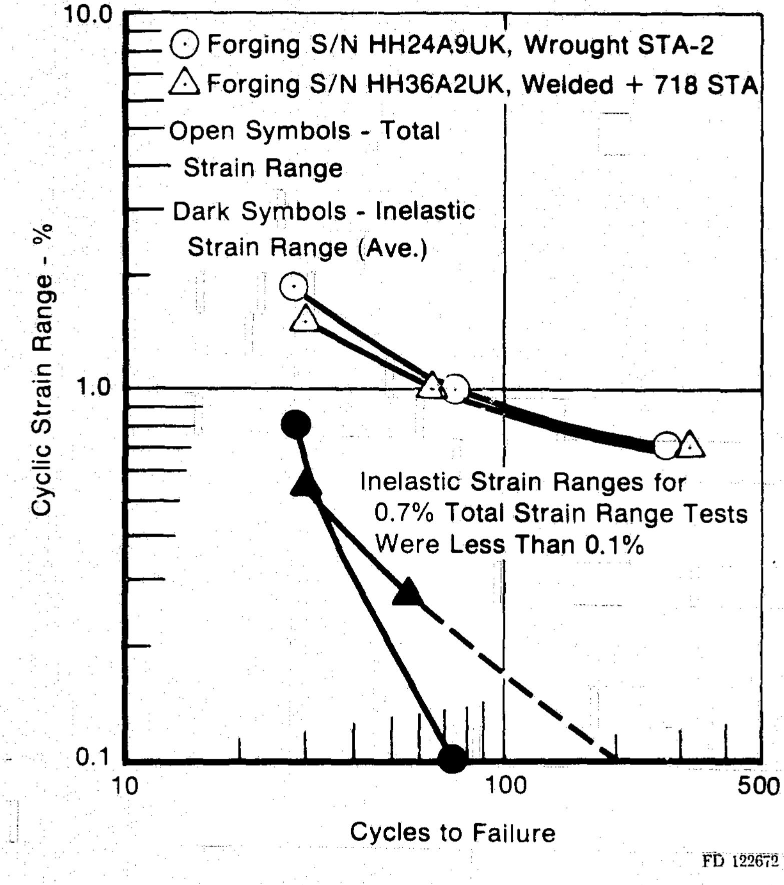
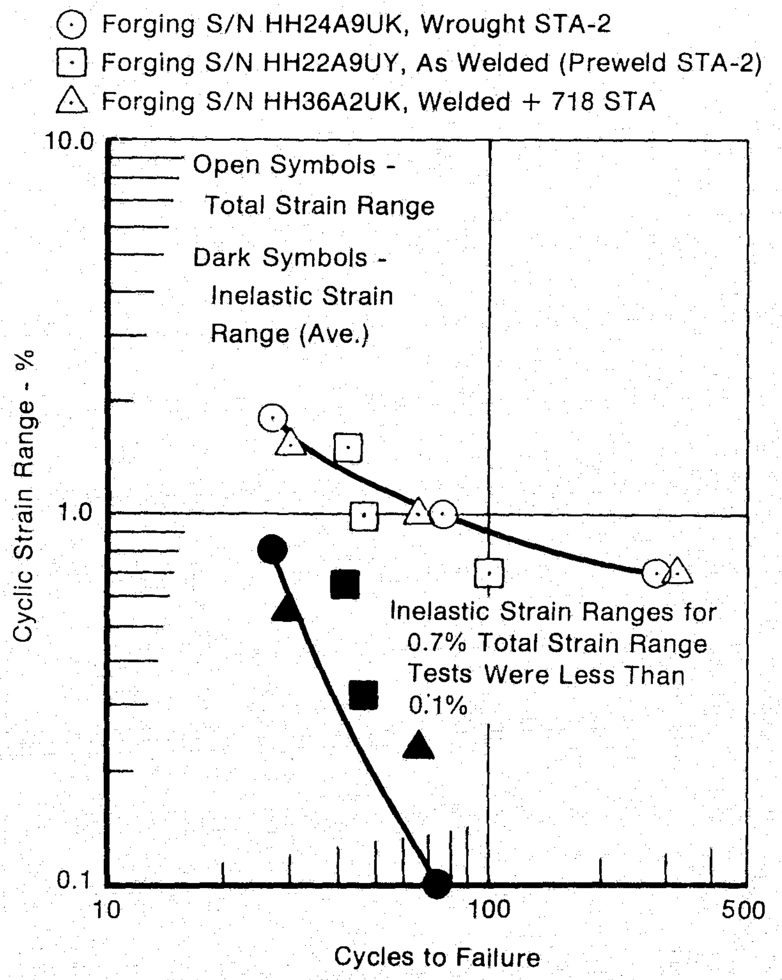


Figure IV-8. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 and Welded + 718 STA; in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 977°K (1300°F)



FD 122673

Figure IV-9. Low-Cycle Fatigue Life of Incoloy 903, Wrought and Welded, in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 977°K (1300°F)

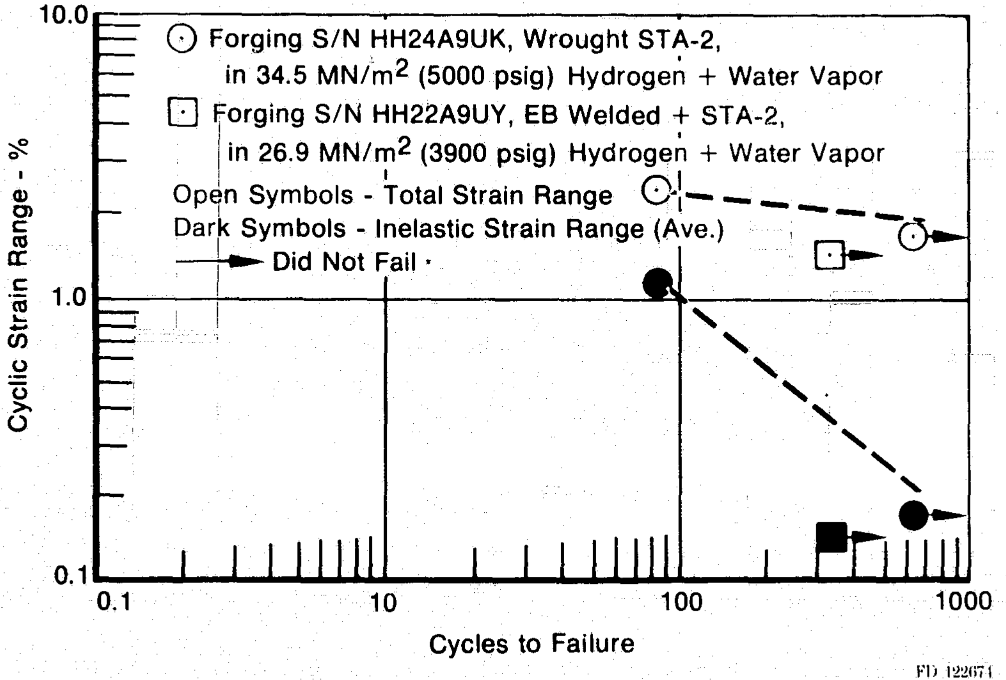


Figure IV-10. Low-Cycle Fatigue Life of Incoloy 903, Wrought STA-2 and EB Welded + STA-2, at 755°K (900°F)

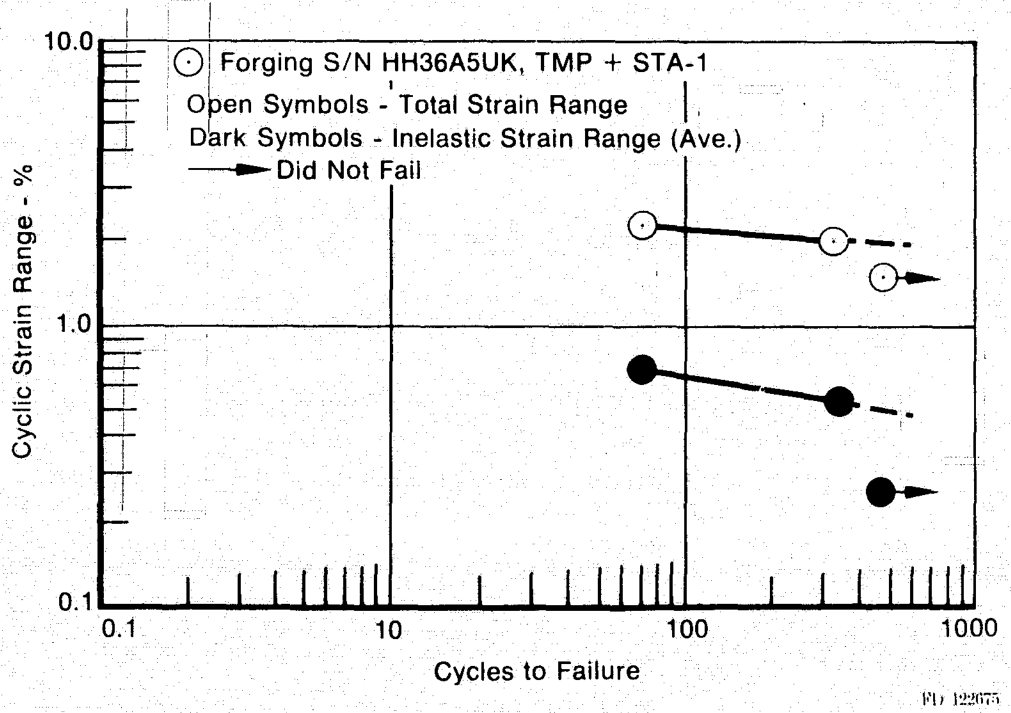
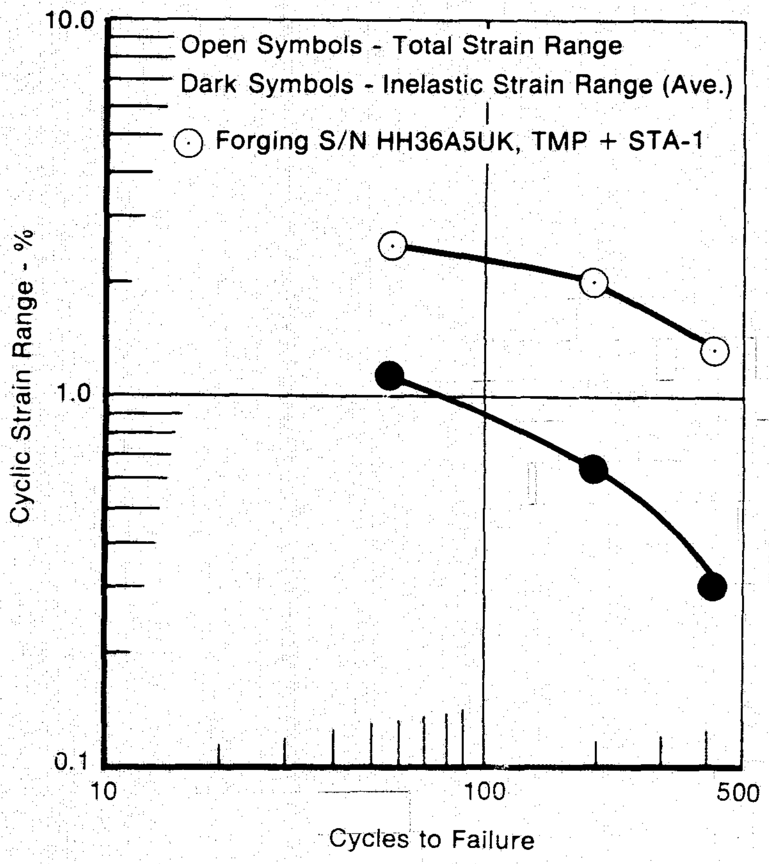
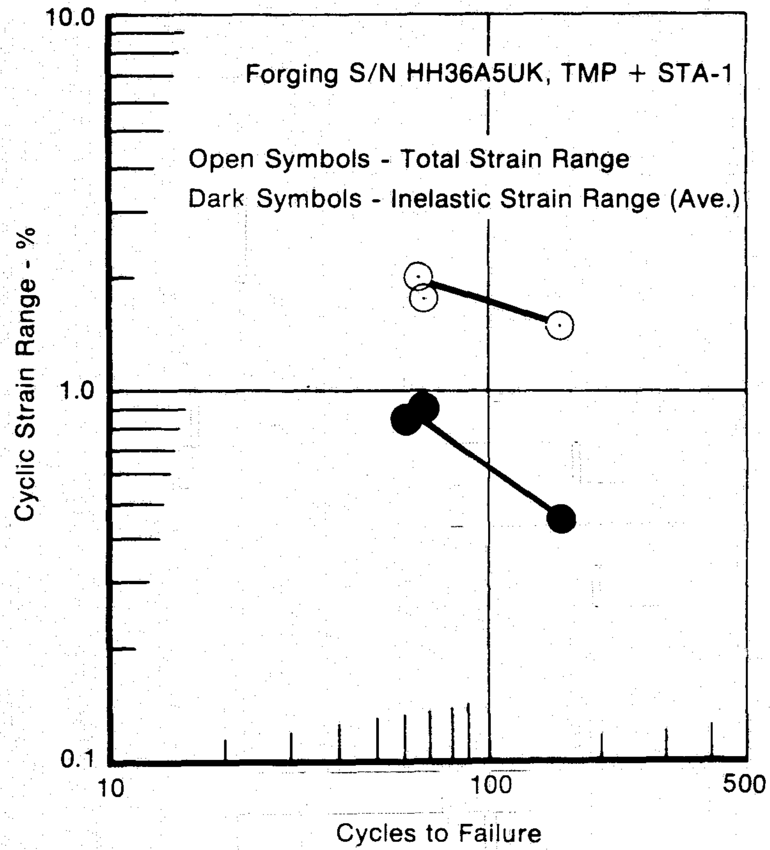


Figure IV-11. Low-Cycle Fatigue Life of Incoloy 903, TMP + STA-1, in 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 644°K (700°F)



FD 122676

Figure IV-12. Low-Cycle Fatigue Life of Incoloy 903, TMP + STA-1, in 31.0 MN/m² (4500 psig) Hydrogen + Water Vapor at 811°K (1000°F)



FD 122677

Figure IV-13. Low-Cycle Fatigue Life of Incoloy 903, TMP + STA-1, in 34.5 MN/m² (5000 psig) Hydrogen + Water Vapor at 922°K (1200°F)

Table IV-1. Low-Cycle Fatigue Properties of Incoloy 903 in Air and High-Pressure Hydrogen and Water Vapor

Material			Test Conditions							Test Results			
			Test Temperature			Pressure					Inelastic Strain Range (%)		
Condition[1]	Heat No.	Specimen S/N	°K	°F	Environment[2]	MN/m²	psig	Total Strain Range[3] (%)	Dwell Time[4] (Seconds)	Cycles to Failure	Minimum	Maximum	Average
Wrought STA-2	HH24A9UK	P1	1033	1400	GH_2+H_2O	34.5	5000	1.00	480	28	0.30	0.70	0.50
Wrought STA-2	HH24A9UK	P2	1033	1400	GH_2+H_2O	34.5	5000	1.00	300	46	0.29	0.40	0.35
Wrought STA-2	HH24A9UK	P4	1033	1400	GH_2+H_2O	34.5	5000	1.00	0	50	0.20	0.33	0.27
Wrought STA-2	HH24A9UK	P5	1033	1400	GH_2+H_2O	34.5	5000	0.87	480	61	0.27	0.52	0.39
Wrought STA-2	HH24A9UK	P6	1033	1400	GH_2+H_2O	34.5	5000	1.27	0	46	0.45	0.53	0.49
Wrought STA-2	HH24A9UK	P7	1033	1400	GH_2+H_2O	34.5	5000	1.44	480	18	0.85	1.00	0.93
Wrought STA-2	HH24A9UK	P8	1033	1400	GH_2+H_2O	34.5	5000	0.64	480	162	0.08	0.37	0.23
Wrought STA-2	HH24A9UK	P9	1033	1400	GH_2+H_2O	34.5	5000	1.57	480	14	0.97	1.05	1.01
Wrought STA-2	HH24A9UK	P10	977	1300	GH_2+H_2O	34.5	5000	1.71	480	27	0.73	0.88	0.81
Wrought STA-2	HH24A9UK	P11	755	900	GH_2+H_2O	34.5	5000	2.50	480	88	0.90	1.23	1.07
Wrought STA-2	HH24A9UK	P12	977	1300	GH_2+H_2O	34.5	5000	1.00	480	73	0.05	0.15	0.10
Wrought STA-2	HH24A9UK	P13	977	1300	GH_2+H_2O	34.5	5000	0.70	480	285	0.02	0.06	0.04
Wrought STA-2	HH24A9UK	P14	755	900	GH_2+H_2O	34.5	5000	1.50	480	672[6]	0.14	0.21	0.18
Wrought STA-2	HH24A9UK	P15	589	600	GH_2+H_2O	34.5	5000	1.50	480	320[6]	0.09	0.14	0.12
Wrought STA-2	HH24A9UK	P16	1033	1400	Air	One Atmosphere		1.50	480	19	0.56	0.69	0.63
Wrought STA-2	HH24A9UK	P17	1033	1400	Air	One Atmosphere		0.70	480	354	0.03	0.16	0.10
Wrought STA-2	HH22A9UY	P20	1033	1400	GH_2+H_2O	34.5	5000	1.50	480	12	0.80	0.95	0.88
Wrought STA-2	HH22A9UY	P21	1033	1400	GH_2+H_2O	34.5	5000	0.70	480	55	0.06	0.13	0.10
Wrought STA-2	HH22A9UY	P22[5]	1033	1400	GH_2+H_2O	34.5	5000	1.50	480	15	1.08	1.11	1.10
Wrought STA-2	HH22A9UY	P23[5]	1033	1400	GH_2+H_2O	34.5	5000	0.70	480	31	0.18	0.26	0.22
TMP + STA-1	HH36A5UK	T1	811	1000	GH_2+H_2O	31.0	4500	2.50	480	58	1.15	1.38	1.27
TMP + STA-1	HH36A5UK	T3	922	1200	GH_2+H_2O	34.5	5000	1.80	480	65	0.80	0.96	0.88
TMP + STA-1	HH36A5UK	T4	644	700	GH_2+H_2O	34.5	5000	2.25	480	70	0.61	0.79	0.70
TMP + STA-1	HH36A5UK	T5	922	1200	GH_2+H_2O	34.5	5000	2.00	480	63	0.79	0.95	0.87
TMP + STA-1	HH36A5UK	T6	922	1200	GH_2+H_2O	34.5	5000	1.50	480	149	0.42	0.50	0.46
TMP + STA-1	HH36A5UK	T7	811	1000	GH_2+H_2O	31.0	4500	1.50	480	420	0.28	0.31	0.30
TMP + STA-1	HH36A5UK	T8	811	1000	GH_2+H_2O	31.0	4500	2.00	480	201	0.62	0.65	0.64
TMP + STA-1	HH36A5UK	T9	644	700	GH_2+H_2O	34.5	5000	1.50	480	482[6]	0.25	0.28	0.27
TMP + STA-1	HH36A5UK	T10	644	700	GH_2+H_2O	34.5	5000	2.00	480	323	0.48	0.58	0.53
As Welded	HH22A9UY	AW10	977	1300	GH_2+H_2O	34.5	5000	1.50	480	41	0.58	0.71	0.65
As Welded	HH22A9UY	AW11	811	1000	GH_2+H_2O	22.4	3250	1.40	480	490[6]	0.68	0.77	0.73
As Welded	HH22A9UY	AW12	977	1300	GH_2+H_2O	34.5	5000	0.70	480	99	0.04	0.07	0.06
As Welded	HH22A9UY	AW13	977	1300	GH_2+H_2O	34.5	5000	1.00	480	46	0.30	0.34	0.32
Welded + 718 STA	HH36A2UK	1-92	977	1300	GH_2+H_2O	34.5	5000	1.50	480	30	0.50	0.59	0.55
Welded + 718 STA	HH36A2UK	2-92	977	1300	GH_2+H_2O	34.5	5000	1.00	480	67	0.20	0.25	0.23
Welded + 718 STA	HH36A2UK	3-92	977	1300	GH_2+H_2O	34.5	5000	0.70	480	319	0.04	0.13	0.09
Welded + 718 STA	HH36A2UK	4-92	644	700	GH_2+H_2O	22.4	3250	1.40	480	304	0.15	0.17	0.16
EB Welded + STA-2	HH22A9UY	EB-1	755	900	GH_2+H_2O	26.9	3900	1.40	480	333[6]	0.06	0.21	0.14

NOTES:

[1] Condition:

STA-2 — 1227°K (1750°F) 1 hr. AC+, 991°K (1325°F) 8 hr + 894°K (1150°F) 8 hr (Total age time = 18 hr) AC

TMP + STA-1 — Finish forging operation (Final 40/50% reduction) performed below 1144°K (1600°F) — (Thermo-mechanical processing) + 1130°K (1575°F) 1 hr, AC+ 991°K (1325°F) 8 hr + 894°K (1150°F) 8 hr (Total age time = 18 hr) AC

As Welded — Preweld STA-2 HT per above + GTA (Gas tungsten arc) weld.

Welded + 718 STA — GTA weld + 1311°K (1900°F) 20 min, AC + 1033°K (1400°F) 10 hr + 922°K (1200°K) for total age time 20 hr, AC

EB Welded + STA-2 — EB (electron beam) welded + STA-2 HT per above.

[2] Environment:

GH_2+H_2O — 50% gaseous hydrogen + 50% water vapor by weight.

Air — Air at atmospheric pressure.

[3] Total Strain Range: Strain Ranges are all compressive strain with mean strain ($\bar{\epsilon}$) equal to ½ the maximum compressive strain (ϵ_{min}). $\bar{\epsilon} = 1/2\ \epsilon_{min}$

[4] Dwell Time: Dwell time is the period of time the specimen is held at the maximum compressive strain.

[5] Specimens P22 and P23 had a cylindrical gage section geometry (see figure III-6). All other tests used standard LCF specimen (Figure III-5).

[6] Test was stopped - specimen did not fail.

C. TEST PROCEDURE

Smooth, round, solid specimens were used for the strain-controlled LCF tests conducted under this contract. The test specimens used are described in Section III and detailed in figures III-5 and III-6. The specimen configuration incorporates integral machined extensometer collars. A calibration procedure has been established to relate the maximum strain-to-collar-deflection during both the elastic and inelastic portion of the strain cycle. The specimen design and calibration procedure were verified both experimentally and analytically.

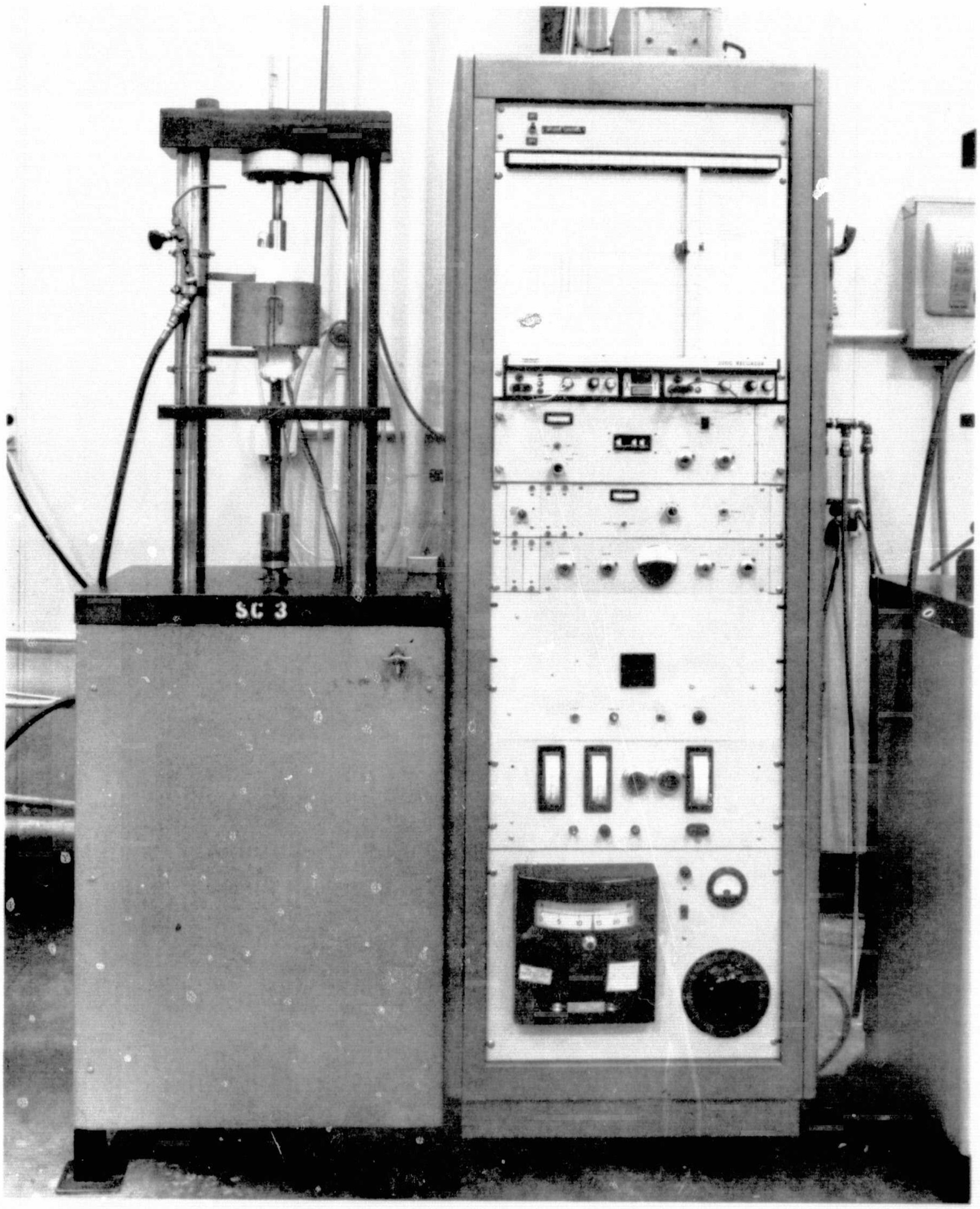
All tests were conducted on P&WA-designed and fabricated, closed-loop-type, hydraulically actuated test machines utilizing the strain-control mode. The machine, controls, and readout instrumentation used for the air tests are shown in figure IV-14. Specimen axial strain was measured and controlled by means of a dual proximity probe extensometer system (figure IV-15). Specimen load was recorded using a commercial tension-compression flat-load cell.

High-pressure environmental tests were conducted on a closed-loop-type, hydraulically actuated test machine, similar to the one used for the air tests. The test machine is located in an isolated test cell with all controls and instrumentation located in an adjacent blockhouse (figure IV-16). A P&WA designed pressure vessel was mounted on the upper platen of the test machine. The vessel incorporates a Grayloc type high-pressure flange for sealing and ease of assembly. The test machine compensates through the servosystem for the load in the specimen due to pressure acting over differential specimen/adapter areas. A pressure transducer provided a feedback signal, proportional to chamber pressure, to the servocontroller. This signal was used in controlling a mean load applied to the linkage so zero strain in the specimen gage was maintained when the vessel assembly was pressurized. This same load was then superimposed on the cyclic load during testing.

Both internal (to the pressure vessel) and external load cells were used to obtain cyclic load; thus, the effect of friction at the load rod seals was known and accounted for. Electrical connections to the load cell, extensometer system, furnace (for elevated temperature tests), and thermocouples were made through the vessel wall via high-pressure bulkhead connectors. Setups of the pressure vessel showing the extensometer system and furnace arrangement are shown in figure IV-17.

For elevated temperature testing, a two-zone resistance furnace with separate control systems for each zone was used. The furnace surrounds the specimen and fits within the frame of the pressure vessel (figure IV-17c). Thermocouples attached to the specimen gage section were used to monitor and control temperature during test.

The hydrogen and water vapor environment was obtained utilizing triple-distilled water in a pure hydrogen-containing retort system so the water was vaporized by furnace heat. The retort system, containing the test specimen and water, fits within the furnace and consists of a piston/tube type arrangement (figures IV-18 and IV-19). The piston, attached to the lower pull rod, incorporates an O-ring which provides a seal against the inner surface of a tube (cylinder), which is attached to the upper pull rod. During test the tube remains basically stationary relative to the piston. The base of the piston incorporates O-ring holes for passage of the extensometer tubes, and check valves which allow hydrogen to enter the retort and prevent water from escaping. Pressure inside the retort and vessel was equalized; therefore, the retort contained the hydrogen and water vapor environment and was not subjected to any stresses due to differential (internal to external) pressure. Thermocouples also exit the retort via connectors installed in the base of the piston. They monitor and control specimen and water vapor temperature. By controlling the lower zone of the furnace, water was vaporized at a temperature which assured 500,000-ppm water vapor (50% by weight).



FC 30186

Figure IV-14. Servohydraulic Closed-Loop Low-Cycle Fatigue Testing Machine (Used for Atmospheric Pressure Air Tests)

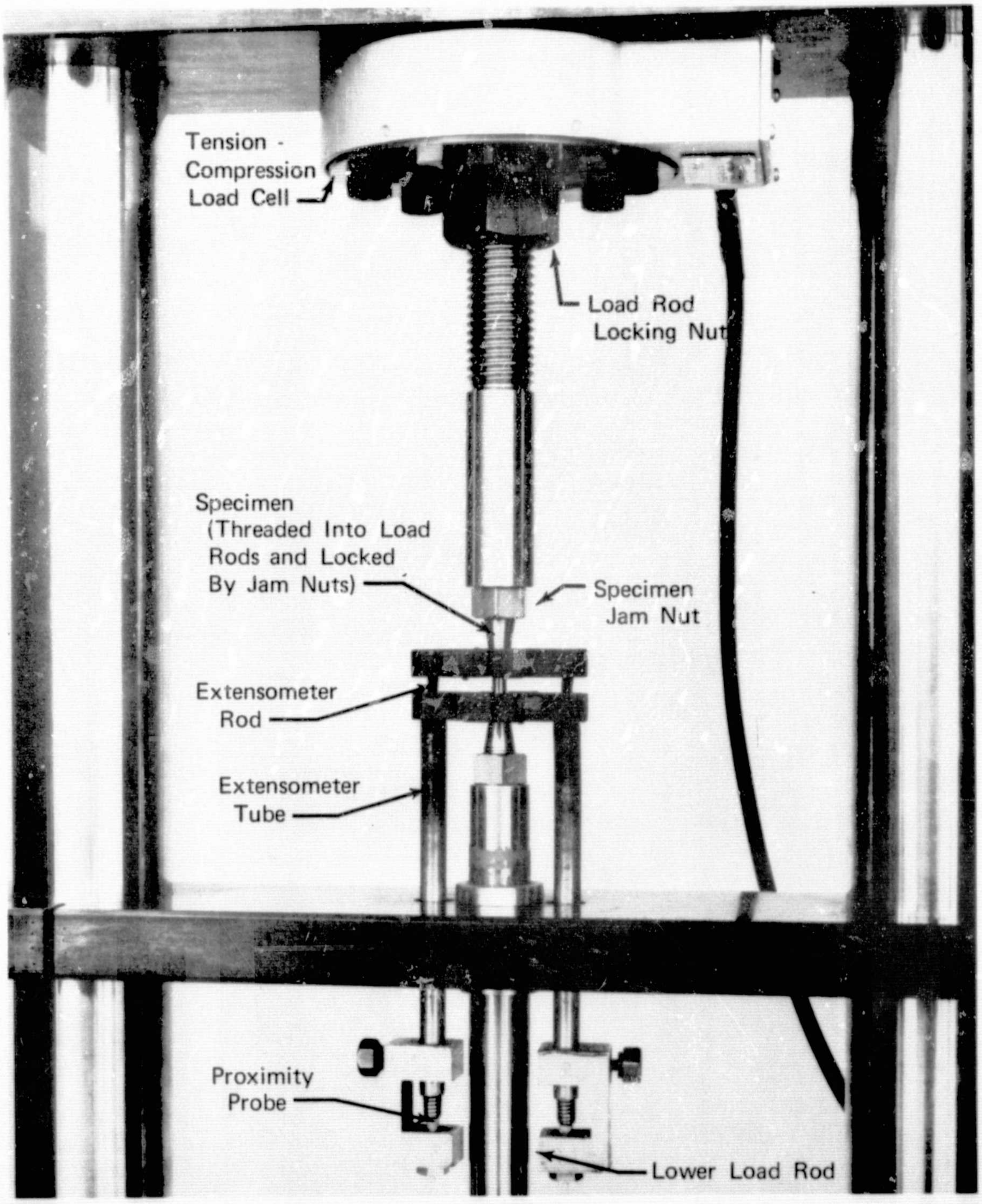
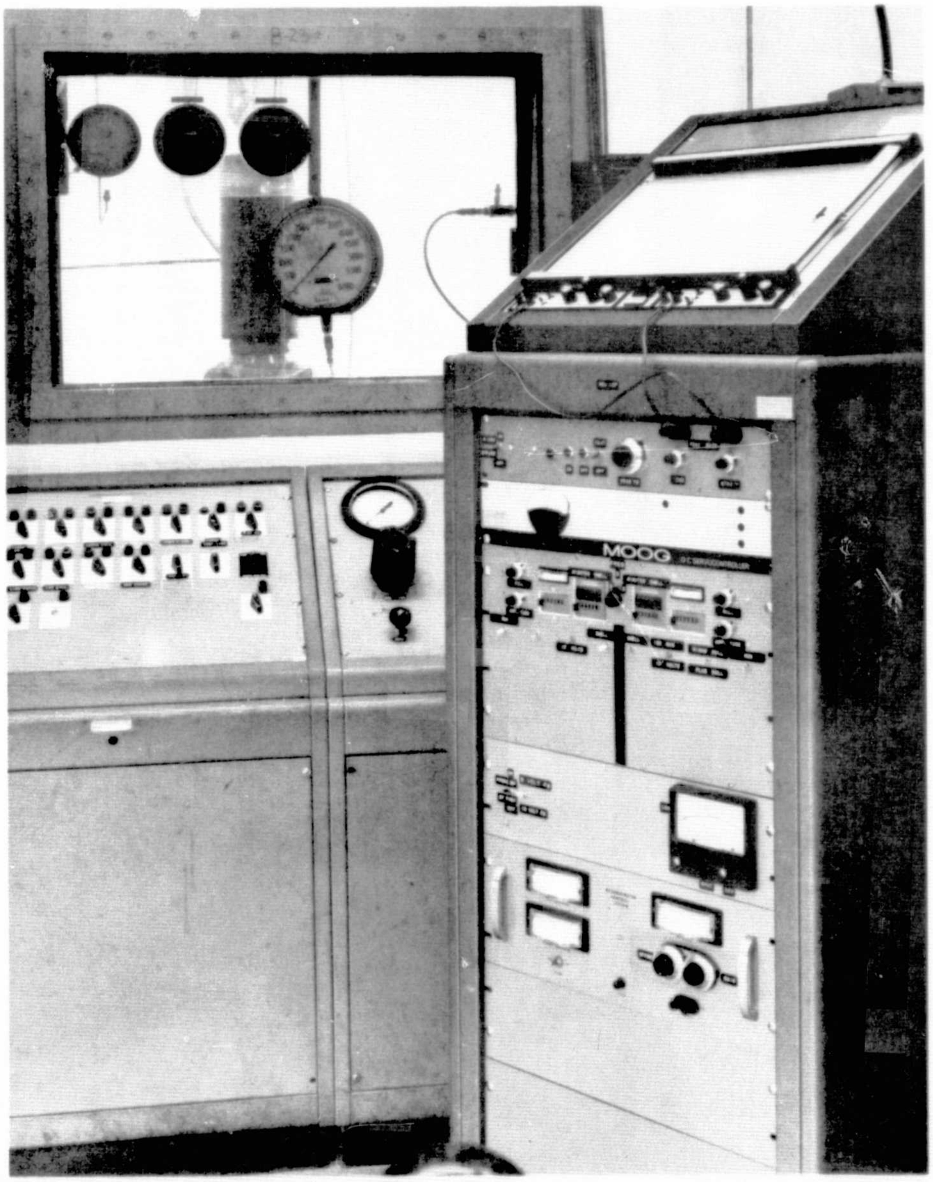


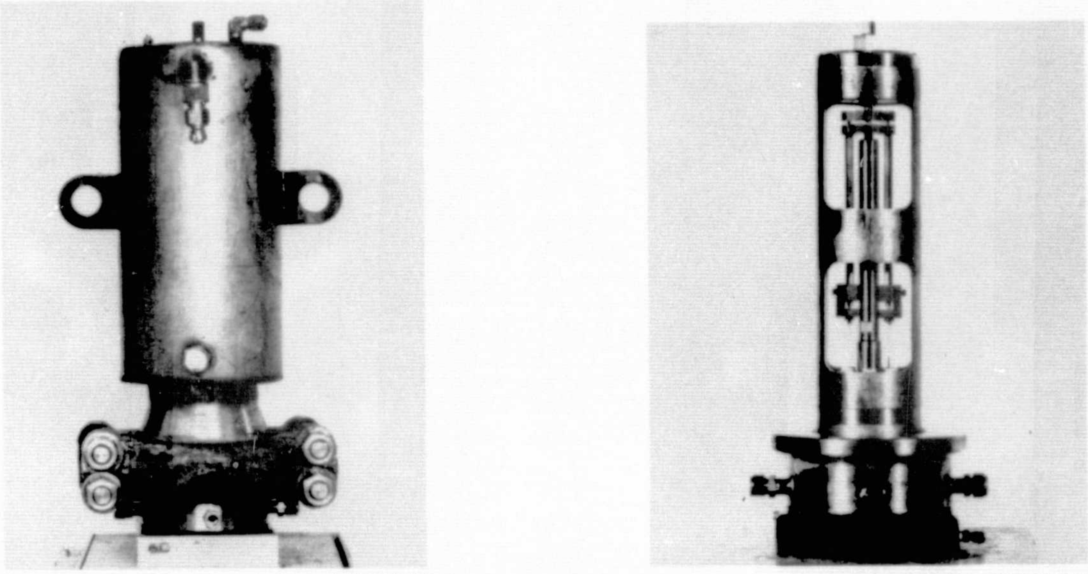
Figure IV-15. Load Cell, Load Rods, Specimen, and Extensometer Assembly Mounted in Low-Cycle Fatigue Testing Machine (Used for Atmospheric Pressure Air Tests)

ORIGINAL PAGE IS
OF POOR QUALITY



FD 92639

Figure IV-16. High-Pressure Environment Low-Cycle Fatigue Testing Machine, Environmental Controls, and Data Acquisition Equipment

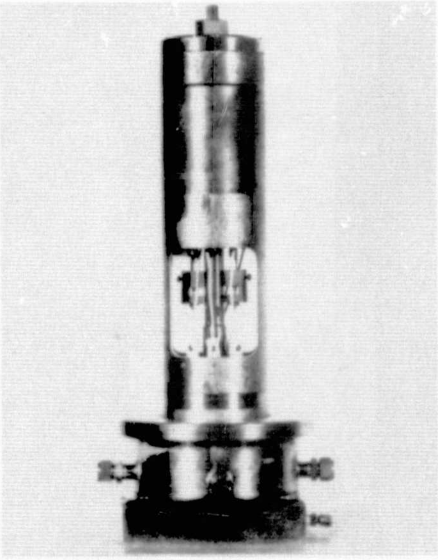


FAE 146129

a) Test Vessel Closed

FAE 146121

b) Test Vessel Open Showing Extensometer System

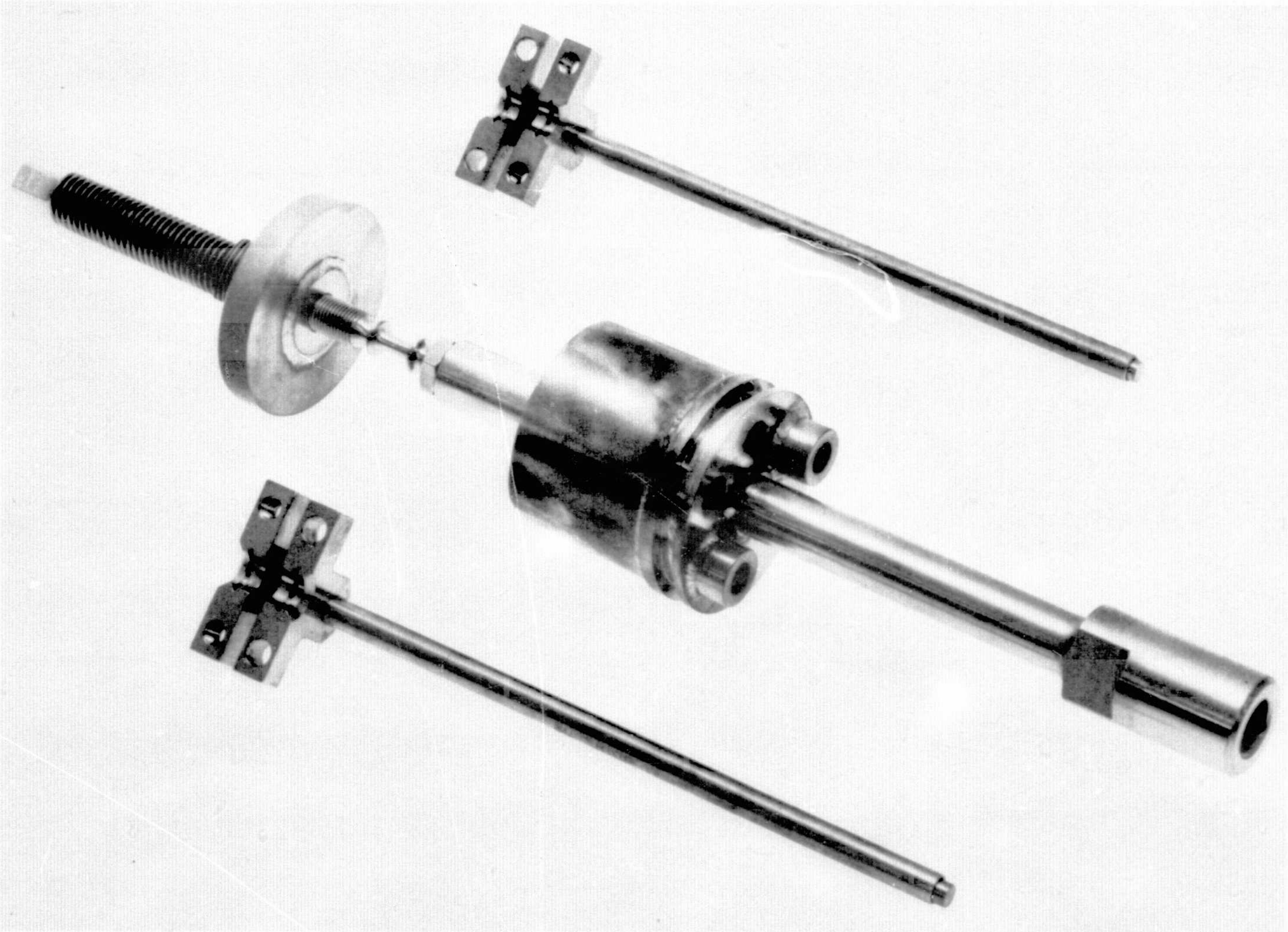


FAE 146122

c) Test Vessel Open Showing Furnace In Place

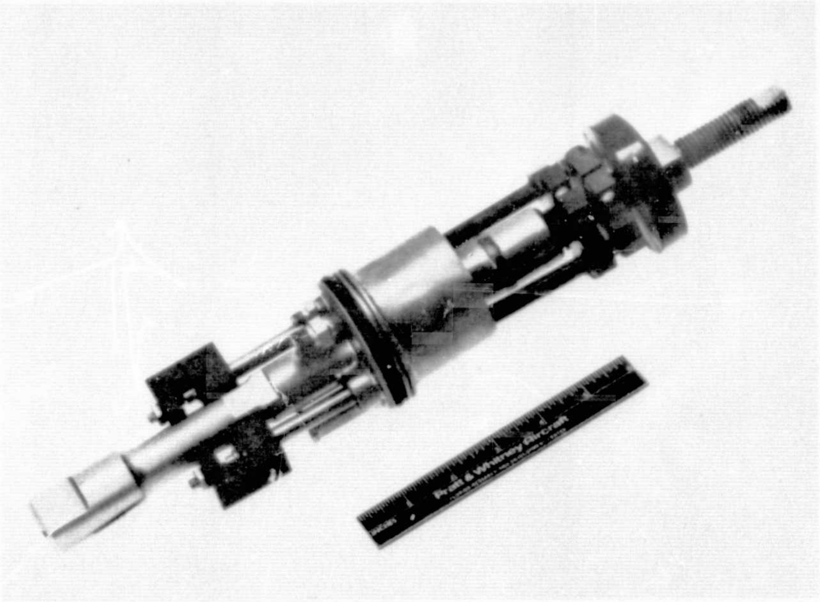
FD 92640

Figure IV-17. Low-Cycle Fatigue High-Pressure Environmental Test Vessel



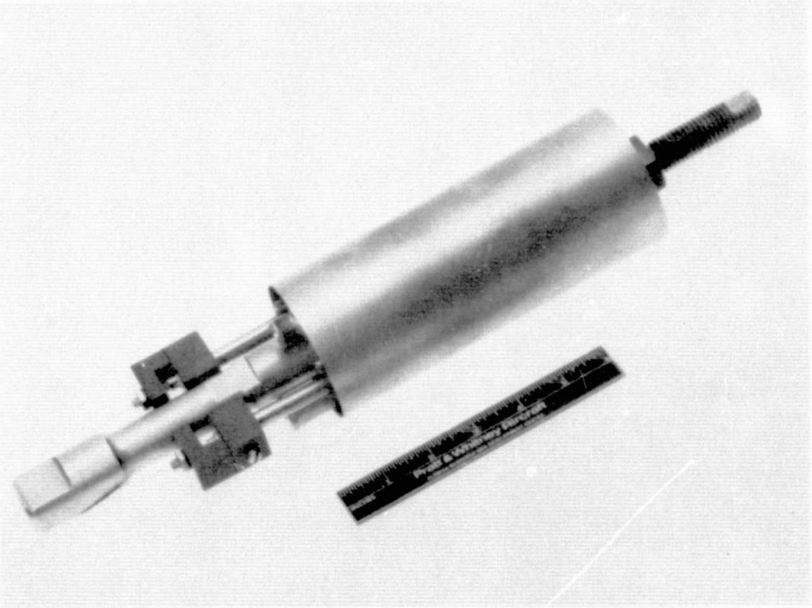
FAE 152723

Figure IV-18. Open Extensometer Head Assembly, Low-Cycle Fatigue Specimen, and Retort Pistons



FAE 146128

a) Retort With Cylinder Removed Showing Piston, Extensometer System and Thermocouple Leads



FAE 146132

b) Retort System With Cylinder In Place

FD 92641

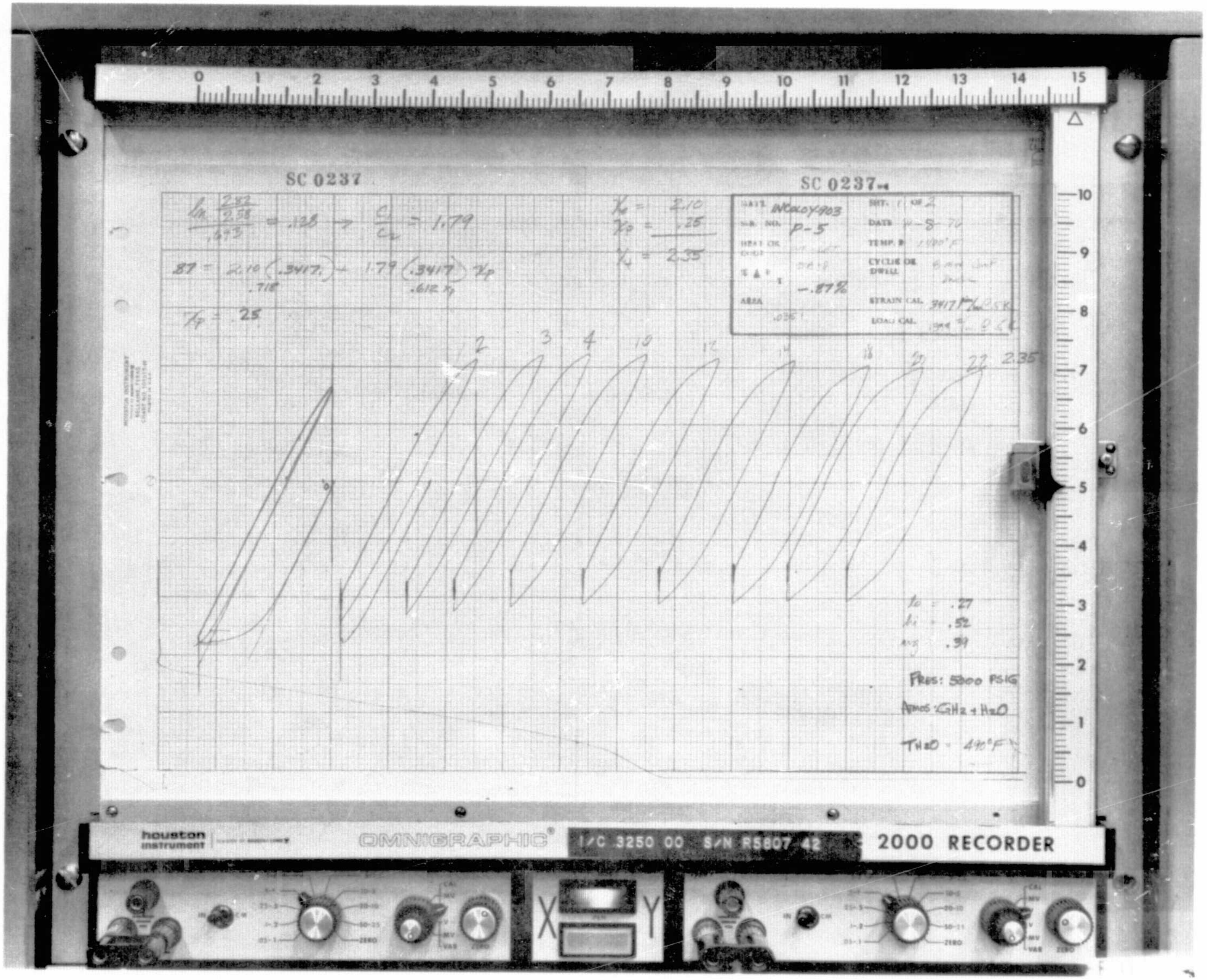
Figure IV-19. Low-Cycle Fatigue Test Retort System

Strain, as sensed by the extensometer system, was recorded on the "X" axis of an "X-Y" recorder, and load (sensed by the external load cell) was recorded on the "Y" axis, thus providing hysteresis loops, as desired, during the cyclic life of all tests. A typical series of LCF hysteresis loops is shown in figure IV-20.

Prior to test, specimens were rinsed with trichlorethylene, wiped dry, rinsed with acetone, wiped dry, and inserted into the test fixture. All handling of specimens was done with clean gloves.

Periodic checks of hydrogen test environments revealed oxygen levels less than 1 ppm. This purity level was obtained using the following test procedure:

1. Secure pressure vessel
2. Pressurize to 0.345 MN/m^2 (50 psig) with nitrogen gas and leak check
3. Vent system to 0.0345 MN/m^2 (5 psig)
4. Repressurize and vent with nitrogen gas (steps 2 and 3) two additional times
5. Pressure purge to 3.45 MN/m^2 (500 psig) with hydrogen gas
6. Evacuate pressure vessel, gas supply, and sampling system to an indicated absolute pressure of 0.00007 MN/m^2 (0.0096 psia, 500 microns)
7. Repressurize and evacuate system (steps 5 and 6) two additional times
8. Pressurize system to 3.45 MN/m^2 (500 psig) with hydrogen gas and obtain gas sample
9. Pressurize to 34.5 MN/m^2 (5,000 psig) with hydrogen gas and conduct test
10. Vent to atmospheric pressure, flow and pressure purge with nitrogen gas, open pressure vessel and remove failed specimen.



FC 36971

Figure IV-20. *Typical Series of Stress-Strain Hysteresis Loops Generated During a Low-Cycle Fatigue Test*

SECTION V
CRACK GROWTH

A. INTRODUCTION

Crack growth rate tests were conducted on Incoloy 903 and MAR-M-246 (Hf modified) at elevated temperatures in 34.5 MN/m² (5000 psig) gaseous hydrogen or hydrogen plus water vapor (50% by weight) environments. Incoloy 903 was tested at 811°K (1000°F) and 922°K (1200°F). MAR-M-246 was tested at 811°K (1000°F) and 1144°K (1600°F).

The test program was designed to generate crack growth rate (da/dn) versus cyclic stress intensity (ΔK) curves to determine the effects of temperature and environment on the crack growth properties of these materials

The Incoloy 903 material was supplied in the form of pancake forgings in the fully heat-treated condition (STA-2). MAR-M-246 (Hf modified) was supplied as plate material in two forms, conventionally cast (CC) and directionally solidified (DS). The test specimen used for all testing was the 1w compact tensile specimen as shown previously in figure III-7 incorporating a chevron-type crack-starter notch and integral knife edges per ASTM E399-74.

Crack growth tests were conducted in the load controlled mode. The test consisted of cyclically loading the specimen between the minimum load and the maximum load until complete fracture. The loading cycle was all tensile with a 491-second hold time at the maximum load. All specimens were tested at an R ratio (minimum load/maximum load) of 0.1. The test loading cycle is shown in figure V-1.

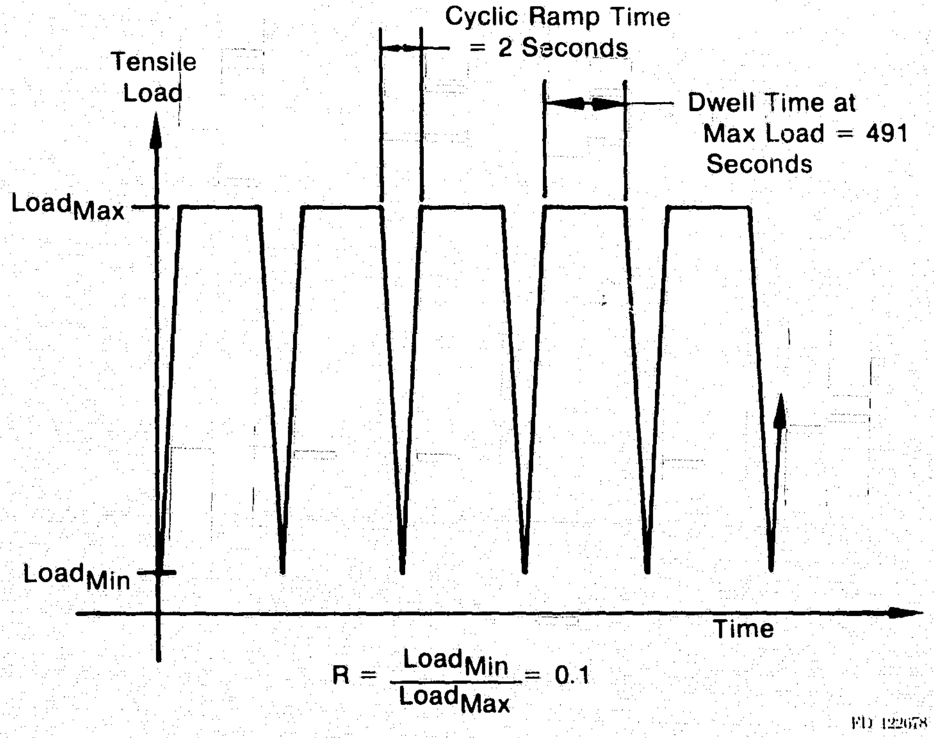


Figure V-1. Typical Crack Growth Load-Controlled Test Cycle

Crack growth data for this program was analyzed using an interpolative model developed for the analysis of elevated temperature fatigue crack propagation data.* The model has been successfully used to describe the parametric effects of three fundamental influences on crack propagation: fequency (ν), stress ratio (R), and temperature (T).

This interpolative model is based on the hyperbolic sine equation,

$$\log (da/dn) = C_1 \sinh (C_2 (\log (\Delta K) + C_3)) + C_4$$

where the coefficients are simple empirical functions of test frequency, stress ratio, and temperature:

C_1 = material constant
$C_2 = f_2 (R, \nu, T)$
$C_3 = f_3 (C_4, \nu, R)$
$C_4 = f_4 (\nu, R, T)$

This model presents a flexible alternative to the familiar Paris equation and has gained acceptance in the aerospace industry.

B. RESULTS AND CONCLUSIONS

Crack growth rate curves (da/dn vs ΔK) were generated for Incoloy 903 STA-2 at 811°K (1000°F) and 922°K (1200°F) in 34.5 MN/m^2 (5000 psig) hydrogen (figures V-2 and V-3). Testing was also conducted at 922°K (1200°F) in 34.5 MN/m^2 (5000 psig) hydrogen and water vapor (figure V-4) to define environmental degradation. A composite plot of all three temperature-environment conditions (figure V-5) revealed that there was a significant increase in the crack growth rate with the increase in test temperature, and an extremely significant increase (three orders of magnitude) in the crack growth rate due to the hydrogen-water vapor environment.

Crack growth rate curves for MAR-M-246 (Hf modified) in the conventionally cast (CC) form are presented in figures V-6 through V-9. Testing was performed at 811°K (1000°F) and 1144°K (1600°F) in 34.5 MN/m^2 (5000 psig) hydrogen, and at 1144°K (1600°F) in 34.5 MN/m^2 (5000 psig) hydrogen and water vapor (50% by weight). As with Incoloy 903, the MAR-M-246 CC material exhibited a considerable increase in crack growth rate with increasing temperature. However, the effect of the hydrogen-water vapor environment was negligible when compared with the hydrogen environment.

The MAR-M-246 (Hf modified) material in the directionally solidified (DS) form was tested at conditions similar to the CC material, and the test results are plotted in figures V-10 through V-13. Increasing temperature and addition of water to the hydrogen environment both caused significant increases in the crack growth rate. The effect on crack growth of increasing temperature was greater for the CC material, but the effect of the water vapor environment was greater for the DS material.

Figure V-14 presents a composite plot of all 9 material/temperature/environment conditions tested. Complete crack growth test conditions and results are listed in table V-1.

---

*Annis, C. G., R. M. Wallace, and D. L. Sims, "An Interpolative Model for Elevated Temperature Fatigue Crack Propagation," Final Report No. AFML-TR-76-176, November 1976.

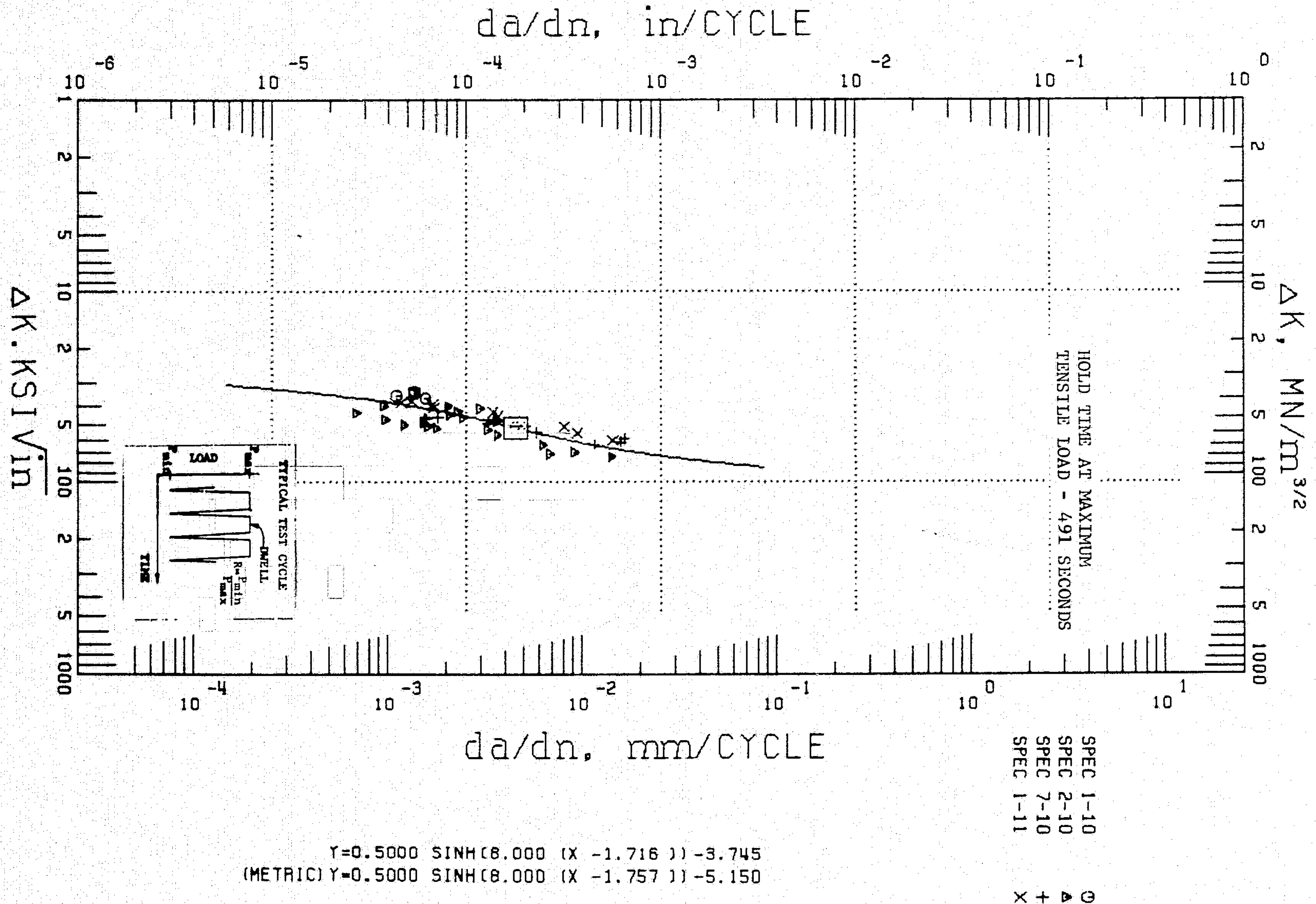


Figure V-2. Crack Growth Rate vs Stress Intensity for Incoloy 903 STA-2 in 34.5 MN/m² (5000 psig) Hydrogen at 811°K (1000°F)

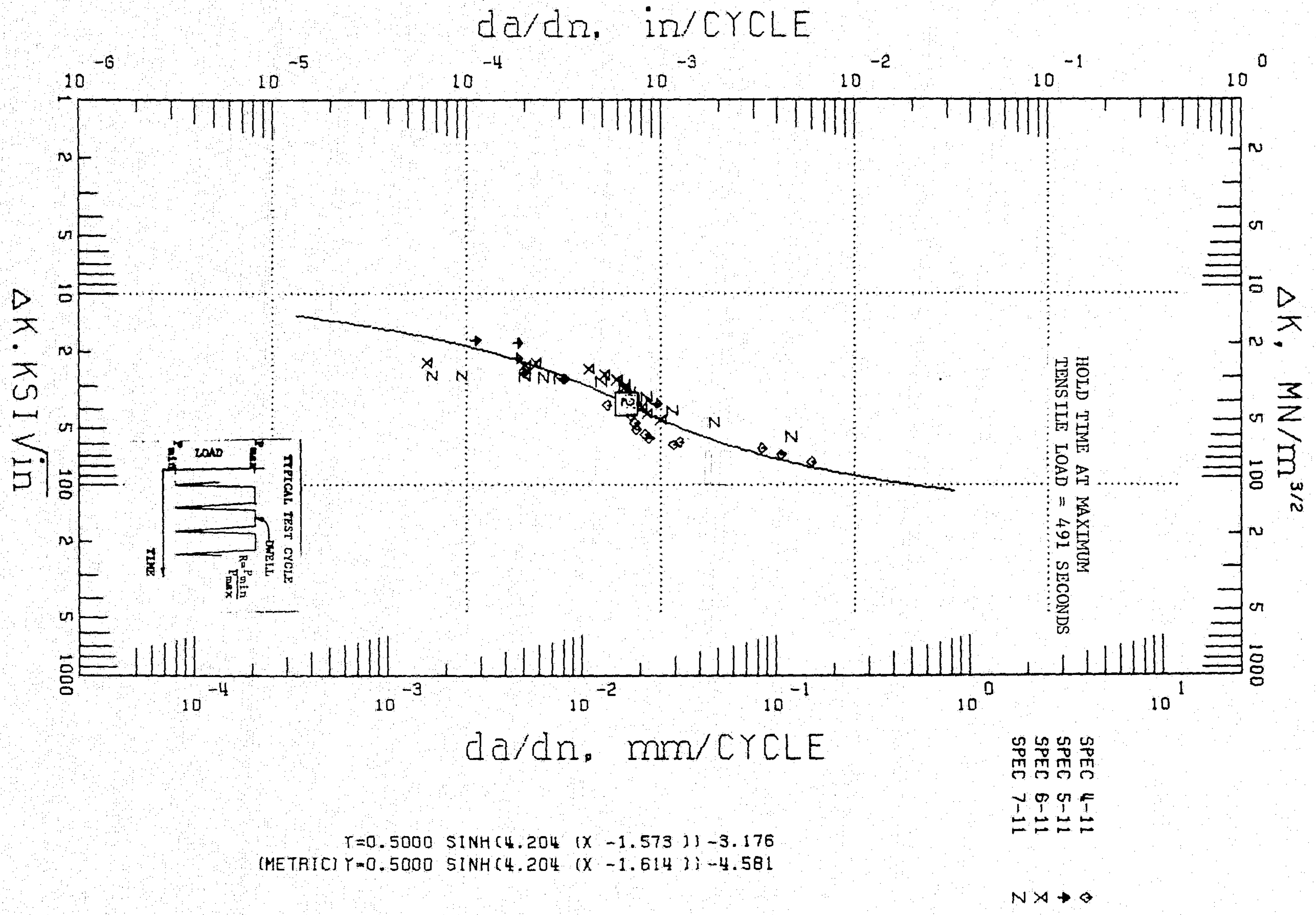


Figure V-3. Crack Growth Rate vs Stress Intensity for Incoloy 903 STA-2 in 34.5 MN/m² (5000 psig) Hydrogen at 922°K (1200°F)

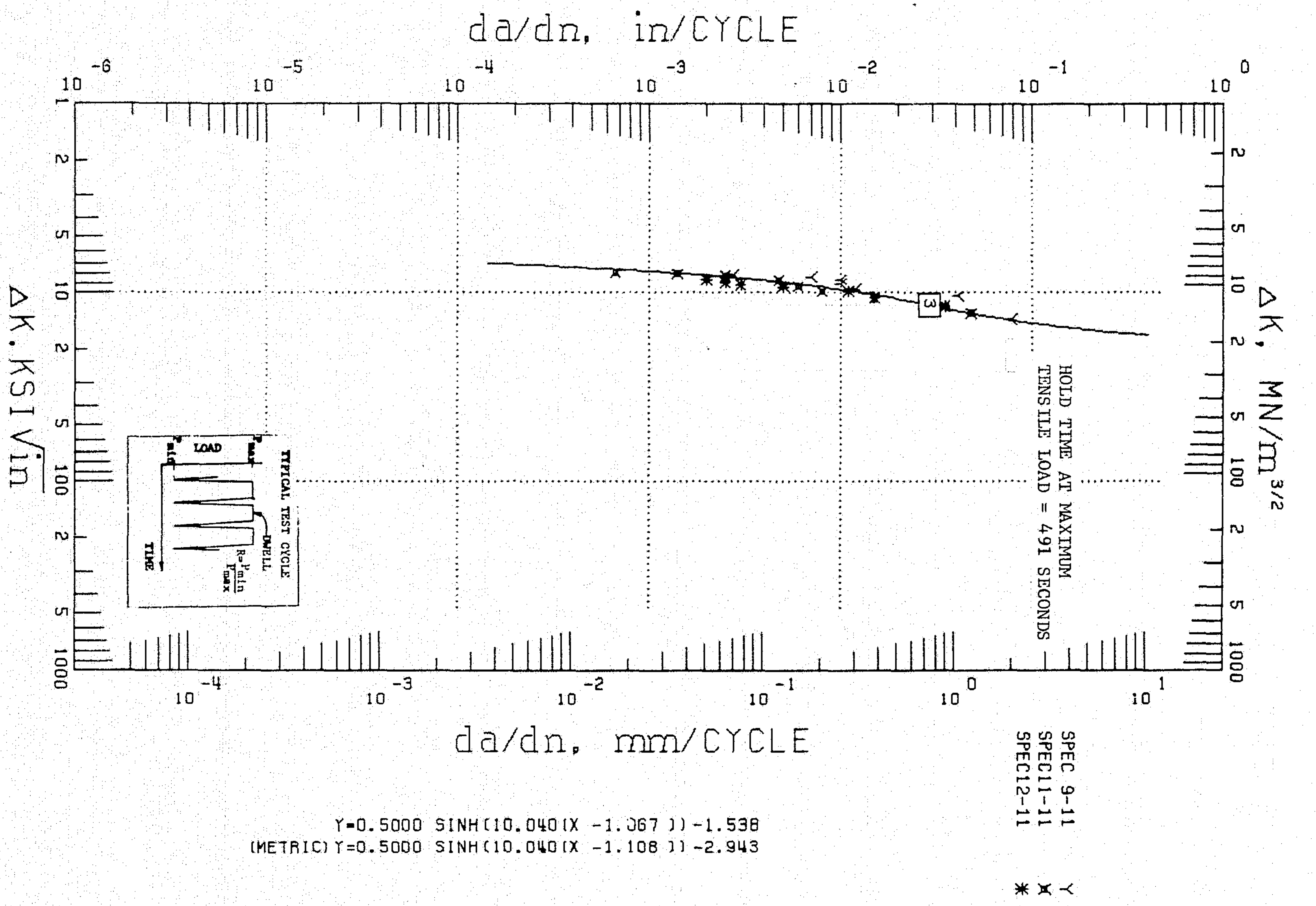
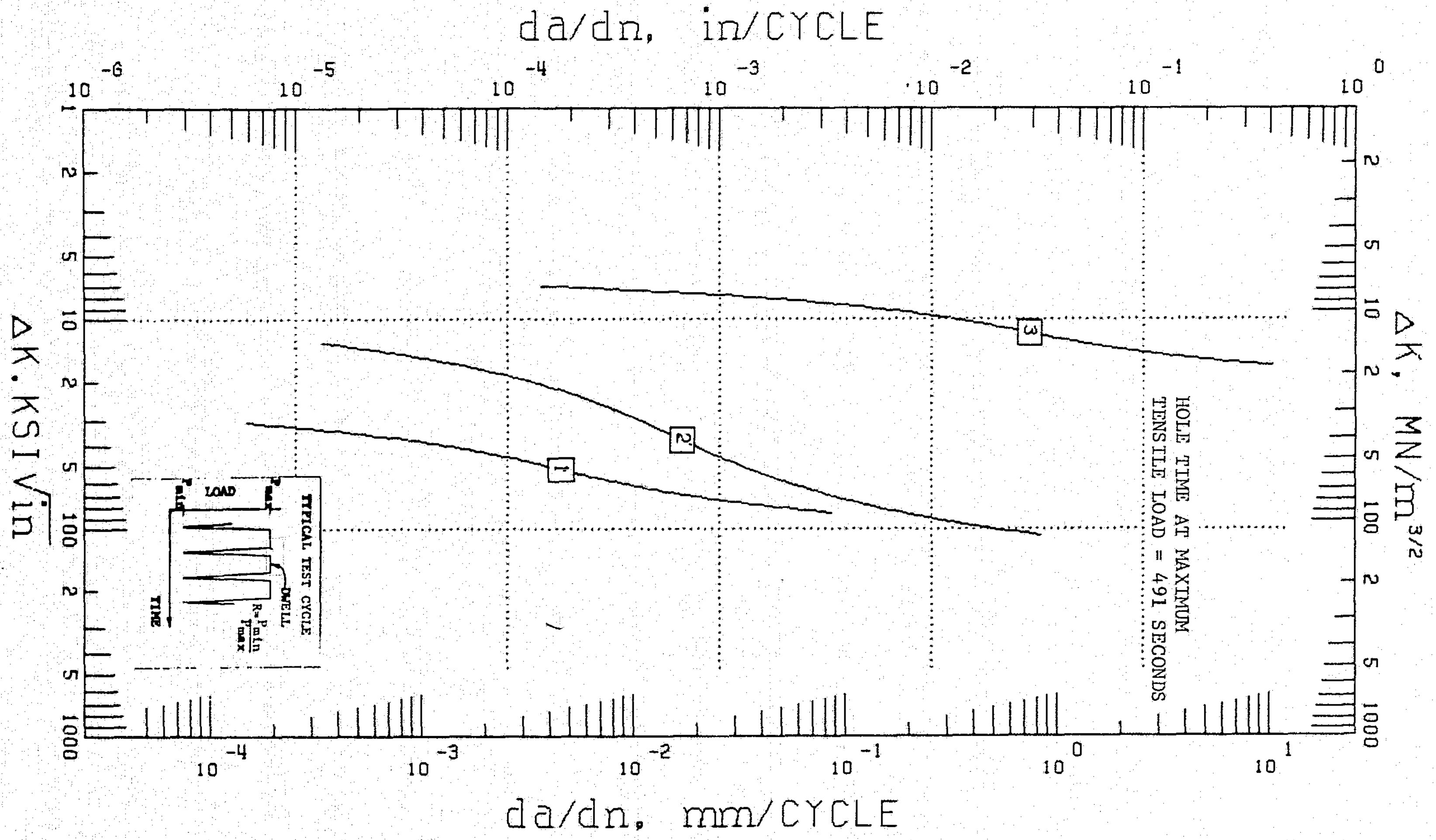


Figure V-4. Crack Growth Rate vs Stress Intensity for Incoloy 903 STA-2 in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 922°K (1200°F)



1 — Y=0.5000 SINH(8.000 (X -1.716)) -3.745 (METRIC) Y=0.5000 SINH(8.000 (X -1.757)) -5.150 — 811°K (1000°F) GH_2

2 — Y=0.5000 SINH(4.204 (X -1.573)) -3.176 (METRIC) Y=0.5000 SINH(4.204 (X -1.614)) -4.581 — 922°K (1200°F) GH_2

3 — Y=0.5000 SINH(10.040 (X -1.067)) -1.538 (METRIC) Y=0.5000 SINH(10.040 (X -1.108)) -2.943 — 922°K (1200°F) $GH_2 + H_2O$

Figure V-5. Crack Growth Rate vs Stress Intensity for Incoloy 903 STA-2 in 34.5 MN/m² (5000 psig) Gaseous Environments at 811°K (1000°F) and 922°K (1200°F)

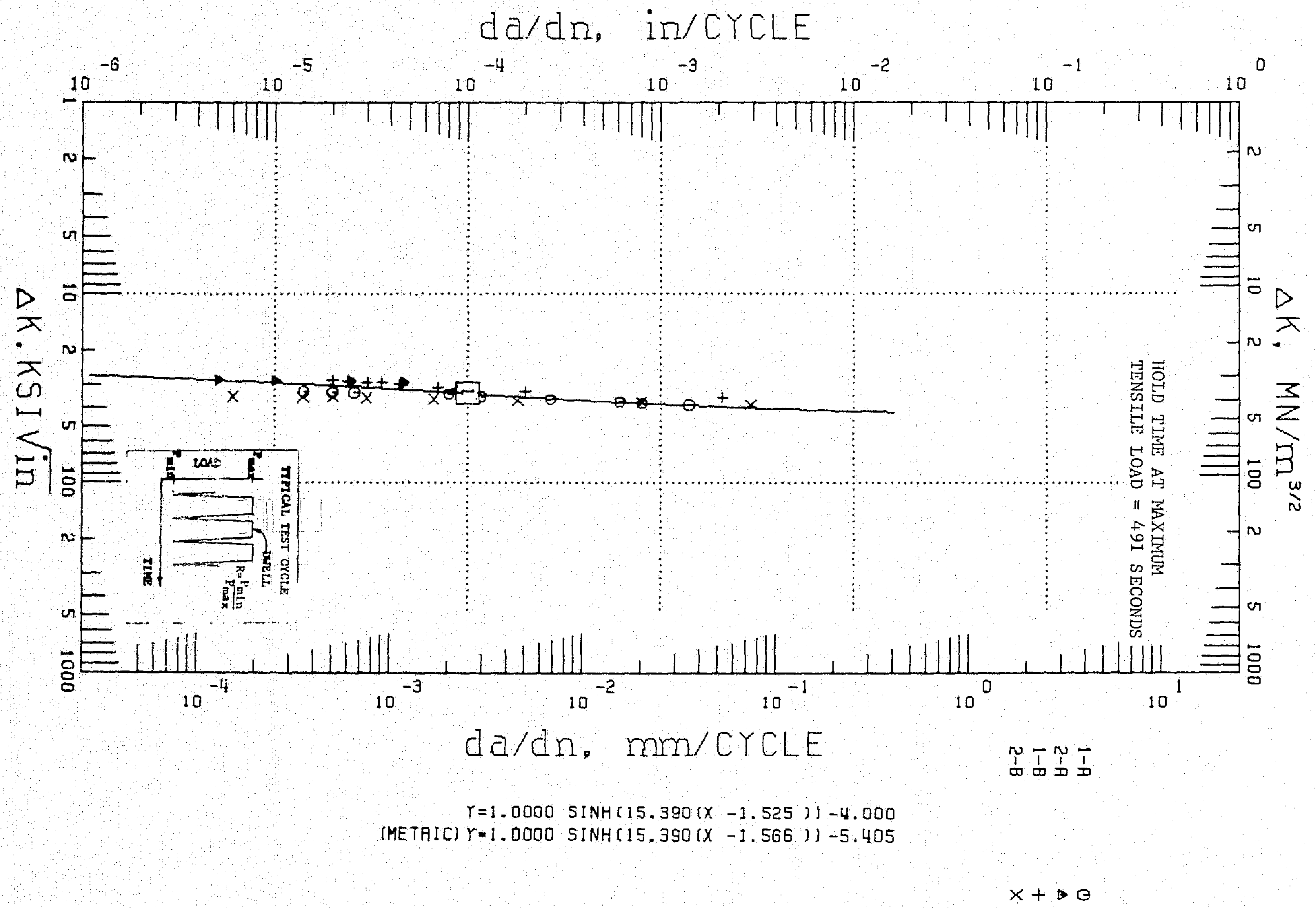


Figure V-6. *Crack Growth Rate vs Stress Intensity for Conventionally Cast MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen at 811°K (1000°F)*

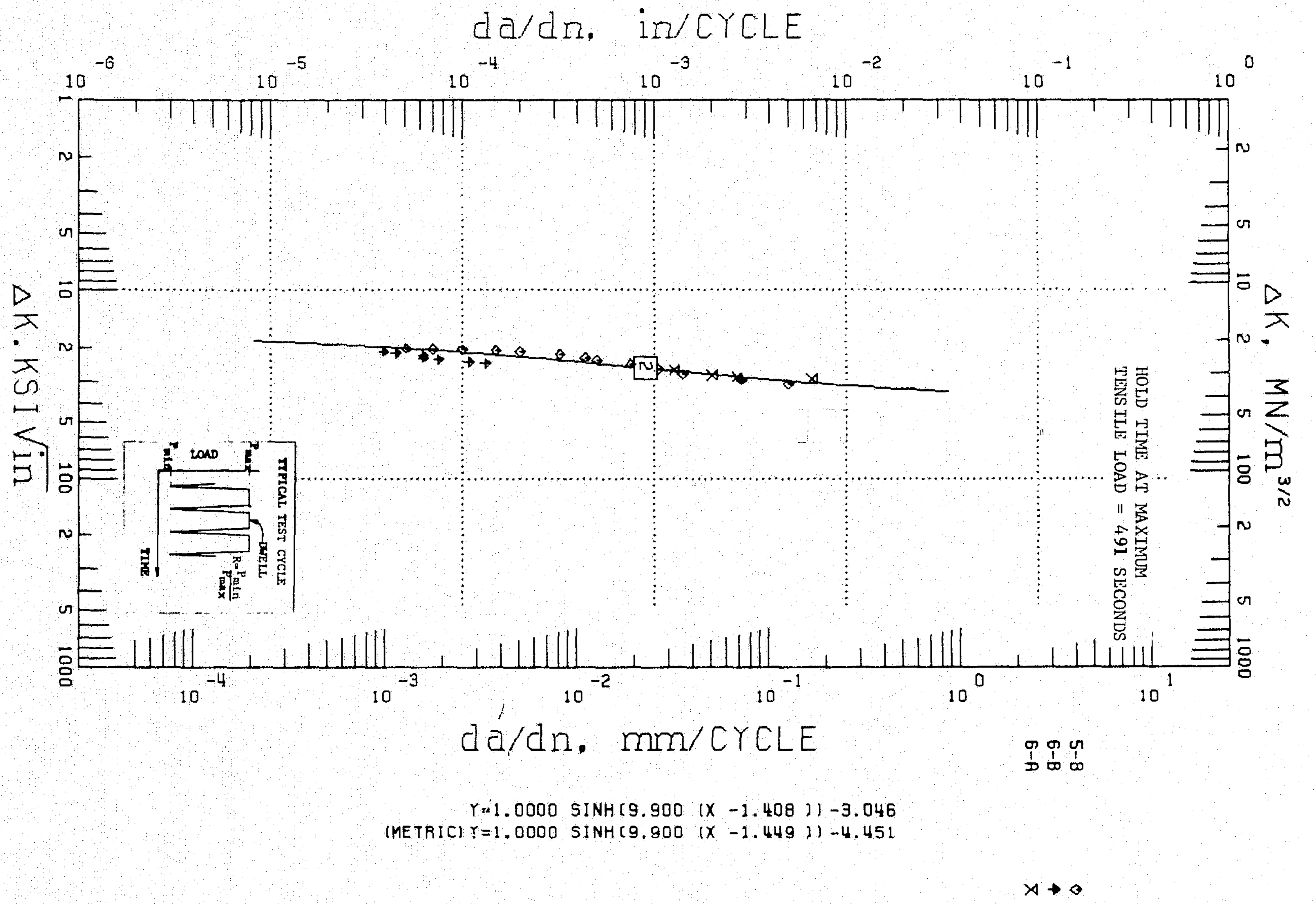


Figure V-7. Crack Growth Rate vs Stress Intensity for Conventionally Cast MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen at 1144°K (1600°F)

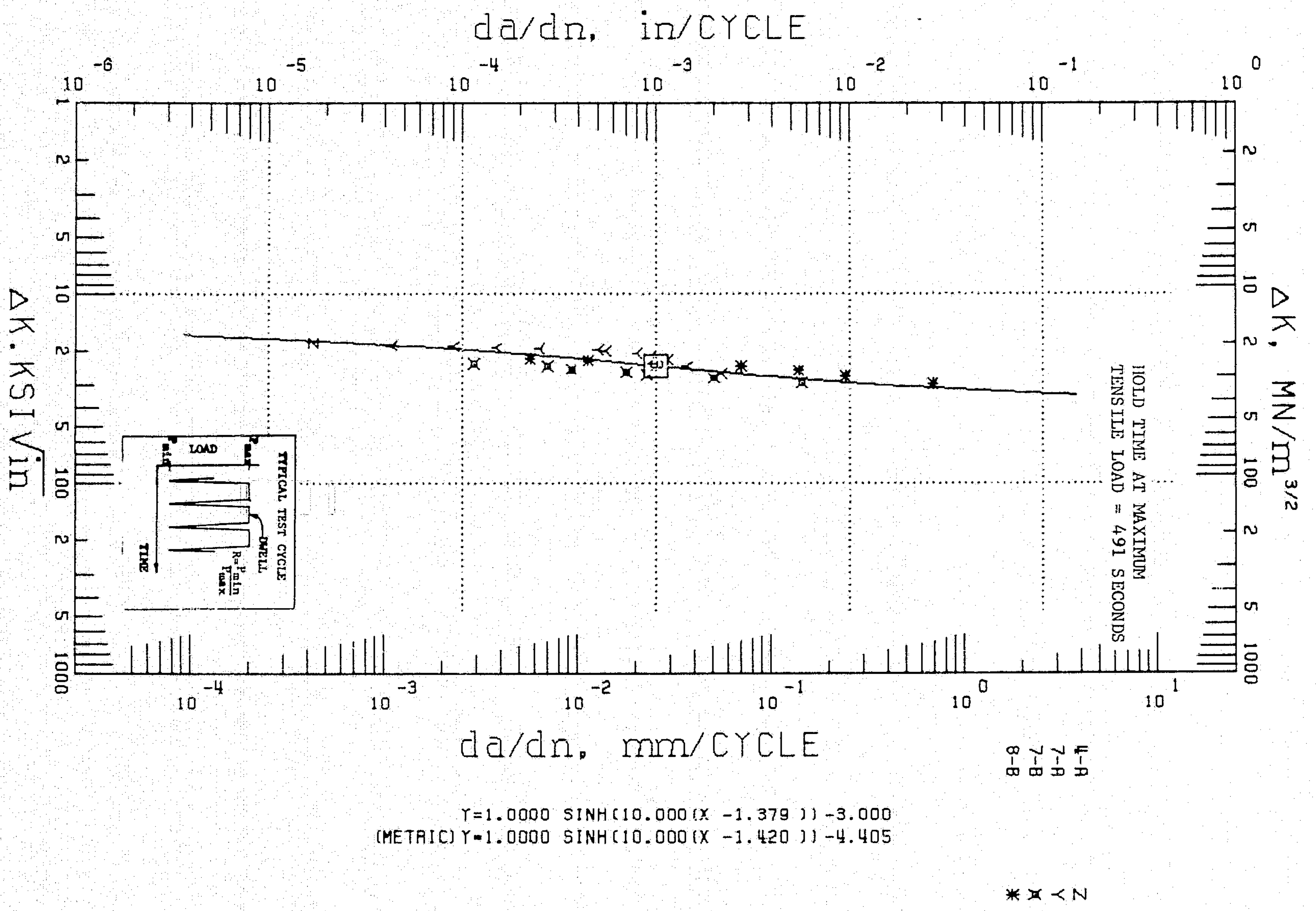


Figure V-8. *Crack Growth Rate vs Stress Intensity for Conventionally Cast MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 1144°K (1600°F)*

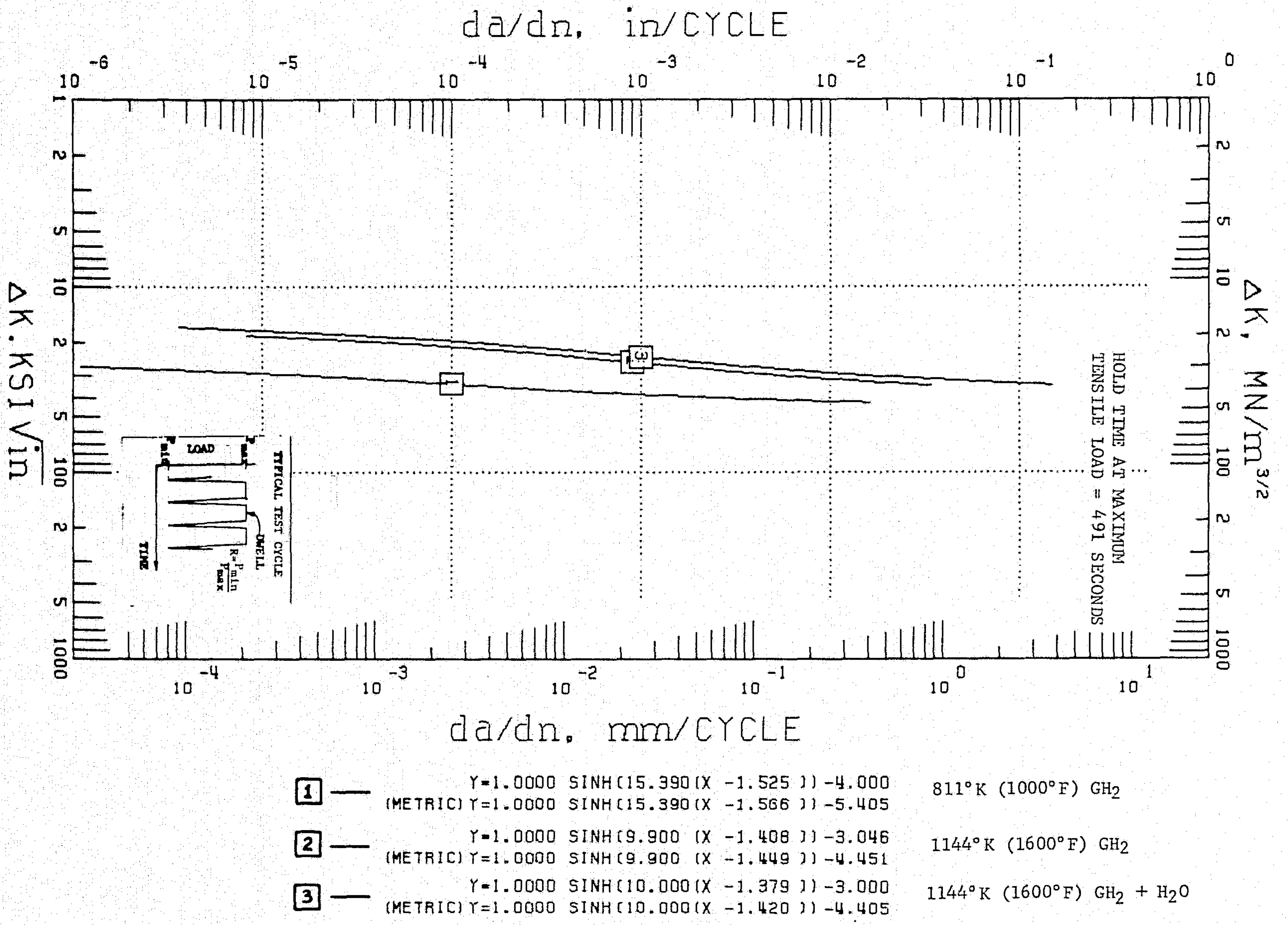


Figure V-9. Crack Growth Rate vs Stress Intensity for Conventionally Cast MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Gaseous Environments at 811°K (1000°F) and 1144°K (1600°F)

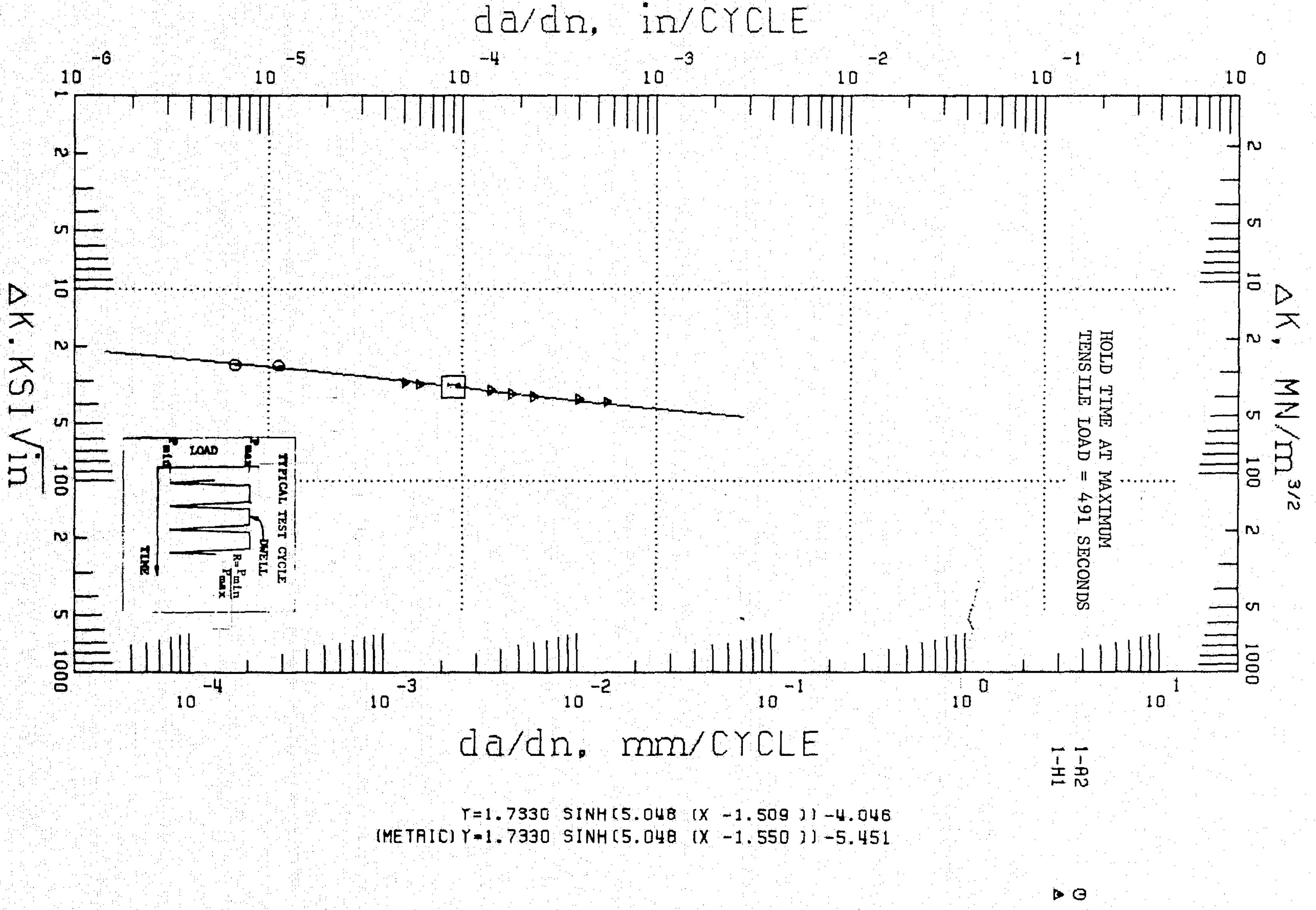


Figure V-10. *Crack Growth Rate vs Stress Intensity for Directionally Solidified MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen at 811°K (1000°F)*

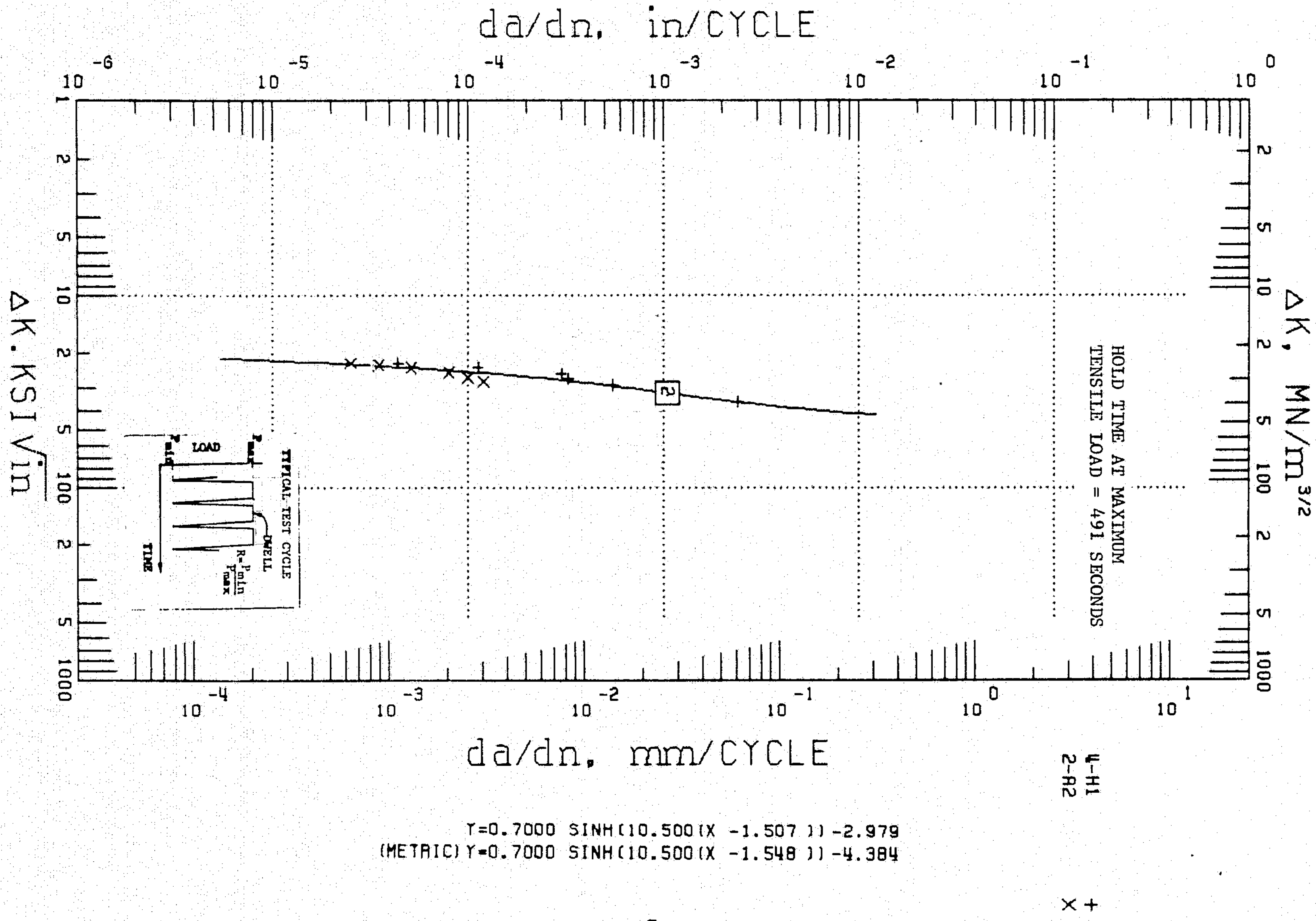


Figure V-11. *Crack Growth Rate vs Stress Intensity for Directionally Solidified MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen at 1144°K (1600°F)*

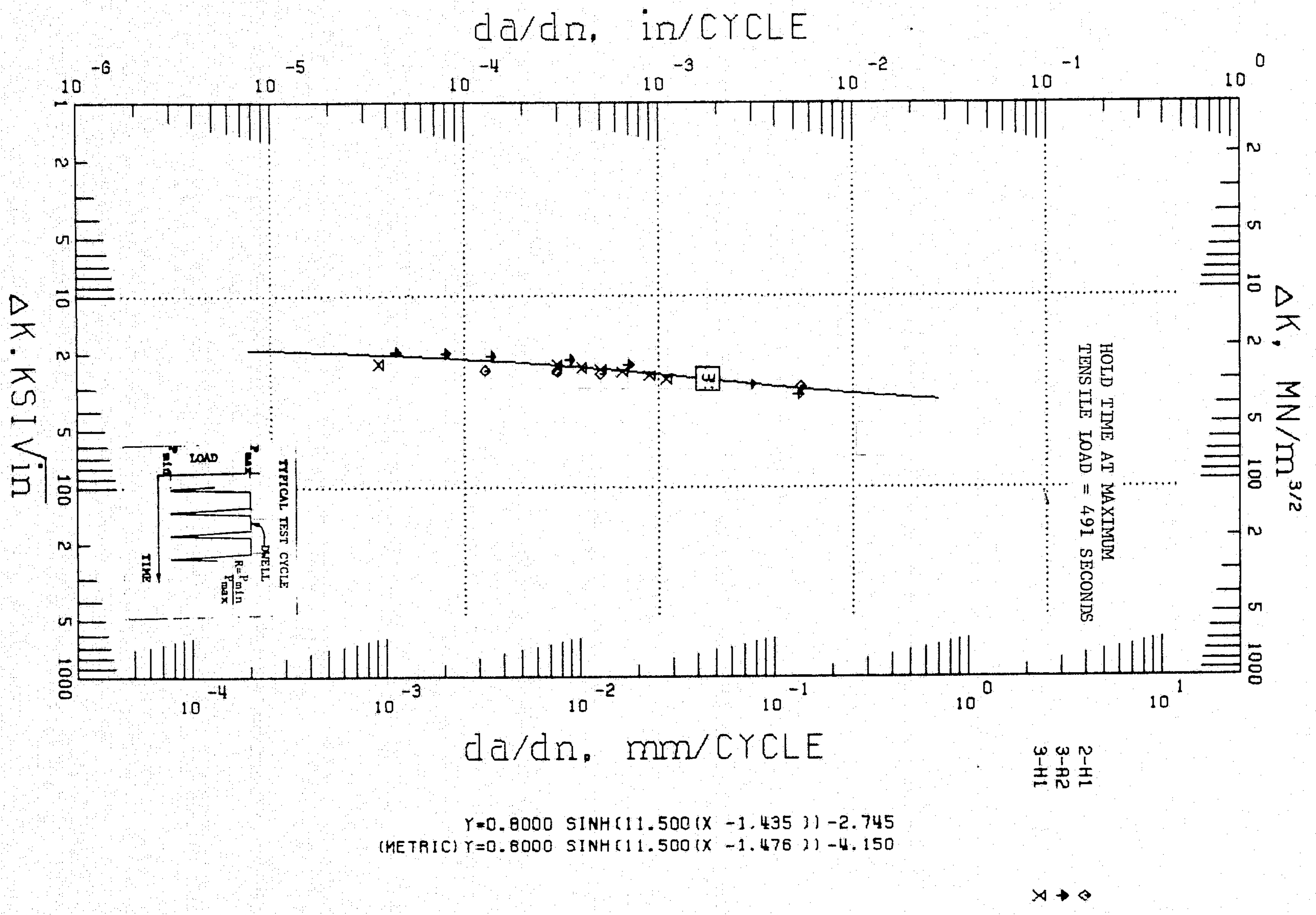
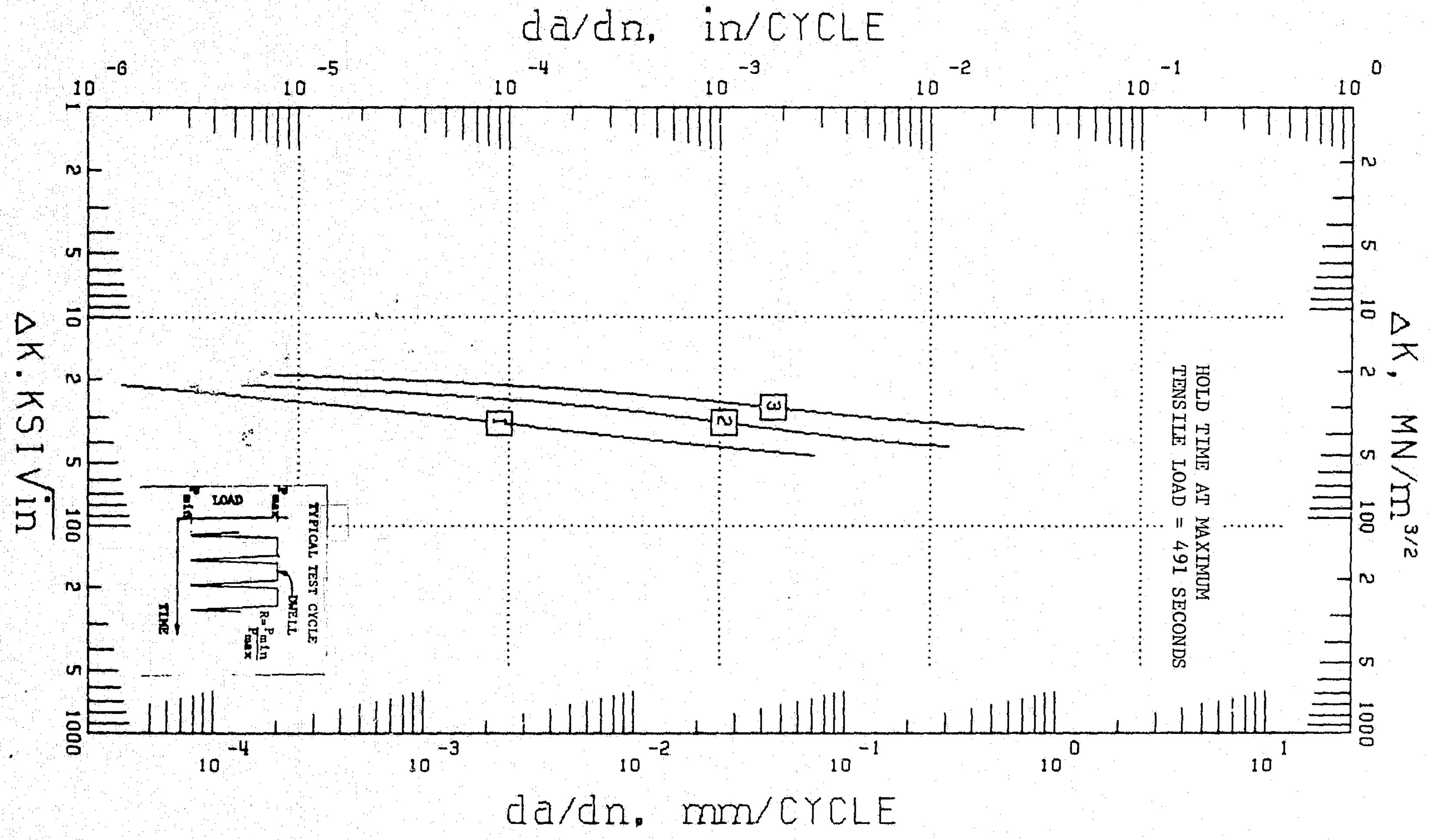


Figure V-12. Crack Growth Rate vs Stress Intensity for Directionally Solidified MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Hydrogen and Water Vapor at 1144°K (1600°F)



1 —	Y=1.7330 SINH(5.048 (X -1.509)) -4.046 (METRIC) Y=1.7330 SINH(5.048 (X -1.550)) -5.451	811°K (1000°F) GH_2
2 —	Y=0.7000 SINH(10.500(X -1.507)) -2.979 (METRIC) Y=0.7000 SINH(10.500(X -1.548)) -4.384	1144°K (1600°F) GH_2
3 —	Y=0.8000 SINH(11.500(X -1.435)) -2.745 (METRIC) Y=0.8000 SINH(11.500(X -1.476)) -4.150	1144°K (1600°F) $GH_2 + H_2O$

Figure V-13. Crack Growth Rate vs Stress Intensity for Directionally Solidified MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Gaseous Environments at 811°K (1000°F) and 1144°K (1600°F)

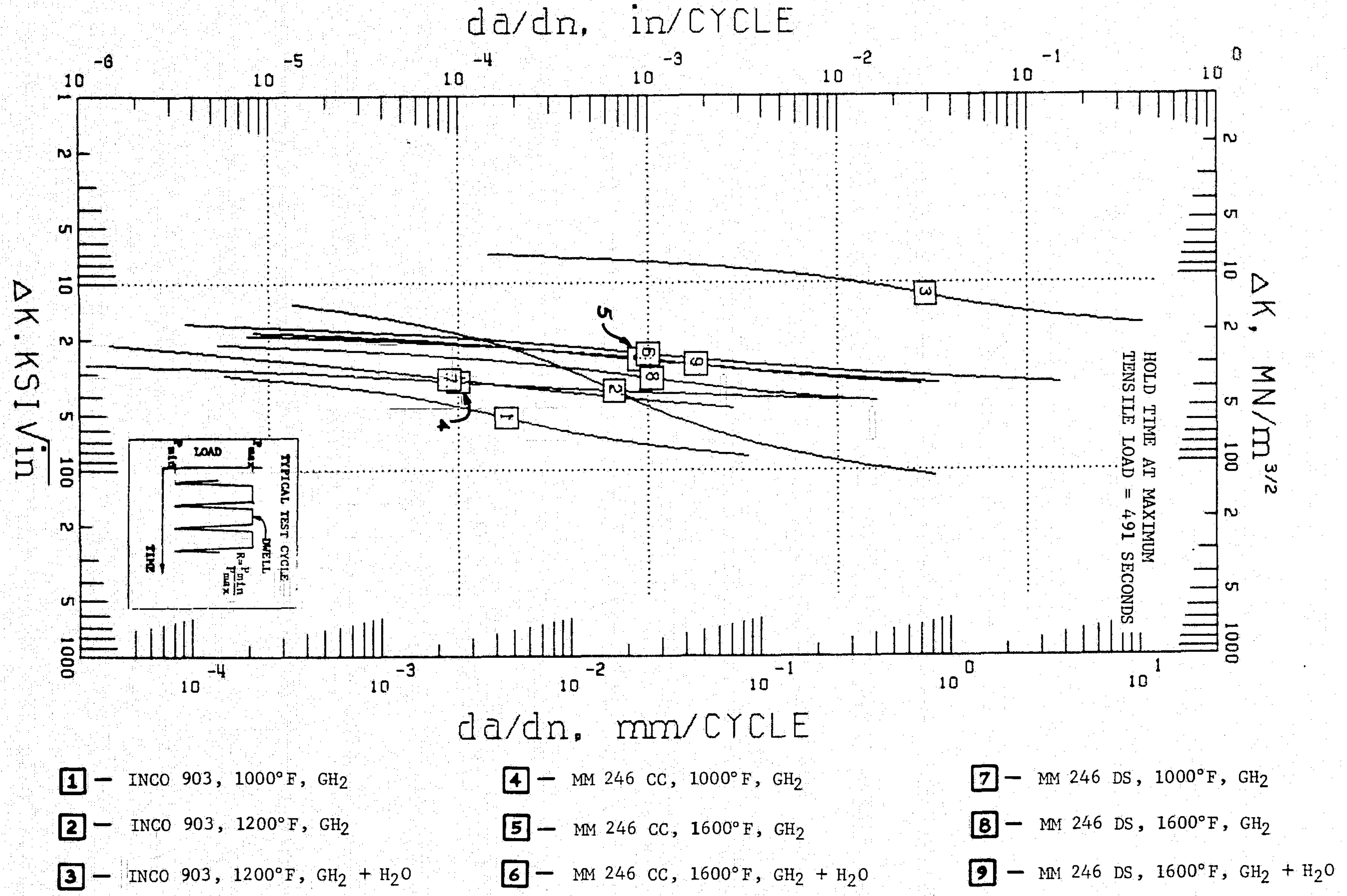


Figure V-14. *Crack Growth Rate vs Stress Intensity for Incoloy 903 and MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Gaseous Environments*

Table V-1. Crack Growth Rate Testing of Incoloy 903 and MAR-M-246 (Hf Modified) in 34.5 MN/m² (5000 psig) Gaseous Environments

Test Material				Test Conditions								Test Results
				Test Temperature			Initial Stress Intensity		Max Load			
Material	Condition[1]	Heat No.	Specimen S/N	°K	°F	Environment[2]	$MN/m^{3/2}$	Ksi √in	N	lb	Dwell Time[3] (Seconds)	Cycles To Failure
Incoloy 903	STA-2	91178	1-10	811	1000	GH_2	39.9	36.3	8,354	1,878	491	699[4]
Incoloy 903	STA-2	91178	2-10	811	1000	GH_2	47.9	43.6	9,475	2,130	491	1592
Incoloy 903	STA-2	91178	7-10	811	1000	GH_2	55.6	50.6	11,054	2,485	491	532
Incoloy 903	STA-2	91178	1-11	811	1000	GH_2	45.4	41.3	8,910	2,003	491	976
Incoloy 903	STA-2	91178	4-11	922	1200	GH_2	45.2	41.1	8,416	1,892	491	181
Incoloy 903	STA-2	91178	7-11	922	1200	GH_2	32.2	29.3	6,352	1,428	491	268
Incoloy 903	STA-2	91178	5-11	922	1200	GH_2	21.3	19.4	4,177	939	491	883
Incoloy 903	STA-2	91178	6-11	922	1200	GH_2	27.7	25.2	4,938	1,110	491	333
Incoloy 903	STA-2	91178	9-11	922	1200	GH_2+H_2O	9.7	8.8	1,979	445	491	13
Incoloy 903	STA-2	91178	10-11	922	1200	GH_2+H_2O	10.5	9.6	1,993	448	491	57[5]
Incoloy 903	STA-2	91178	11-11	922	1200	GH_2+H_2O	9.6	8.7	1,842	414	491	27
Incoloy 903	STA-2	91178	12-11	922	1200	GH_2+H_2O	10.3	9.4	1,895	426	491	18
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	1-A	811	1000	GH_2	39.9	36.3	6,672	1,500	491	537
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	2-B	811	1000	GH_2	42.3	38.5	6,672	1,500	491	511
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	2-A	811	1000	GH_2	31.2	28.4	5,560	1,250	491	395[4]
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	1-B	811	1000	GH_2	31.2	28.4	5,035	1,132	491	1729[6]
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE					34.7	31.6	5,560	1,250	491	891
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	5-B	1144	1600	GH_2	24.5	22.3	4,448	1,000	491	279
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	4-B	1144	1600	GH_2	26.4	24.0	4,448	1,000	491	2[4]
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	6-B	1144	1600	GH_2	25.1	22.8	4,448	1,000	491	838
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	6-A	1144	1600	GH_2	28.7	26.1	4,448	1,000	491	42
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	7-A	1144	1600	GH_2+H_2O	22.6	20.6	3,559	800	491	244
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	4-A	1144	1600	GH_2+H_2O	22.1	20.1	3,559	800	491	119[4]
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	7-B	1144	1600	GH_2+H_2O	28.2	25.7	4,448	1,000	491	149
MAR-M-246 (Hf Modified)	Conventionally Cast	L99-HBE	8-B	1144	1600	GH_2+H_2O	26.4	24.0	4,448	1,000	491	70
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	1-A2	811	1000	GH_2	30.0	27.3	3,559	800	491	653[4]
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	1-H1	811	1000	GH_2	37.2	33.9	6,583	1,480	491	518
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	4-H1	1144	1600	GH_2	26.9	24.5	4,893	1,100	491	351
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	2-A2	1144	1600	GH_2	27.1	24.7	4,893	1,100	491	577[6]
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002					32.1	29.2	5,338	1,200	491	338
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	2-H1	1144	1600	GH_2+H_2O	29.6	26.9	4,448	1,000	491	92
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	3-A2	1144	1600	GH_2+H_2O	23.7	21.6	4,270	960	491	374
MAR-M-246 (Hf Modified)	Directionally Solidified	DE-002	3-H1	1144	1600	GH_2+H_2O	27.3	24.8	4,737	1,065	491	248

Notes:

[1] Condition:

STA-2 — 1227°K (1750°F) 1hr. AC + 991°K (1325°F) 8 hr. + 894°K (1150°F) 8 hr (with Total Age Time = 18 hr) AC

Conventionally Cast — As Cast

Directionally Solidified — 1494°K (2230°F) 2 hr. FC + 1144°K (1600°F) 24 hr. FC

[2] Environment:

GH_2 — Gaseous hydrogen (with oxygen content less than 1 part per million)

GH_2+H_2O — 50% gaseous hydrogen + 50% water vapor by weight

[3] Dwell Time is the period of time the specimen is held at the maximum tensile load

[4] Electrical Power Failure caused overstress and premature failure

[5] Temperature Controller malfunction - entire test not run at specified temperature

[6] Specimen had no appreciable growth at this stress; test load was increased and test continued until failure

[7] Max Load is as tabulated; Min load = 10% of Max load. R = $\text{Load}_{min}/\text{Load}_{max}$ = 0.1.

C. TEST PROCEDURE

The specimen configuration used for crack growth testing was the 1w compact tensile specimen detailed in figure III-7. This specimen incorporated a chevron-type crack-starter notch and integrally machined knife edges for Crack Opening Displacement (COD) extensometry attachment as recommended by ASTM E399-74, "Plane-Strain Fracture Toughness of Metallic Materials." Specimen thickness was chosen to conform to supplied raw material dimensions, and to the high pressure test vessel retort size.

Incoloy 903 STA-2 specimens were machined from pancake forgings such that crack growth properties in the radial direction would be obtained (C-R orientation per ASTM E399-74). The MAR-M-246 (Hf modified) DS material was machined such that crack growth properties could be determined parallel to the primary gain direction (see figure III-2; T-L orientation per ASTM E399-74).

A compliance calibration was conducted to relate the COD measured by the test extensometry to the test specimen crack length. A compliance calibration specimen from each material and/or form was machined. The compliance between the measured COD and the handbook* prediction was compared at various crack length, load, and temperature conditions for each specimen. Results are presented in table V-2 and figure V-15. The measured COD agreed well with the handbook predictions, and the handbook relationship was used for all environmental testing.

The compliance calibrations were conducted in air at atmospheric pressure. It was assumed that the elevated temperature, high-pressure hydrogen test environments would cause crack-tip embrittlement which should ensure that the handbook assumptions of plane strain and linear elasticity would be met. Examination of the fracture appearance of tested specimens confirmed that the assumptions of plane strain were valid. Very small shear lips were formed (% oblique ≈ 5%), indicative of a small crack-tip plastic zone when compared to crack length and specimen dimensions (figure III-2).

Stress intensity values (K) were calculated from the equation:

$$K = \frac{P}{BW^{1/2}}\left[29.6\left(\frac{a}{W}\right)^{1/2} - 185.5\left(\frac{a}{W}\right)^{3/2} + 655.7\left(\frac{a}{W}\right)^{5/2} \quad 1017.0\left(\frac{a}{W}\right)^{7/2} + 638.9\left(\frac{a}{W}\right)^{9/2}\right]$$

where

K = stress intensity (psi-in.$^{1/2}$)
P = applied load (pounds)
B = specimen thickness (inches)
W = specimen width (inches)
a = crack length (inches),

and cyclic stress intensity (ΔK) was calculated by the equation:

$$\Delta K = K_{max} - K_{min} = (1-R)\,K_{max} = 0.9\,K_{max}$$

---

*Tada, H., P. C. Paris, and G. R. Irwin, "The Stress Analysis of Cracks Handbook," Del Research Corporation, Hellertown, Pennsylvania, 1973.

where

ΔK = cyclic stress intensity (psi-in.$^{1/2}$)
K_{max} = stress intensity at max cyclic load
K_{min} = stress intensity at min cyclic load

with

R = 0.1 (R = minimum load/maximum load)

Test specimens were axial tension-tension fatigue precracked at 300°K (80°F) using a fatigue machine operating at a frequency of 30 Hz (1800 cpm) and R ratio = 0.1. Maximum precrack loads were approximately 80% of the maximum loads used during crack growth testing.

High-pressure environmental tests were conducted on a closed-loop, hydraulically actuated test machine located in an isolated test cell (figure V-16). The pressure vessel with test frame and hydraulic actuator system are shown in figure V-17. The pressure vessel was similar to the LCF vessel, and is shown in figures V-18 and V-19.

The test machine compensates for internal gas pressure loading of the test specimen through a pressure transducer feedback signal to the servosystem. The signal is used to control a steady force to the load linkage that is equal and opposite to the internal hydrogen pressure load against the load rod.

Table V-2. Crack Length Vs Crack Opening Displacement (COD) Compliance Calibration Data for Incoloy 903 and MAR-M-246 (Hf Modified)

								Crack Opening Displacement			
	Specimen	*Temperature*		*Calibration Load*		*Measured Average Crack Length*		*Experimental Measurement*		*Handbook[1] Prediction*	
Material	*S/N*	*°K*	*°F*	*N*	*lb*	*mm*	*inch*	*mm*	*inch*	*mm*	*inch*
Incoloy 903 STA 2	1	300	80	11120	2500	12.67	0.499	0.272	0.0107	0.264	0.0104
		300	80	11120	2500	13.87	0.546	0.345	0.0136	0.328	0.0129
		300	80	11120	2500	15.06	0.593	0.422	0.0166	0.417	0.0164
		700	800	6672	1500	15.06	0.593	0.249	0.0098	0.244	0.0096
		811	1000	6672	1500	15.06	0.593	0.254	0.0100	0.246	0.0097
		922	1200	6672	1500	15.06	0.593	0.267	0.0105	0.254	0.0100
	3	300	80	4448	1000	13.54	0.533	0.117	0.0046	0.124	0.0049
		300	80	4448	1000	14.91	0.587	0.152	0.0060	0.163	0.0064
		300	80	4448	1000	16.43	0.647	0.216	0.0085	0.229	0.0090
		300	80	4448	1000	17.20	0.677	0.264	0.0104	0.277	0.0109
		300	80	3336	750	18.29	0.720[2]	0.272	0.0107	0.284	0.0112
MAR-M-246 (Hf Modified) Conventionally Cast	9-B	300	80	4448	1000	13.23	0.521	0.114	0.0045	0.117	0.0046
		300	80	3336	750	14.27	0.562	0.107	0.0042	0.107	0.0042
		300	80	2669	600	15.21	0.599	0.102	0.0040	0.104	0.0041
		811	1000	2669	600	15.21	0.599	0.114	0.0045	0.122	0.0048
		300	80	2224	500	16.33	0.643	0.104	0.0041	0.112	0.0044
MAR-M-246 (Hf Modified) Directionally Solidified	6-A2	300	80	3559	800	13.79	0.543	0.107	0.0042	0.104	0.0041
		811	1000	3114	700	13.79	0.543	0.104	0.0041	0.104	0.0041
		300	80	3336	750	14.45	0.569	0.114	0.0045	0.112	0.0044
		811	1000	3336	750	14.45	0.569	0.127	0.0050	0.127	0.0050
		300	80	2669	600	15.67	0.617	0.117	0.0046	0.117	0.0046
		811	1000	2669	600	15.67	0.617	0.130	0.0051	0.132	0.0052
		300	80	2669	600	16.18	0.637	0.127	0.0050	0.130	0.0051

---

[1] *The Stress Analysis of Cracks Handbook;* H. Tada, with cooperation of P. C. Paris and G. R. Irwin, Del Research Corporation, Hellertown, Pennsylvania, 1973.

[2] Near boundary of valid test range for specimen.

Note: All compliance tests conducted in air at atmospheric pressure.

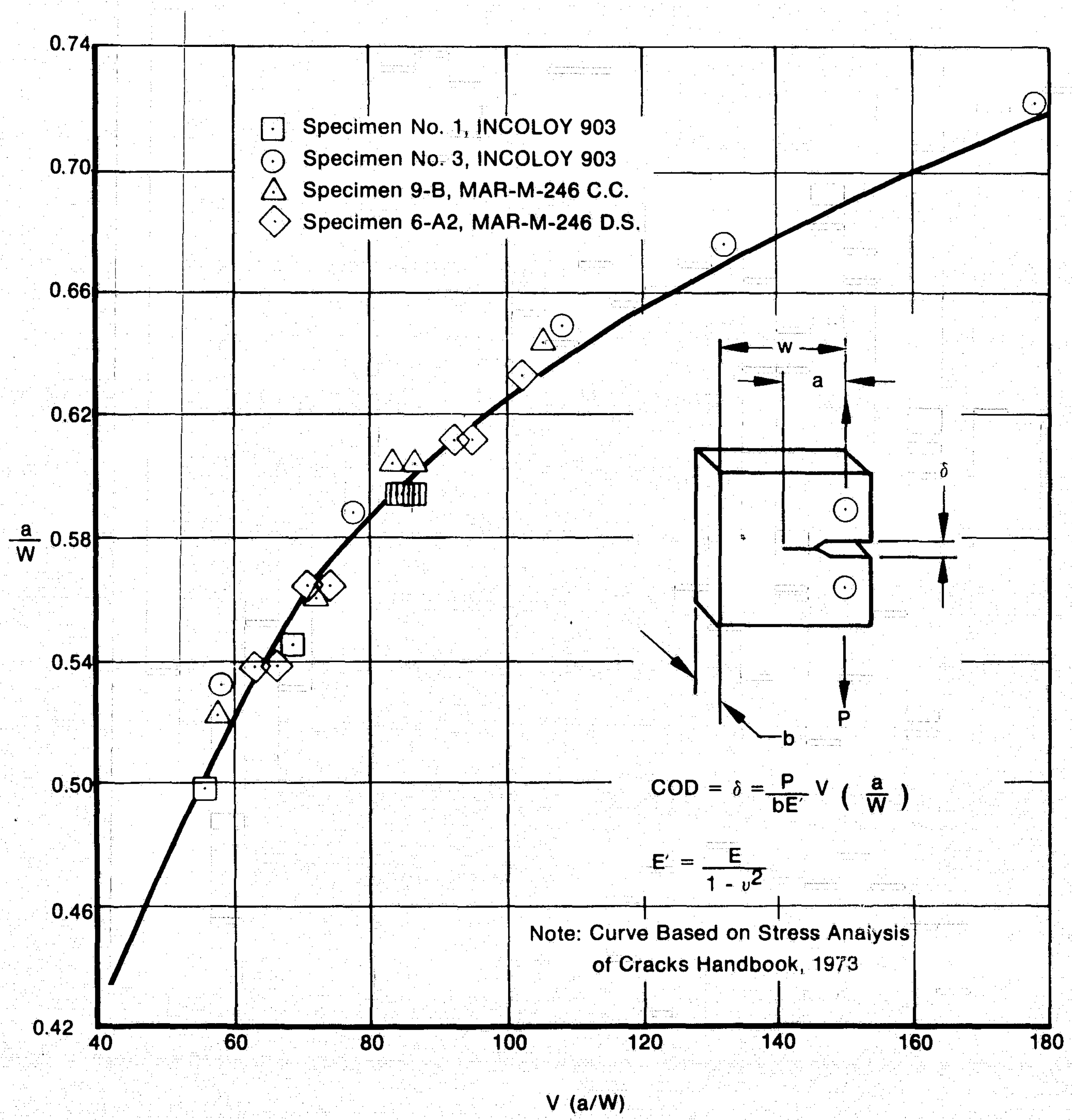
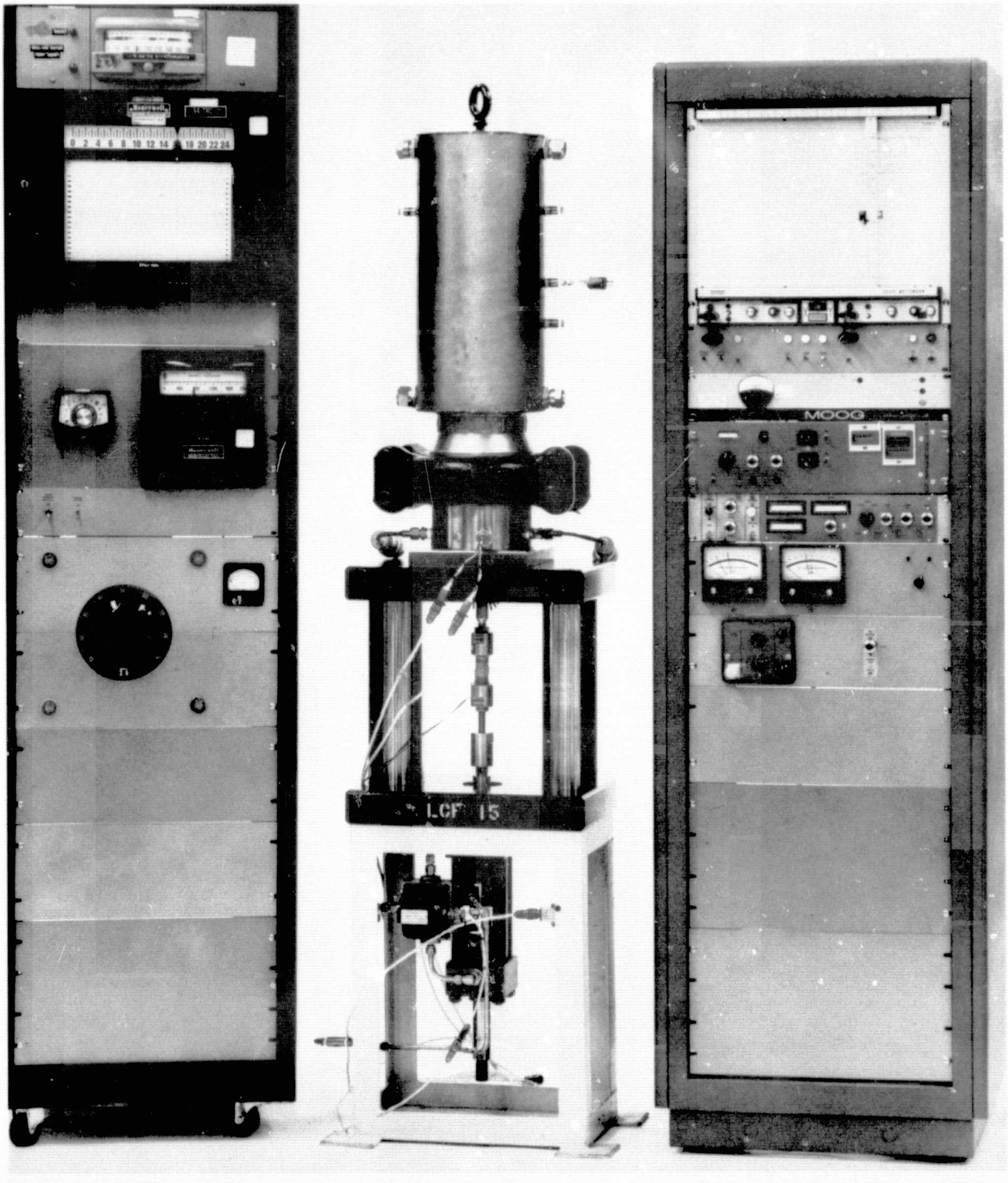
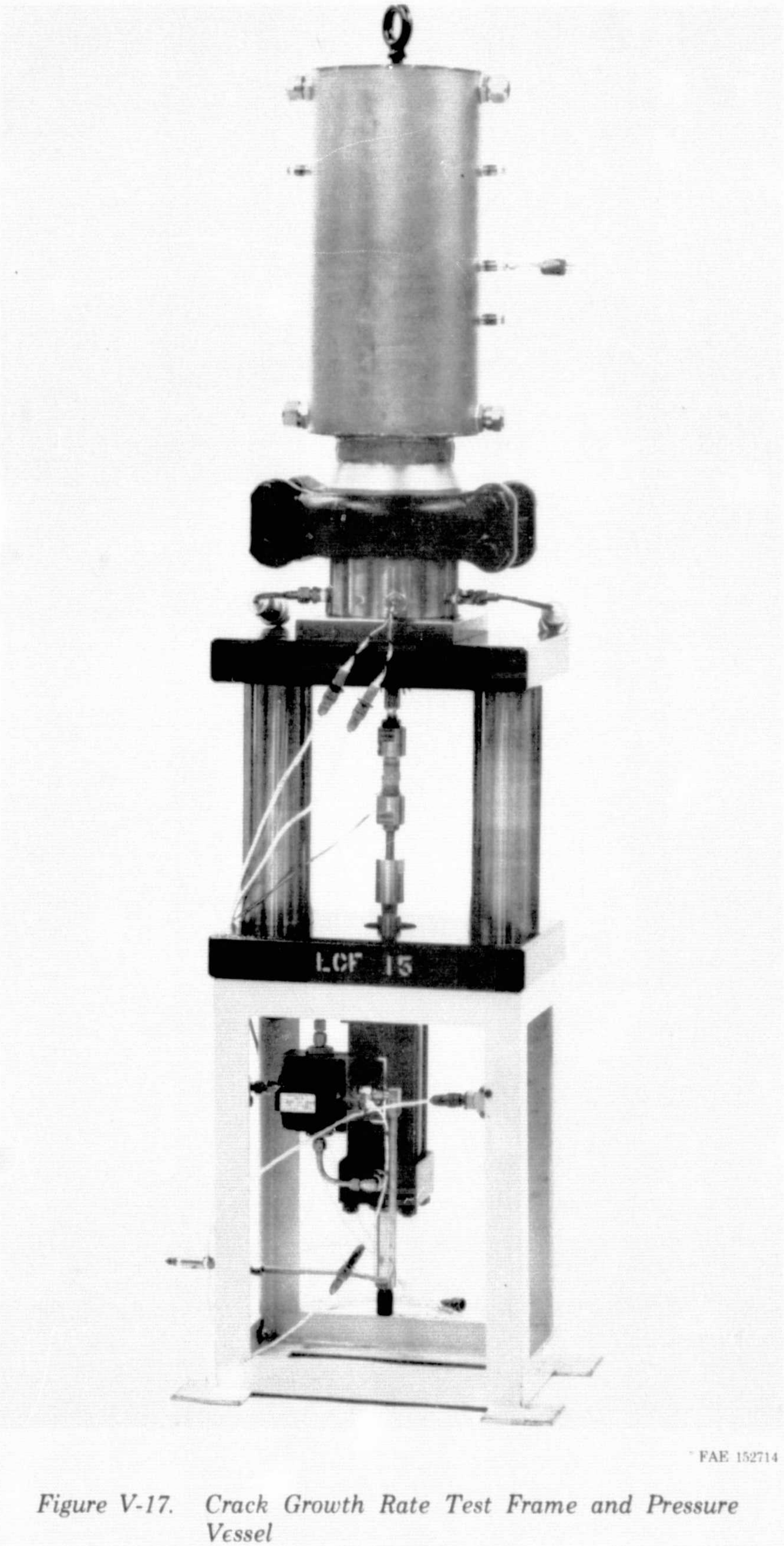


Figure V-15. Crack Length vs Crack Opening Displacement (COD) Compliance Calibration Results for Incoloy 903 and MAR-M-246 (Hf Modified)



FAE 152713

Figure V-16. High-Pressure Environment Crack Growth Rate Testing Machine and Data Acquisition Equipment



FAE 152714

Figure V-17. Crack Growth Rate Test Frame and Pressure Vessel

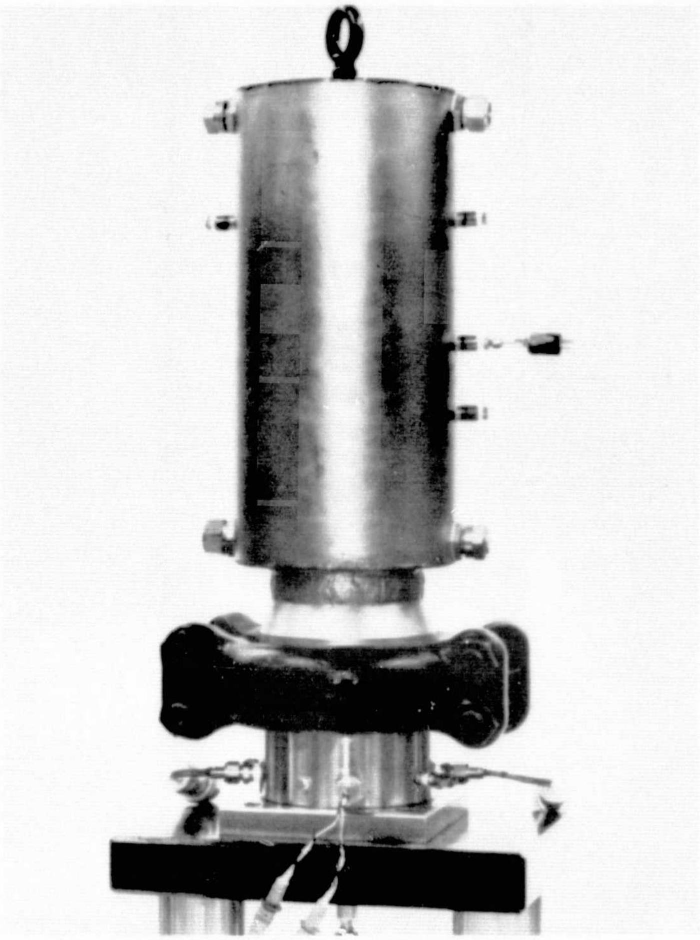

FAE 152715

Figure V-18. Crack Growth Rate Pressure Vessel, Closed

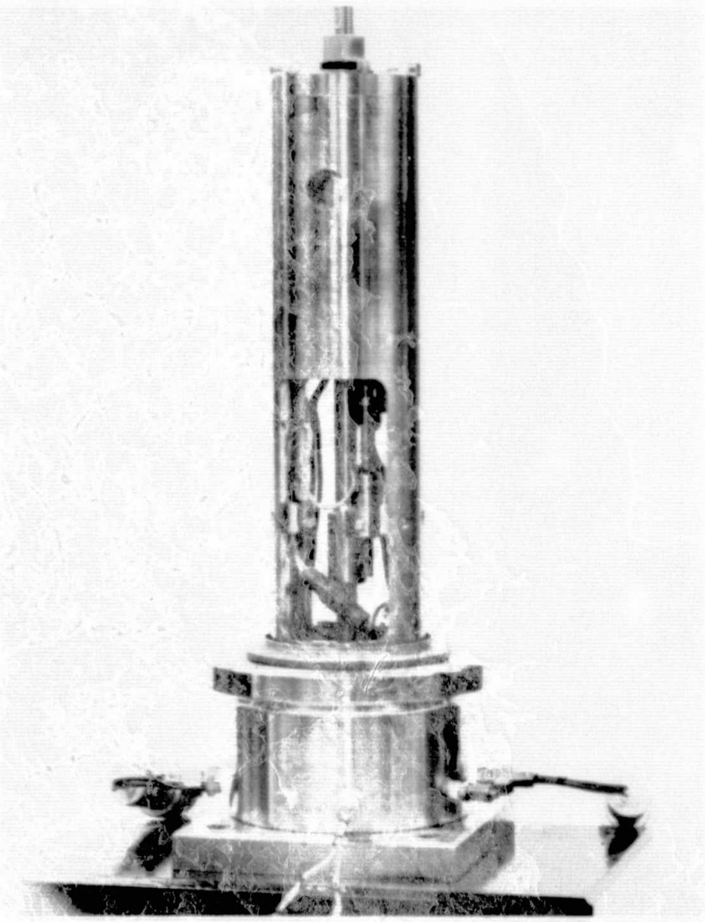
For elevated temperature testing, a two-zone resistance furnace with separate control systems for each zone was used. The furnace surrounds the specimen and fits within the frame of the pressure vessel (figure V-19). Thermocouples attached to the specimen gage section were used to monitor and control temperature during test.

Hydrogen and water vapor environment was obtained utilizing triple-distilled water in a pure hydrogen-containing retort system so the water was vaporized by furnace heat. The retort system, containing the test specimen and water, fits within the furnace and consists of a piston/tube type arangement (figures V-20 and V-21). The piston, attached to the lower pull rod, incorporates an O-ring which provides a seal against the inner surface of a tube (cylinder), which is attached to the upper pull rod. During test the tube remains basically stationary relative to the piston. The base of the piston incorporates an O-ring hole for passage of the extensometer tube, and check valves which allow hydrogen to enter the retort and prevent water from escaping. Pressure inside the retort and vessel was equalized; therefore, the retort contained the hydrogen-water vapor environment and was not subjected to any stresses due to differential (internal to external) pressure. Thermocouples also exit the retort via connectors installed in the base of the piston. They monitor and control specimen and water vapor temperature. By controlling the lower zone of the furnace, water was vaporized at a temperature which assured 50% by weight water vapor.

Specimen COD was measured and recorded throughout the test duration using a Linear Variable Displacement Transducer (LVDT) type extensometer system (figure V-22). COD was monitored and recorded on a stripchart recorder, and on the "X" axis of an "X-Y" recorder. The load sensed by the external load cell was recorded on the "Y" axis of the recorder.

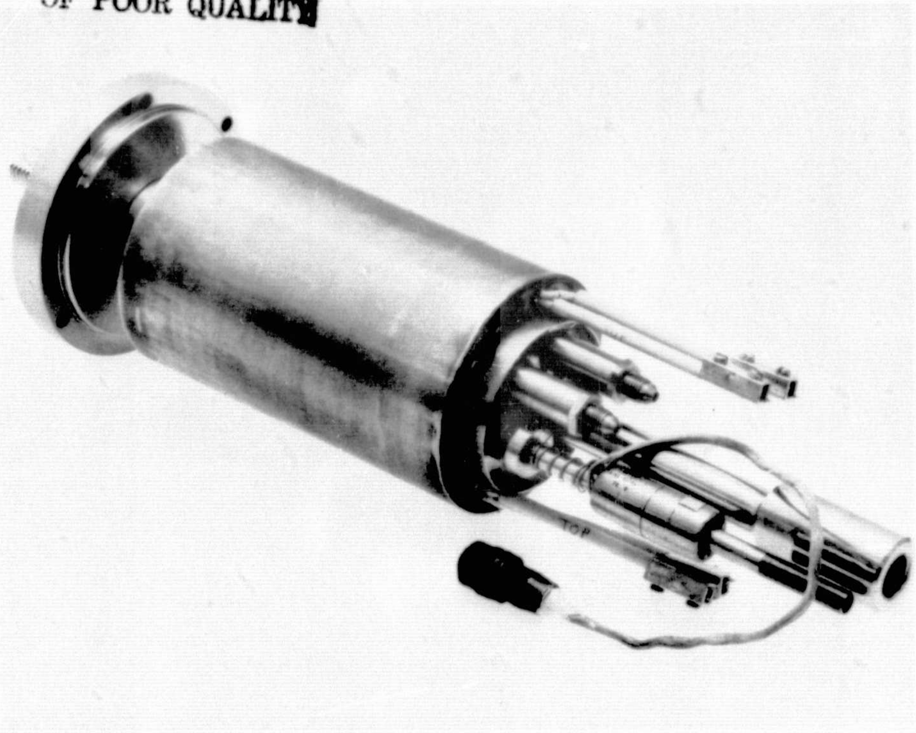
Prior to test, specimens were rinsed with trichlorethylene, wiped dry, rinsed with acetone, wiped dry, and inserted into the test fixture. All handling of specimens was done with clean gloves.

Periodic checks of hydrogen test environments revealed oxygen levels less than 1 ppm. The test and gas handling procedures used for Crack Growth rate testing were similar to those used for the LCF tests performed under this contract (Section IV-C).



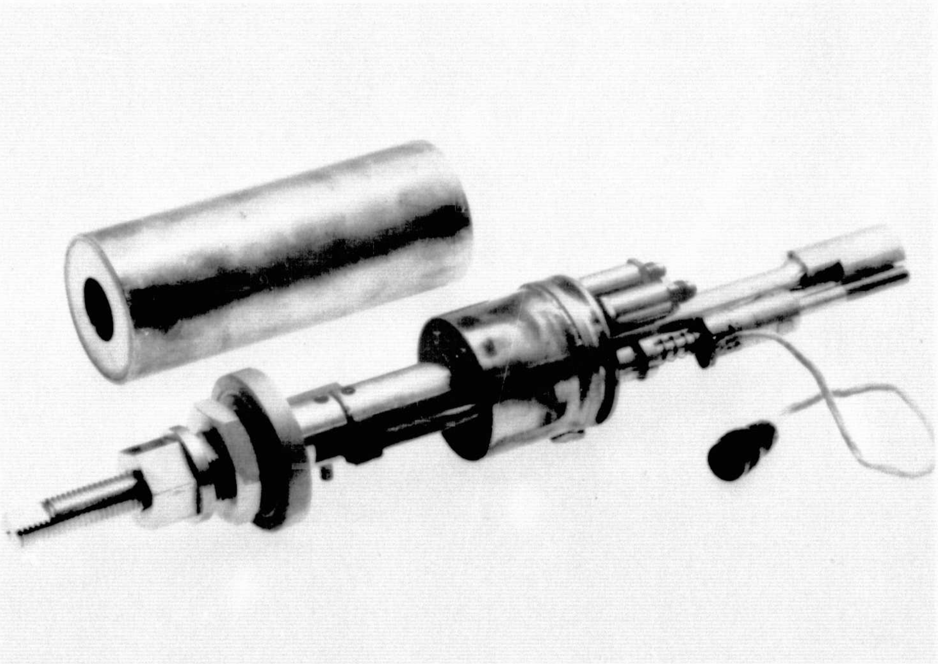
FAE 152716

Figure V-19. Crack Growth Rate Pressure Vessel, Opened



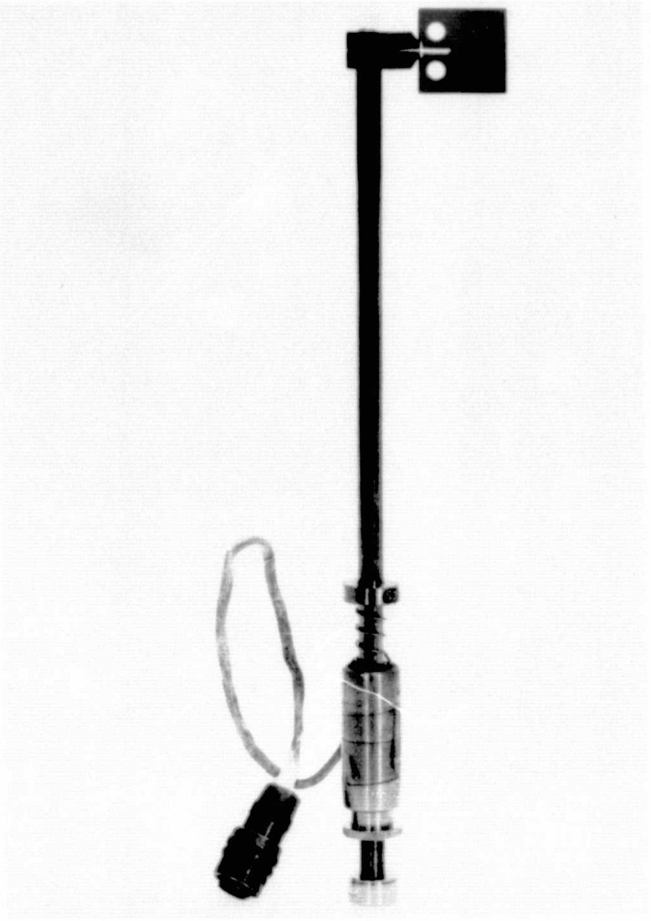
FAE 152717

Figure V-20. Crack Growth Rate Retort System with Furnace in Place



FAE 152719

Figure V-21. Crack Growth Rate Retort System and Extensometry



FAE 152722

Figure V-22. Crack Growth Rate Test Specimen with Extensometer and LVDT Attached