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31 JULY 1973

PROPERTIES OF MATERIALS IN HIGH PRESSURE HYDROGEN AT CRYOGENIC, ROOM, AND ELEVATED TEMPERATURES

FINAL REPORT

Contract NAS8-26191

Prepared for:
George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Marshall Space Flight Center, Alabama 35812

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SECTION I
INTRODUCTION

SECTION I
INTRODUCTION

This report is submitted in accordance with the requirements of Contract NAS8-26191, "Influence of Elevated Temperature on Metals in Gaseous Hydrogen Environments," and represents the final report covering work performed under this contract for the period 29 June 1970 to 31 July 1973. Experimental efforts in this program have consisted of mechanical property tests of seven nickel-, two iron-, two titanium-, and one cobalt-base alloys in gaseous environments at temperatures from 111 to 1144°K (-260 to 1600°F) and pressures from 3.45 to 34.5 MN/m^2 (500 to 5000 psig). The objective of this program was to obtain the mechanical properties of specific materials in a pure or partial hydrogen environment at different temperatures and compare with results of tests made in helium at the same conditions. The specific environments included helium, hydrogen, hydrogen and water vapor, and dissociated hydrazine (obtained by dissociating anhydrous ammonia).

The effort under this contract extended over a 3-year period and included 594 various tests. The overall test program is outlined in table I-1. The number of tests and test conditions for each year's effort are specified in table I-2 through I-4. The primary goal of these tests was to document, rather than define, the hydrogen phenomenon and provide data of use in designing structures exposed to pressurized gaseous hydrogen environments.

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

1. High-Cycle Fatigue - High-cycle fatigue life was established by load (stress) controlled tension-tension testing using smooth specimens and servo-actuated, closed-loop machine.

2. Low-Cycle Fatigue - Low-cycle fatigue life was established by constant total strain testing using smooth specimens and closed-loop testing machine.

3. Fracture Mechanics - Fracture toughness, threshold stress intensity, and cyclic stress intensity were determined using center-notched, fatigue-precracked, plate specimens or single-edged, notched, fatigue-precracked, compact tensile specimens.

4. Creep-Rupture - Creep rate and time to failure were determined using smooth specimens and a standard creep-rupture machine equipped with a recording extensometer.

5. Tensile - Smooth and notched tensile tests were conducted on solid specimens using ASTM tensile testing techniques.

This report is arranged in sections that cover the program conclusions, materials tested, and results and conclusions of the individual property tests. It includes the information covered in the two annual reports, FR-4566[1] and FR-5129[2], previously issued under the contract.

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

This program was conducted using the Program Manager-Project Group System by the Pratt & Whitney Aircraft, Florida Research and Development Center, Materials Development Laboratory, under the cognizance of Mr. W. B. McPherson, Materials Division, Astronautics Laboratory, Marshall Space Flight Center.

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

Mr. J. L. Bearden	- High-Cycle Fatigue Testing
Mr. R. B. Bogard	- Low-Cycle Fatigue Testing
Mr. J. Doyle	- Tensile Testing
Mrs. A. F. Kirkpatrick	- Proposal and Report Efforts
Mr. T. M. Pruitt	- Test Support, Rocket Test Facility
Mr. J. F. Schratt	- Creep-Rupture Testing and Report Efforts
Mrs. C. B. Stevens	- Metallurgical Investigation
Mr. D. J. Stoddard	- Cost and Planning Efforts
Mr. B. H. Walker	- Fracture Mechanics Testing
Miss M. Zaccagnino	- Proposal and Report Efforts

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(1) "Properties of Materials in High Pressure Hydrogen at Cryogenic, Room, and Elevated Temperatures," Annual Report, Contract NAS8-26191, FR-4566, dated 30 June 1971.

(2) "Properties of Materials in High Pressure Hydrogen at Room and Elevated Temperatures," Annual Report, Contract NAS8-26191, FR-5129, dated 30 June 1972.

Table I-1. Experimental Program Listing Type of Tests and Test Conditions Used to Determine the Susceptibility of Various Alloys to Environmental Degradation

Material	Temperature, K	Temperature, °F	Pressure, MN/m²	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (K_{IE} or K_{IC})	Threshold Stress Intensity (K_{TH})	Cyclic Stress Intensity (K_I/K_C)	Smooth Tensile (ST)	Notched Tensile (NT)
Inconel 718	111	-260	34.5	5000	Helium	X							
	111	-260	34.5	5000	Hydrogen	X							
	300	80	34.5	5000	Helium	X	X		X			X	X
	300	80	34.5	5000	Hydrogen	X	X		X			X	X
	951	1250	34.5	5000	Helium	X	X	X				X	X
	951	1250	34.5	5000	Hydrogen	X	X	X				X	X
Inconel 718 Welds	300	80	34.5	5000	Helium				X			X	X
	300	80	34.5	5000	Hydrogen				X			X	X
Inconel 625	300	80	34.5	5000	Helium	X			X			X	X
	300	80	34.5	5000	Hydrogen	X			X			X	X
	951	1250	34.5	5000	Helium			X				X	X
	951	1250	34.5	5000	Hydrogen			X				X	X
Inconel 625 Welds	300	80	34.5	5000	Helium				X			X	X
	300	80	34.5	5000	Hydrogen				X			X	X
WASPALOY®	300	80	34.5	5000	Helium				X				
	300	80	34.5	5000	Hydrogen				X	X	X		
	951	1250	34.5	5000	Helium	X		X				X	
	951	1250	34.5	5000	Hydrogen	X		X				X	
	951	1250	34.5	5000	Hydrogen and Water Vapor			X					
Astroloy	300	80	3.45	500	Helium								X
	300	80	3.45	500	Hydrogen								X
	951	1250	3.45	500	Helium								X
	951	1250	3.45	500	Hydrogen								X
	951	1250	3.45	500	Hydrogen and Water Vapor								X
	951	1250	3.45	500	Hydrazine(1)			X					
	1144	1600	3.45	500	Helium							X	X
	1144	1600	3.45	500	Hydrogen							X	X
	1144	1600	3.45	500	Hydrogen and Water Vapor								X
	1144	1600	3.45	500	Hydrazine			X					
	951	1250	34.5	5000	Helium			X					X
	951	1250	34.5	5000	Hydrogen			X					X
	1144	1600	34.5	5000	Helium								X
	1144	1600	30.3	4400	Hydrogen								X
Hastelloy X	300	80	34.5	5000	Helium	X						X	X
	300	80	34.5	5000	Hydrogen	X						X	X
	951	1250	34.5	5000	Helium							X	X
	951	1250	34.5	5000	Hydrogen							X	X
IN-100	300	80	34.5	5000	Helium							X	
	300	80	34.5	5000	Hydrogen							X	
	951	1250	34.5	5000	Helium	X	X	X				X	
	951	1250	34.5	5000	Hydrogen	X	X	X				X	
	951	1250	34.5	5000	Hydrogen and Water Vapor			X					
MAR M-200 DS	1144	1600	3.45	500	Helium			X					X
	1144	1600	3.45	500	Hydrogen		X	X					X

Table I-1. Experimental Program Listing Type of Tests and Test Conditions Used to Determine the Susceptibility of Various Alloys to Environmental Degradation (Continued)

Material	Temperature, °K	Temperature, °F	Pressure, MN/m^2	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (K_{IE} or K_{IC})	Threshold Stress Intensity (K_{TH})	Cyclic Stress Intensity (K_I/K_C)	Smooth Tensile (ST)	Notched Tensile (NT)
	1144	1600	3.45	500	Hydrogen and Water Vapor								X
	1144	1600	3.45	500	Hydrazine		X	X					
	300	80	34.5	5000	Helium							X	
	300	80	34.5	5000	Hydrogen							X	
	951	1250	34.5	5000	Helium	X	X	X				X	X
	951	1250	34.5	5000	Hydrogen	X	X	X				X	X
	951	1250	34.5	5000	Hydrogen and Water Vapor			X					X
A-286	951	1250	3.45	500	Helium			X					
	951	1250	3.45	500	Hydrogen			X					
	951	1250	3.45	500	Hydrazine			X					
	300	80	34.5	5000	Helium	X			X			X	X
	300	80	34.5	5000	Hydrogen	X			X			X	X
	951	1250	34.5	5000	Helium	X		X				X	X
	951	1250	34.5	5000	Hydrogen	X		X				X	X
AISI 347	111	-260	34.5	5000	Helium							X	X
	111	-260	34.5	5000	Hydrogen							X	X
	300	80	34.5	5000	Helium	X	X		X			X	X
	300	80	34.5	5000	Hydrogen	X	X		X			X	X
	951	1250	34.5	5000	Helium			X				X	X
	951	1250	34.5	5000	Hydrogen			X				X	X
Ti-6Al-4V	300	80	34.5	5000	Helium	X			X			X	X
	300	80	34.5	5000	Hydrogen	X			X			X	X
	366	200	34.5	5000	Helium	X		X				X	X
	366	200	34.5	5000	Hydrogen	X		X				X	X
Ti-5Al-2.5Sn (A-110)	300	80	34.5	5000	Helium	X	X		X			X	X
	300	80	34.5	5000	Hydrogen	X	X		X			X	X
	366	200	34.5	5000	Helium	X		X				X	X
	366	200	34.5	5000	Hydrogen	X		X				X	X
Haynes 188	300	80	3.45	500	Helium								X
	300	80	3.45	500	Hydrogen								X
	951	1250	3.45	500	Helium			X					X
	951	1250	3.45	500	Hydrogen			X					X
	951	1250	3.45	500	Hydrogen and Water Vapor								X
	951	1250	3.45	500	Hydrazine		X	X					
	1144	1600	3.45	500	Helium			X					X
	1144	1600	3.45	500	Hydrogen			X					X
	1144	1600	3.45	500	Hydrogen and Water Vapor								X
	1144	1600	3.45	500	Hydrazine			X					
	951	1250	34.5	5000	Helium	X		X					
	951	1250	34.5	5000	Hydrogen	X		X					
	951	1250	34.5	5000	Hydrogen and Water Vapor			X					

(1) Dissociated Hydrazine Environment Obtained by Dissociating Anhydrous Ammonia.

Specific Heat Treatments and/or Material Forms are Listed in Tables I-2 Through I-4, or in Section III, "Materials and Specimens."

Table I-2. Experimental Program for the Period of June 1970 to June 1971, Under Contract NAS8-26191, Listing Type, Condition, and Number of Tests Conducted

Material	Temperature, °K	Temperature, °F	Pressure, MN/m^2	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (K_{IE} or K_{IC})	Smooth Tensile (ST)	Notch[1] Tensile (NT)
						Type and Number of Tests					
Inconel 718[2]	300	80	34.5	5000	Helium	4	4		2	2	2
	300	80	34.5	5000	Hydrogen	4	4		2	2	3
	951	1250	34.5	5000	Helium	4	4	2		2	2
	951	1250	34.5	5000	Hydrogen	4	4	2		2	3
Inconel 718[3]	111	-260	34.5	5000	Helium	4					
	111	-260	34.5	5000	Hydrogen	4					
	300	80	34.5	5000	Helium	4	4		2	2	2
	300	80	34.5	5000	Hydrogen	4	4		2	2	3
	951	1250	34.5	5000	Helium	4	4	2		2	2
	951	1250	34.5	5000	Hydrogen	4	4	2		2	3
Inconel 718 Welds[3]	300	80	34.5	5000	Helium				2	2	2
	300	80	34.5	5000	Hydrogen				2	2	3
Inconel 625	300	80	34.5	5000	Helium	4			2	2	2
	300	80	34.5	5000	Hydrogen	4			2	2	3
	951	1250	34.5	5000	Helium			2		2	2
	951	1250	34.5	5000	Hydrogen			2		2	3
Inconel 625 Welds	300	80	34.5	5000	Helium				2	2	2
	300	80	34.5	5000	Hydrogen				2	2	3
Hastelloy X	300	80	34.5	5000	Helium	4				2	2
	300	80	34.5	5000	Hydrogen	4				2	3
	951	1250	34.5	5000	Helium					2	2
	951	1250	34.5	5000	Hydrogen					2	3
A-286	300	80	34.5	5000	Helium	4			2	2	2
	300	80	34.5	5000	Hydrogen	4			2	2	3
	951	1250	34.5	5000	Helium	4		2		2	2
	951	1250	34.5	5000	Hydrogen	4		2		2	3

Table I-2. Experimental Program for the Period of June 1970 to June 1971, Under Contract NAS8-26191, Listing Type, Condition, and Number of Tests Conducted (Continued)

AISI-347	111	-260	34.5	5000	Helium					2	2
	111	-260	34.5	5000	Hydrogen					2	3
	300	80	34.5	5000	Helium	4	4		2	2	2
	300	80	34.5	5000	Hydrogen	4	4		2	2	3
	951	1250	34.5	5000	Helium			2		2	2
	951	1250	34.5	5000	Hydrogen			2		2	3
Ti-6Al-4V	300	80	34.5	5000	Helium	4			2	2	2
	300	80	34.5	5000	Hydrogen	4			2	2	3
	366	200	34.5	5000	Helium	4		2		2	2
	366	200	34.5	5000	Hydrogen	4		2		2	3
Ti-5Al-2.5Sn (A-110)	300	80	34.5	5000	Helium	4	4		2	2	2
	300	80	34.5	5000	Hydrogen	4	4		2	2	3
	366	200	34.5	5000	Helium	4		2		2	2
	366	200	34.5	5000	Hydrogen	4		2		2	3

(1) Notched Tensile Strength for $K_T = 8$

(2) Annealed at 1227°K (1750°F) and Aged (See Section III)

(3) Solution Treated at 1313°K (1900°F) and Aged (See Section III)

Table I-3. Experimental Program for the Period of June 1971 to June 1972, Under Contract NAS8-26191, Listing Type, Condition, and Number of Tests Conducted

Material	Temperature, °K	Temperature, °F	Pressure, MN/m²	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (K_{IE} or K_{IC})	Threshold Stress Intensity (K_{TH})	Cyclic Stress Intensity (K_I/K_C)	Smooth Tensile (ST)	Notch[(1)] Tensile (NT)
Astroloy	300	80	3.45	500	Helium								1
	300	80	3.45	500	Hydrogen								2
	951	1250	3.45	500	Helium								1
	951	1250	3.45	500	Hydrogen							1	2
	1144	1600	3.45	500	Helium							1	1
	1144	1600	3.45	500	Hydrogen								2
	951	1250	34.5	5000	Helium			2					1
	951	1250	34.5	5000	Hydrogen			2					2
	1144	1600	34.5(2)	5000	Helium								1
	1144	1600	34.5(2)	5000	Hydrogen								2
WASPALOY®	300	80	34.5	5000	Helium				1	3	5		
	300	80	34.5	5000	Hydrogen				3			1	
	951	1250	34.5	5000	Helium	2		2				2	
	951	1250	34.5	5000	Hydrogen	4		2					
	951	1250	34.5	5000	Hydrogen and Water Vapor			2					
IN-100	300	80	34.5	5000	Helium							1	
	300	80	34.5	5000	Hydrogen							2	
	951	1250	34.5	5000	Helium	2	2	2				1	
	951	1250	34.5	5000	Hydrogen	4	4	2				2	
	951	1250	34.5	5000	Hydrogen and Water Vapor			2					
MAR M-200 DS	300	80	34.5	5000	Helium							1	
	300	80	34.5	5000	Hydrogen							2	
	951	1250	34.5	5000	Helium	2	2	2				1	
	951	1250	34.5	5000	Hydrogen	4	4	2				2	
	951	1250	34.5	5000	Hydrogen and Water Vapor			2					
Haynes 188	951	1250	34.5	5000	Helium		2	2					
	951	1250	34.5	5000	Hydrogen		4	2					
	951	1250	34.5	5000	Hydrogen and Water Vapor			2					

(1) Notched Tensile Strength for K_T = 8

(2) Or Maximum Attainable Pressure at 1144°K (1600°F).

Table I-4. Experimental Program for the Period of June 1972 to July 1973, Under Contract NAS8-26191, Listing Type, Condition, and Number of Tests Conducted

Material	Temperature, °K	Temperature, °F	Pressure, MN/m^2	Pressure, psig	Environment	Type and Number of Tests: High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Notch[1] Tensile (NT)
Astroloy	951	1250	3.45	500	Hydrogen and Water Vapor			3
	951	1250	3.45	500	Hydrazine[2]		3	
	1144	1600	3.45	500	Hydrogen and Water Vapor			3
	1144	1600	3.45	500	Hydrazine		3	
MAR M-200 DS	951	1250	34.5	5000	Helium			1
	951	1250	34.5	5000	Hydrogen			3
	951	1250	34.5	5000	Hydrogen and Water Vapor			3
	1144	1600	3.45	500	Helium		2	1
	1144	1600	3.45	500	Hydrogen	4	3	3
	1144	1600	3.45	500	Hydrogen and Water Vapor			3
	1144	1600	3.45	500	Hydrazine	4	3	
A-286	951	1250	34.5	5000	Hydrogen		3	
	951	1250	3.45	500	Helium		2	
	951	1250	3.45	500	Hydrogen		3	
	951	1250	3.45	500	Hydrazine		3	
Haynes 188	300	80	3.45	500	Helium			1
	300	80	3.45	500	Hydrogen			3
	951	1250	3.45	500	Helium		2	1
	951	1250	3.45	500	Hydrogen		3	3
	951	1250	3.45	500	Hydrogen and Water Vapor			3
	951	1250	3.45	500	Hydrazine	4	3	
	1144	1600	3.45	500	Helium		2	1
	1144	1600	3.45	500	Hydrogen		3	3
	1144	1600	3.45	500	Hydrogen and Water Vapor			3
	1144	1600	3.45	500	Hydrazine		3	

(1) Notched Tensile Strength for K_T 8

(2) Dissociated Hydrazine Environment Obtained by Dissociating Anhydrous Ammonia.

SECTION II
CONCLUSIONS

SECTION II
CONCLUSIONS

A. GENERAL

The efforts under this contract consisted of conducting various tests to determine mechanical properties of 12 alloys that are commonly used or proposed for use in pressurized gaseous hydrogen or hydrogen containing environments (hydrogen and water vapor and dissociated hydrazine). Properties determined in the hydrogen environments were compared to properties determined in a pure helium environment at the same conditions to establish environmental degradation. In some cases, properties in the hydrogen environments only were determined to establish design information.

The following system was established to determine the degree of degradation and serve as an aid in comparing the various alloys.

1. Extremely Degraded (ED) - Hydrogen environment(s) reduced the property or life (in helium) greater than 50%.

2. Severely Degraded (SD) - Hydrogen environment(s) reduced the property or life (in helium) greater than 25%, but less than 50%.

3. Degraded (D) - Hydrogen environment(s) reduced the property or life (in helium) greater than 10%, but less than 25%.

4. Negligible Degradation (ND) - Hydrogen environment(s) reduced the property or life (in helium) less than 10% or had no detrimental effect.

Using this rating system, table II-1 displays the degree of degradation for each alloy and condition tested, where a comparable test in helium was conducted. In the case of the tensile tests, if any property (yield strength, smooth or notch ultimate strength, elongation, and reduction of area) was degraded, the degradation rating was that of the most severely degraded property.

Detailed conclusions are presented in the various sections pertaining to type of test. General conclusions, as pertaining to the various alloys, are presented below.

B. NICKEL-BASE ALLOYS

Tested were:

Inconel 718 (Two Heat Treatments)
Inconel 718 Welded
Inconel 625
Inconel 625 Welded
WASPALOY®
Astroloy
Hastelloy X
IN-100 (Cast)
MAR M-200 DS (Cast).

Table II-1. Degree of Environmental Degradation of Various Alloys

Material	Temperature, °K	Temperature, °F	Pressure, MN/m^2	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (KIE or KIC)	Tensile
Inconel 718	300	80	34.5	5000	Hydrogen	ED	ED		ND	ED
1227°K (1750°F)	951	1250	34.5	5000	Hydrogen	SD	ED	SD		ND
Inconel 718	111	-260	34.5	5000	Hydrogen	ND				
1313°K (1900°F)	300	80	34.5	5000	Hydrogen	ED	ED		ND	D
	951	1250	34.5	5000	Hydrogen	SD	ED	SD		ND
Inconel 718 Welds	300	80	34.5	5000	Hydrogen				ND	SD
Inconel 625	300	80	34.5	5000	Hydrogen	ED			ND	ED
	951	1250	34.5	5000	Hydrogen			SD		D
Inconel 625 Welds	300	80	34.5	5000	Hydrogen				ND	ND
WASPALOY®	300	80	34.5	5000	Hydrogen				SD	
	951	1250	34.5	5000	Hydrogen	D		D		D
	951	1250	34.5	5000	Hydrogen and Water Vapor			ND		
Astroloy	300	80	3.45	500	Hydrogen					ND
	951	1250	3.45	500	Hydrogen					ND
	951	1250	3.45	500	Hydrogen and Water Vapor					ND
	951	1250	3.45	500	Hydrazine			ND		
	1144	1600	3.45	500	Hydrogen					ND
	1144	1600	3.45	500	Hydrogen and Water Vapor					D
	1144	1600	3.45	500	Hydrazine			ND		
	951	1250	34.5	5000	Hydrogen			ND		ND
	1144	1600	30.3	4400	Hydrogen					ND
Hastelloy X	300	80	34.5	5000	Hydrogen	ED				D
	951	1250	34.5	5000	Hydrogen					ND
IN-100	300	80	34.5	5000	Hydrogen					ED
	951	1250	34.5	5000	Hydrogen	ED	SD	D		SD
	951	1250	34.5	5000	Hydrogen and Water Vapor			ND		

Table II-1. Degree of Environmental Degradation of Various Alloys (Continued)

Material	Temperature, °K	Temperature, °F	Pressure, MN/m²	Pressure, psig	Environment	Low-Cycle Fatigue (LCF)	High-Cycle Fatigue (HCF)	Creep-Rupture (C-R)	Fracture Toughness (KIE or KIC)	Tensile
MAR M-200 DS	1144	1600	3.45	500	Hydrogen			D		ND
	1144	1600	3.45	500	Hydrogen and Water Vapor					D
	1144	1600	3.45	500	Hydrazine			ND		
	300	80	34.5	5000	Hydrogen					ED
	951	1250	34.5	5000	Hydrogen	ND	ED	ND		SD
	951	1250	34.5	5000	Hydrogen and Water Vapor			ND		D
A-286	951	1250	3.45	500	Hydrogen			ND		
	951	1250	3.45	500	Hydrazine			ND		
	300	80	34.5	5000	Hydrogen	ND			ND	ND
	951	1250	34.5	5000	Hydrogen	ND		D		D
AISI 347	111	-260	34.5	5000	Hydrogen					ND
	300	80	34.5	5000	Hydrogen	ND	ND		ND	ND
	951	1250	34.5	5000	Hydrogen			ND		ND
Titanium-6-4	300	80	34.5	5000	Hydrogen	ND			ND	D
	366	200	34.5	5000	Hydrogen	D		D		SD
A-110	300	80	34.5	5000	Hydrogen	ND	ND		ND	D
	366	200	34.5	5000	Hydrogen	ED		ND		SD
Haynes 188	300	80	3.45	500	Hydrogen					ND
	951	1250	3.45	500	Hydrogen			ND		ND
	951	1250	3.45	500	Hydrogen and Water Vapor					ND
	951	1250	3.45	500	Hydrazine			ND		
	1144	1600	3.45	500	Hydrogen			ND		ND
	1144	1600	3.45	500	Hydrogen and Water Vapor					ND
	1144	1600	3.45	500	Hydrazine			ND		
	951	1250	34.5	5000	Hydrogen	SD		ND		
	951	1250	34.5	5000	Hydrogen and Water Vapor			ND		

The nickel-base alloys as a class were the most suseptible to hydrogen environment degradation. With two exceptions, the low-cycle and high-cycle fatigue life, both strain-controlled and tension-tension load-controlled, was degraded for all nickel-base alloys tested in 34.5-MN/m^2 (5000-psig) hydrogen. The exceptions were the low-cycle fatigue lives of Inconel 718 with 1313°K (1900°F) solution plus age heat treatment at 111°K (-260°F) and MAR M-200 DS at 951°K (1250°F). At these two conditions, those alloys displayed better lives in the hydrogen than in the helium environments. The degree of degradation was influenced by temperature, being most severe at 300°K (80°F).

The fracture toughness, K_{IE} or K_{IC} was degraded for only one material, WASPALOY. This was the only alloy tested using a center notch specimen. The other alloys were tested using the ASTM compact tensile specimen, and all exhibited negligible degradation (ND).

The rupture life, on a stress for specific life basis, was degraded only by the pure hydrogen environment, with Inconel 718 (both heat treatments) and Inconel 625 being severely degraded, and WASPALOY, IN-100 and MAR M-200 DS slightly degraded (less than 25%). Materials tested in the hydrogen and water vapor and dissociated hydrazine environments were not degraded by those environments. Astroloy was the only material that exhibited negligible degradation in rupture life at any of the conditions tested; however, this was based upon stress level degradation. When compared on time to rupture for a given stress level in helium and hydrogen, Astroloy was degraded (Reference FR-5129).

Tensile property degradation was more evident at room temperature with ductility being most affected. None of the alloys exhibited degradation of smooth tensile yield strength, and only IN-100 and MAR M-200 DS, the cast alloys, smooth tensile ultimates were degraded. Elongation and reduction of area were quite degraded, with IN-100 the most degraded, followed by MAR M-200 DS, Inconel 718 (1227°K solution), Inconel 625, Inconel 718 welded (1313°K solution), and Inconel 718 (1313°K solution) in that order. Smooth tensile properties were not significantly affected for Astroloy, Hastelloy X, or Inconel 625 welds, at any conditions tested, and for Inconel 718 of both heat treatments at 951°K (1250°F). Notch ultimate strengths were degraded in some cases where smooth strength was not. This included Inconel 718 (1227°K solution), Inconel 718 welds (1313°K solution), and Hastelloy X, all at room temperature. Pure hydrogen environment caused no significant degradation of notch strength at elevated temperatures. Astroloy and MAR M-200 DS were tested in the hydrogen and water vapor environment. Notch strength degradation at 951°K (1250°F) occurred for Mar M-200 DS only; however, at 1144°K (1600°F) both alloys were degraded. It was suspected that the oxidation caused by dissociation of the water at the specimen surface at 1144°K (1600°F) and not the hydrogen caused the reduction in strength from the helium environment, as a pure hydrogen environment did not cause degradation at these temperatures.

C. IRON-BASE ALLOYS

Tested were:

A-286
AISI 347.

These alloys as a class exhibited the least severe degradation of all materials tested. In fact, AISI 347 had negligible property degradation for all tests and conditions investigated during this program. A-286 exhibited negligible property degradation at 300°K (80°F). The elevated temperature (951°K [1250°F]) creep-rupture and smooth tensile properties only were degraded at 34.5MN/m^2 (5000-psig) pressure. At 3.45-MN/m^2 (500-psig) pressure, rupture life was not significantly degraded in either the pure hydrogen or dissociated hydrazine (ammonia) environments.

D. TITANIUM-BASE ALLOYS

Tested were:

Titanium 6-4
Titanium A-110.

These two alloys did not exhibit significant degradation in any property at room temperature except tensile properties. The smooth tensile ductilities and notch tensile strength were degraded to severely degraded. At a temperature of 366°K (200°F) the low-cycle fatigue life and tensile properties of both alloys were degraded, with A-110 more severely affected. However, Titanium 6-4 was degraded in creep-rupture at this temperature, whereas A-110 was not. There was also evidence of the formation of surface hydrides on specimens tested at 366°K (200°F).

E. COBALT-BASE ALLOY

Tested was Haynes 188.

This alloy was tested in three hydrogen containing environments. At 3.45-MN/m^2 (500-psig) pressure, rupture life, based upon stress, was not degraded by hydrogen or dissociated hydrazine (ammonia) at either 951 or 1144°K (1250 or 1600°F), nor was notch tensile strength in the hydrogen or hydrogen and water vapor environments. At 34.5-MN/m^2 (5000-psig) pressure, creep-rupture, in hydrogen and hydrogen and water vapor environments, was not significantly degraded. The only property evaluated that was degraded was low-cycle fatigue life at 951°K and 34.5-MN/m^2 (1250°F and 5000-psig) pressure, which was severely degraded.

F. ENVIRONMENTS AND PRESSURE

Four gaseous environments were used during the various phases of this program. They were: pure helium, pure hydrogen, hydrogen with water vapor, and dissociated hydrazine. The dissociated hydrazine environment was obtained by dissociating pure anhydrous ammonia. (Reference Section III.) A post test gas sample of the hydrazine/ammonia verified the almost total dissociation of the ammonia into hydrogen and nitrogen in the proportions indicated by the analytical equations. The experimental test matrix was not complete enough to enable absolute statements as to the effect of the different environments and pressures upon materials properties; however, some general observations can be made. The presence of water vapor in the hydrogen appeared to inhibit or reduce degradation as compared to the pure hydrogen environment. This was true for WASPALOY and IN-100 materials, which were degraded in creep-rupture life in hydrogen, but not in hydrogen and water vapor. However, as temperatures increased to 1144°K (1600°F), Astroloy and MAR M-200 DS were both more degraded in the properties tested by the addition of water vapor to the hydrogen. The beneficial effects of water vapor in the hydrogen appear limited to those temperatures at which the water vapor would not dissociate into hydrogen and oxygen, with resulting oxidation of the material. It must be emphasized, however, that 1144°K (1600°F) is normally beyond the operating range of Astroloy, and that MAR M-200 DS is usually given a protective coating to prevent oxidation. Also, this testing was conducted under static pressure conditions and the dynamic conditions existing in a propulsion system could produce different effects.

For materials and properties, where comparisons could be made, the dissociated hydrazine (ammonia) environment had negligible effect in that none of the properties tested were degraded.

Specimens that were tested in the dissociated hydrazine (ammonia) environment were examined for nitride formation on the specimen surfaces. Metallography (light and electron), X-ray diffraction, and electron microprobe analyses did not detect surface nitrides on any of the specimens.

Two pressure levels were used in this program. Comparison of the effect of pressure upon degradation was restricted due to lack of comparable test conditions, or to lack of single material heat results. The most pronounced effect of pressure in this program occurred in the notch tensile properties of Astroloy. While tensile strength at both pressures was rated negligible (10% or less), at 3.45-MN/m^2 (500-psig) hydrogen degradation was approximately half that at 34.5 MN/m^2 (5000 psig) for temperatures below 977°K (1300°F). This is in general agreement with other investigators' observations that the effect of hydrogen upon property degradation decreases with decreasing pressure.

G. DISCUSSION

This program was established to determine specific material properties and to enable general observations in regard to the susceptibility of a particular material to hydrogen degradation. The experience of this program has been that creep-rupture and low-cycle fatigue, both of which involve relatively long exposures to the environment at high strain/stress levels, are the most severe tests of a material for hydrogen degradation, followed by high-cycle fatigue, tensile, and fracture toughness tests, in that order.

Certain tests, however, did not indicate any conclusive degradation due to the hydrogen environment on materials known to be degraded. An example of this is the fracture toughness of Inconel 718 with 1227°K (1750°F) solution plus age heat treatment. This test indicated negligible degradation of this material by the hydrogen environment. The low- and high-cycle fatigue, creep-rupture, and tensile tests, however, indicated extreme hydrogen degradation.

A-286 material exhibited negligible degradation at 300°K (80°F) in all tests. At elevated temperature, the creep-rupture test indicated degradation. This emphasizes the fact that no one test will provide enough data to evaluate the degree of hydrogen degradation to be used in analyzing a structure from a design-material-life standpoint. For the individual responsible for a structure that must operate in hydrogen environments, there can be no substitute for a test that will supply data appropriate to the particular loading spectrum expected. In this case, a test that simply indicates susceptibility to hydrogen degradation is of no real value; the test must also provide some measure of the degree of degradation on a meaningful property. It is felt that this program has contributed to the store of information necessary for the design of structures, as well as providing a measure of the hydrogen degradation of common engineering materials. While answering many questions concerning properties in specific environments, many more questions have been raised. It is hoped that continued work in this area will enable explanation, as well as documentation, of the hydrogen effect on metals.

SECTION III
MATERIALS AND SPECIMENS

SECTION III
MATERIALS AND SPECIMENS

A. TEST MATERIAL

The purpose of this contract was to determine the susceptibility of various alloys to environmental degradation. The testing evaluated the mechanical properties of wrought and case nickel-base, wrought iron-base, wrought titanium-base, and wrought cobalt-base alloys in phases over a 3-year period. Table III-1 lists the materials and conditions in which they were received and tested. Also included are the types of tests performed on each material and the phase, or year, of the contract that they were performed. The chemical composition of the materials is listed in table III-2. Wrought material was purchased from commercial sources to industry standard (AMS) specifications where possible. In the absence of industry specifications, the applicable Pratt & Whitney Aircraft specification was used. Cast test bars were produced by the Experimental Foundary of the P&WA Connecticut Operations.

Material was ordered separately for each phase of the testing program in sufficient quantity of single heats to perform the types of tests required. For this reason, materials that were tested in more than one phase of the program were from different heats. All materials were routed through the PWA-FRDC Materials Control Laboratory to ensure compliance with purchasing specifications; all materials were in conformance. The majority of materials were purchased in the form of wrought barstock, except for Astroloy, MAR M-200 DS (directionally solidified) and IN-100. The Astroloy material was purchased in the form of flat pancake forgings. The MAR M-200 DS and IN-100 materials were in the form of short cast test bars, which were judged radiographically sound prior to acceptance.

Some microshrinkage was evident in the cast MAR M-200 DS and IN-100 material. As no industry standard specifications for these materials have been established, acceptable microshrinkage levels were based upon experience with gas turbine engine thin-wall castings. Metallurgical examination of some specimens that produced questionable test results did reveal excessive microshrinkage and/or porosity at or near the fracture surfaces. In these cases, retests were conducted.

Specimen blanks were cut from the barstock and heat treated as required. The Inconel 718 material was received in the annealed condition and was subsequently heat treated according to two contract requirements. The WASPALOY®, MAR M-200 DS, and IN-100 materials were also received in the annealed condition and subsequently heat treated. The Astroloy pancake forgings and A-286 barstock materials were received fully heat treated. Astroloy specimen blanks were oriented with the axis in the circumferential direction and were taken from the periphery of the forging. All other materials were tested in the solution-annealed (as-received) condition.

Table III-1. Materials Used to Determine the Susceptibility of Various Alloys to Environmental Degradation

Material	Base	Form	Purchase Specification	Heat Code	As-Received Condition	As-Tested Condition (Heat Treatment)	Year Tested	Types of Tests [(1)]
Inconel 718	Nickel	Wrought	AMS 5662	BVTO	19.05 mm (0.75 in.) Diameter Barstock	1227°K (1750°F) 1 Hr, Air Cool, 993°K (1325°F) 8 hr, Furnace Cool to 899°K (1150°F), Hold at 899°K (1150°F) for Total Age Time of 18 hr, Air Cool	1	LCF, HCF, CR, NT, ST
Inconel 718	Nickel	Wrought	AMS 5662	BVTO	19.05 mm (0.75 in.) Diameter Barstock	1227°K (1750°F) 1 hr, Air Cool, 1313°K (1900°F) 1 hr, Air Cool 28°K (50°F) Per min. 993°K (1325°F) 8 hr, Furnace Cool to 899°K (1150°F), Hold at 899°K (1150°F) for Total Age Time of 18 hr, Air Cool	1	LCF, HCF, CR, NT, ST
Inconel 718	Nickel	Wrought	AMS 5662	BZMK	101.62 mm (4.00 in.) Diameter Barstock	Same as Above Two Conditions	1	K_C
Inconel 625	Nickel	Wrought	AMS 5666	BZCV	19.05 mm (0.75 in.) Diameter Barstock	1255°K (1800°F), 1 hr, Air Cool	1	LCF, CR, NT, ST
Inconel 625	Nickel	Wrought	AMS 5666	BYAP	107.95 mm (4.25 in.) Diameter Barstock	1255°K (1800°F), 1 hr, Air Cool	1	K_C
WASPALOY®	Nickel	Wrought	AMS 5706	L-1288K13	19.05 mm (0.75 in.) Diameter Barstock	1293°K (1870°F), 4 hr, Oil Quench 1116°K (1550°F), 4 hr, Air Cool 1033°K (1400°F), 4 hr, Air Cool	2	LCF, CR, ST
WASPALOY®	Nickel	Wrought	AMS 5706	BWKJ	50.8 by 10.16 mm (2.00 by 0.40 in) Barstock	Same as Above WASPALOY®	2	K_C, K_{TH}, K_T/K_C
Astroloy	Nickel	Wrought	PWA Specification	LKKC	447.0 mm (17.6 in.) Diameter 41.9 mm (1.65 in.) Thick Pancake Forging	1380°K (2025°F) 4 hr, Oil Quench 1144°K (1600°F) 8 hr, Air Cool 1255°K (1800°F) 4 hr, Air Cool 922°K (1200°F) 24 hr, Air Cool 1033°K (1400°F) 8 hr, Air Cool	2	CR, ST NT
Astroloy	Nickel	Wrought	PWA Specification	BYQO	445.8 mm (17.55 in.) Diameter 72.4 mm (2.9 in.) Thick Pancake Forging	Same as Above Astroloy Except Salt Quench From 1380°K	3	NT, CR
Hastelloy X	Nickel	Wrought	AMS 5754	BZCR	19.05 mm (0.75 in.) Diameter Barstock	1451°K (2150°F), 1 hr, Water Quench	1	LCF, NT, ST
Inconel 718	Nickel	Wrought	AMS 5832		Weld Wire	Annealed	1	K_C, NT, ST
Inconel 625	Nickel	Wrought	AMS 5837		Weld Wire	Annealed	1	K_C, NT, ST
IN-100	Nickel	Cast	PWA Specification	P-9245	12.7 mm (0.50 in.) Diameter by 139.7 mm (5.5 in.), 19.05 mm (0.75 in.) Diameter by 155.5 mm (6.1 in.) Cast Bars	1144°K (1600°F) 12 hr, Air Cool	2	LCF, HCF, CR, ST
MAR M-200 DS	Nickel	Cast	PWA Specification	P-9108	Directionally Solidified Cast Bar 12.7 mm (0.50 in.) Diameter by 139.7 mm (5.50 in.), 19.05 mm (0.75 in.) Diameter by 155.5 mm (6.12 in.)	1477°K (2200°F) 2 hr, Air Cool 1144°K (1600°F), 32 hr, Air Cool	[illegible]	HCF, ST

Table III-1. Materials Used to Determine the Susceptibility of Various Alloys to Environmental Degradation (Continued)

Material	Base	Form	Purchase Specification	Heat Code	As-Received Condition	As-Tested Condition (Heat Treatment)	Year Tested	Types of Tests[1]
MAR M-200 DS	Nickel	Cast	PWA Specification	P-9199	Directionally Solidified Cast Bar 15.88 mm (0.625 in.) Diameter by 127.0 mm (5.0 in.), 19.05 mm (0.75 in.) Diameter by 152.4 mm (6.0 in.)	Same as Above MAR M-200 DS	2	HCF, CR, NT
A-286	Iron	Wrought	AMS 5735	BZCV	19.05 (0.75 in.) Diameter Barstock	1255°K (1800°F) 1 hr, Oil Quench 1005°K (1350°F), 16 hr, Air Cool	1	LCF, CR, NT, ST
						Same as Above A-286	1, 3	K_C, CR
A-286	Iron	Wrought	AMS 5735	BXOY	114.3 mm (4.50 in.) Diameter Barstock			
AISI 347	Iron	Wrought	AMS 5646	BZCT	19.05 mm (0.75 in.) Diameter Barstock	1297°K (1875°F), 1 hr, Air Cool Cold Finished	1	LCF, HCF CR, NT, ST
AISI 347	Iron	Wrought	AMS 5646	BUJR	107.9 mm (4.25 in.) Diameter Barstock	1297°K (1875°F), 1 hr, Air Cool	1	K_C
Titanium 6AL-4V	Titanium	Wrought	AMS 4928	BZCR	19.05 mm (0.75 in.) Diameter Barstock	1019°K (1375°F), 1 hr, Air Cool	1	LCF, CR, NT, ST
Titanium 6AL-4V	Titanium	Wrought	AMS 4928	BZAX	114.3 mm (4.50 in.) Diameter Barstock	1019°K (1375°F), 1 hr, Air Cool	1	K_C
A-110	Titanium	Wrought	AMS 4926	BZCW	19.05 mm (0.75 in.) Diameter Barstock	922°K (1200°F), 1 hr, Air Cool	1	LCF, HCF, CR, NT, ST
A-110	Titanium	Wrought	AMS 4926	BZMP	114.3 mm (4.50 in.) Diameter Barstock	922°K (1200°F), 1 hr, Air Cool	1	K_C
Haynes 188	Cobalt	Wrought	PWA Specification	YFYR	12.7 mm (0.50 in.) and 15.87 mm (0.65 in.) Diameter Barstock	1450°K (2150°F) 30 min, Air Cool	2	LCF, CR
Haynes 188	Cobalt	Wrought	PWA Specification	YGDM	25.4 mm (1.0 in.) Diameter Barstock	1450°K (2150°F) 30 min, Air Cool	3	HCF, CR, NT

(1) Types of Tests:

LCF - Low-Cycle Fatigue
HCF - High-Cycle Fatigue
CR - Creep-Rupture
K_C - Fracture Toughness
K_{TH} - Threshold Stress Intensity
K_I/K_C - Cyclic Stress Intensity
NT - Notch Tensile
ST - Smooth Tensile

Table III-2. Chemical Composition of Materials Used to Determine the Susceptibility of Various Alloys to Environmental Degradation

Material	Heat Code	Elemental Composition - % by Weight Mn	Si	Cu	Ni	Cr	V	W	Ti	Co	Fe	Al	B	Zr	Mo	C	Sn	Cb + Ta	La	P	S	H_2	O_2	N_2
Inconel 718	BVTO	0.04	0.10	<0.05	53.38	18.18			1.02	0.080	18.15	0.48	0.0056		3.04	0.049		5.46		0.003	0.006			
Inconel 718	BZMK	<0.10	0.14	0.05	52.86	19.13			1.04	0.20	17.8	0.50	0.0040		3.11	0.04		5.37						
Inconel 625	BZCV	0.01	0.08		62.91	21.60			0.06	0.05	2.14	0.17			9.10	0.052		3.82		0.005	0.005			
Inconel 625	BYAP	0.02	0.10		62.79	21.92			0.20		1.79	0.18			9.05	0.04		3.92		0.004	0.005			
WASPALOY®	L1288K13	0.03	0.07	0.01	59.8	18.2			2.9	12.3	0.65	1.50	0.005	0.05	4.4					<0.015	<0.015			
WASPALOY®	BWKJ	0.02	0.07	0.03	53.04	18.5			2.89	13.68	0.70	1.39	0.003	0.06	4.58	0.04								
Astroloy	LKKC	0.015	0.05	0.02	55.31	15.1			3.31	16.65	0.15	3.99	0.0255	<0.01	5.3	0.08								
Astroloy	BYQO	0.02	0.05	0.07	54.13	15.4			3.3	16.9	0.5	4.45	0.025	<0.01	5.1	0.05								
Hastelloy X	BZCR	0.61	0.71		47.53	22.31		0.51		1.60	18.36		0.0014		8.27	0.074				0.016	0.006			
IN-100	P-9245	<0.10	<0.10		62.92	9.02	0.86		4.76	13.59	0.41	5.36	0.012	0.06	2.84	0.18								
MAR M-200 DS	P-9108	<0.10	<0.10	<0.10	60.1	8.62		12.57	1.80	10.54	<0.10	4.96	0.019	0.05				1.25 Cb						
MAR M-200 DS	P-9199	<0.10	<0.10	<0.10	62.6	8.08		12.0	1.99	9.15	0.14	4.97	0.015	0.04				0.92 Cb						
A-286	BZCU	1.62	0.48		25.1	15.3	0.26		2.2		53.2	0.31	0.002		1.36	0.08								
A-286	BXOY	1.74	0.81		25.9	14.4	0.29		2.23		53.1	0.27	0.010		1.25									
AISI 347	BZCT	2.0	0.94	<0.10	10.10	18.4					67.4				0.26	0.06		0.83			0.010			
AISI 347	BIJR	1.68	0.62	0.17	9.61	17.0					69.7				0.22	0.056		0.81		0.027	0.022			
Ti-6Al-4V	BZCS						3.98		89.1		0.21	6.59				0.030						0.007	0.17	0.008
Ti-6Al-4V	BZAX						4.29		88.9		0.18	6.58				0.040						0.090	0.11	0.006
A-110	BZCW								92.1		0.33	5.24				0.01	2.23					0.011	0.164	0.013
A-110	BZMP								92.62		0.029	5.15				0.04	2.15					0.004	0.18	0.01
Haynes 188	YFYR	0.62	0.29		22.70	20.6		13.45		40.1	2.06		0.004			0.09			0.04	0.010	0.006			
Haynes 188	YGDM	1.12	0.31		22.30	21.8		14.05		38.7	1.56		0.004			0.06			0.05	0.010	0.006			

Inconel 718 and Inconel 625 materials were tested in both the parent and welded condition. Specimen blanks were prepared, as shown in figure III-1, and manually gas-tungsten-arc (GTA) welded. The Inconel 718 blanks were welded using AMS 5832 as filler material; Inconel 625 blanks were welded using AMS 5837 filler material. Both materials were welded in the solution-anneal condition. The Inconel 718-welded blanks were then subjected to the heat treatment No. 2 (1313°K [1900°F] solution) described in table III-1. Inconel 625-welded specimen blanks were subjected to an 1255°K (1800°F), 1-hr, air-cooled stress-relief cycle.

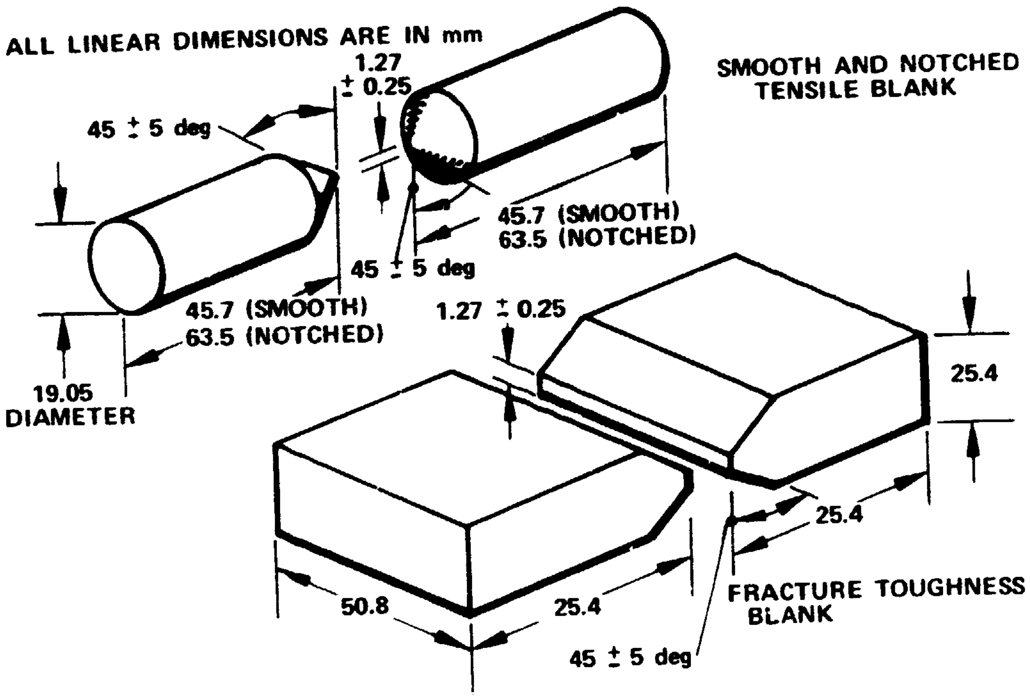

Figure III-1. Specimen Blank Preparation Prior To Welding FD 51835

B. TEST CASES AND MATERIALS

Helium, hydrogen, and anhydrous ammonia were used during the testing of specimens, and nitrogen was used as a preliminary purge gas. Hydrogen was provided under Military Specification P-27201, which requires the gas to have an oxygen content of less than 1 part per million. Analyses verified gas to be of this purity. Anhydrous ammonia was provided typically under Federal Specification O-A-445 and was rated 99.95% pure. Both the hydrogen and ammonia were supplied in liquid form and vaporized. The helium and hydrogen gases were used to directly provide the test environments. The hydrogen and water vapor environments were obtained by injecting distilled water into the pressurized hydrogen environment such that the water was vaporized by furnace heat within the test chamber.

The ammonia was used to obtain the environment noted as hydrazine in the test programs. Since hydrazine is considered to dissociate according to the following equations:

$$3N_2H_4 \longrightarrow 4NH_3 + N_2 + 1.553 \times 10^7 \text{ KgM } (1.443 \times 10^5 \text{ Btu) exothermic}$$

ammonia

$$4NH_3 \longrightarrow 2N_2 + 6H_2 - 8.523 \times 10^6 \text{ KgM } (0.792 \times 10^5 \text{ Btu) endothermic}$$

pure anhydrous ammonia, with the specimen heated to 951°K or 1144°K (1250°F or 1600°F), produced an environment similar to dissociated hydrazine.

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 using a modified 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

Surfaces of all specimens were machined(1) and finished to an average roughness of 16-μin. RMS or less except for one outer surface of the compact tensile fracture toughness specimens, which was machined to a roughness of 32-μin. RMS in accordance with ASTM E-399. Gage sections of specimens were polished prior to testing. The notch used for tensile specimens to obtain a stress concentration factor of 8.0 was designed according to Peterson(2) and was machined by grinding. Smooth tensile specimens had a gage section diameter of 6.37 mm (0.251 in.). Two types of specimens were used for the fracture mechanics testing. Compact tensile specimens were used for fracture toughness tests in the contract's first phase. Fracture mechanics tests under the second phase of the contract were conducted on center slot (or flaw) type specimens with a thickness of 2.54 mm (0.10 in.). The slot was machined into the specimen by use of electrical discharge machining (ELOX). The specimen was polished in the area of the slot.

Welded specimens were machined from blanks prepared as previously discussed; manual GTA welding was performed on oversized blanks to ensure specimen finished dimensions. Both the root pass and the finished weld were X-ray inspected and judged radiographically sound. Prior to finish machining, specimens were given a light etch to define weld location. After finish machining, all specimens were polished to produce desired surface finish.

A typical set of specimens is shown in figure III-2; specimen prints are listed in table III-3 and are shown in figures III-3 through III-9. Specimens prints are dimensioned in conventional units only.

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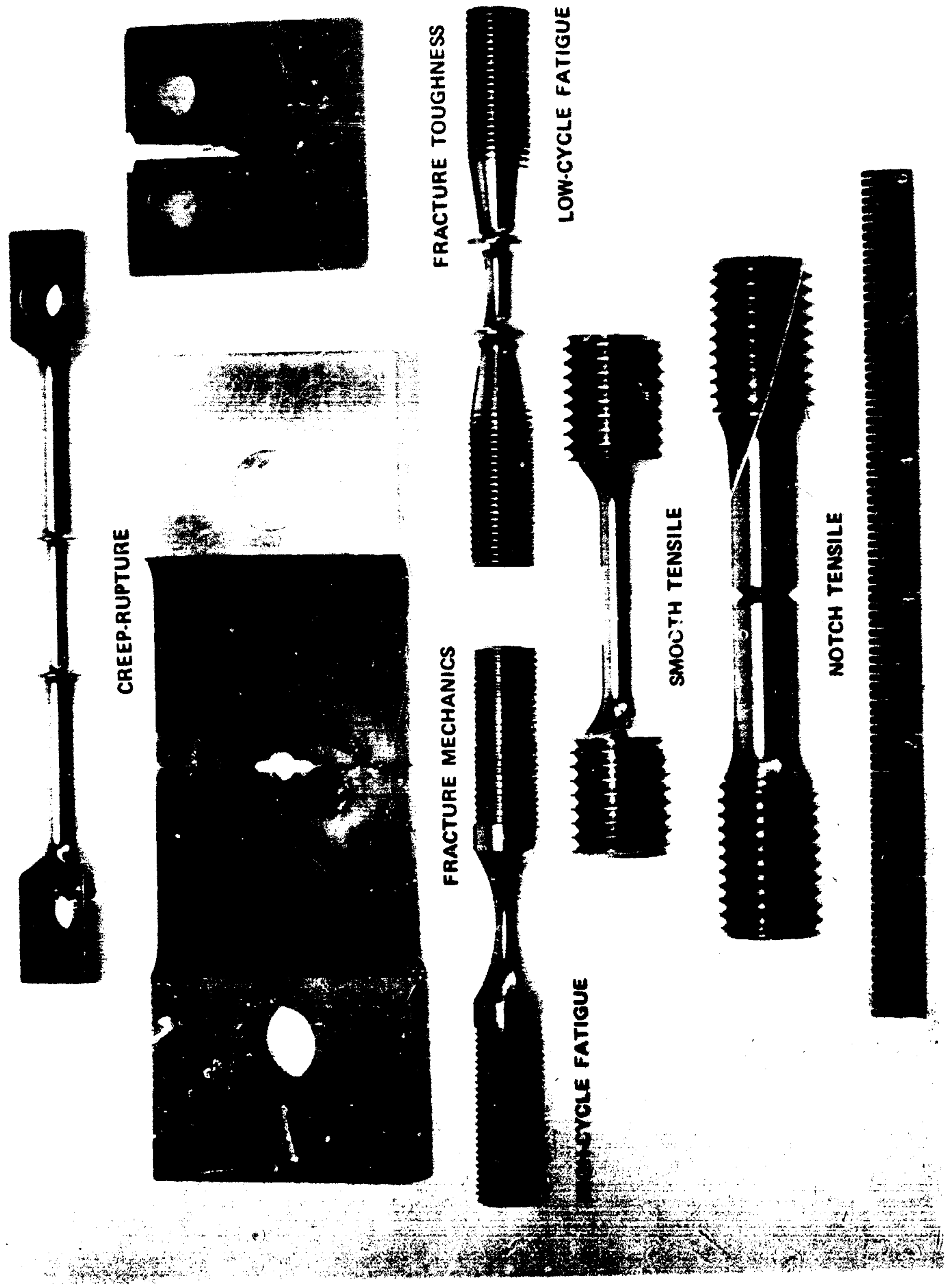
(1) Test specimens were machined by both the Pratt & Whitney Aircraft Laboratory Machine Shop and outside vendors operating under Pratt & Whitney Materials Control Laboratory surveillance and control.

(2) R. E. Peterson, Stress Concentration Design Factors, John Wiley & Sons, Inc., New York, 1953.

Table III-3. Specimens Used to Determine the Susceptibility of Various Alloys to Environmental Degradation

Name	Print Number	Figure
Constant Strain Low-Cycle Fatigue	FML 95500B	III-3
Smooth Axial Fatigue (High-Cycle Fatigue)	FML 95212B	III-4
Compact Tensile Fracture Toughness	FML 95559C	III-5
Center Slot (Notch) Fracture Mechanics	FML 95810	III-6
Flat End Creep-Rupture	FML 95623B	III-7
Notch Tensile	FML 95620B	III-8
Smooth Tensile	FML 95224B	III-9



FC 72531

Figure III-2. Typical Test Specimens Used to Determine Effect of High Pressure Gaseous Hydrogen Environments on Mechanical Properties of Materials

FML 95500B

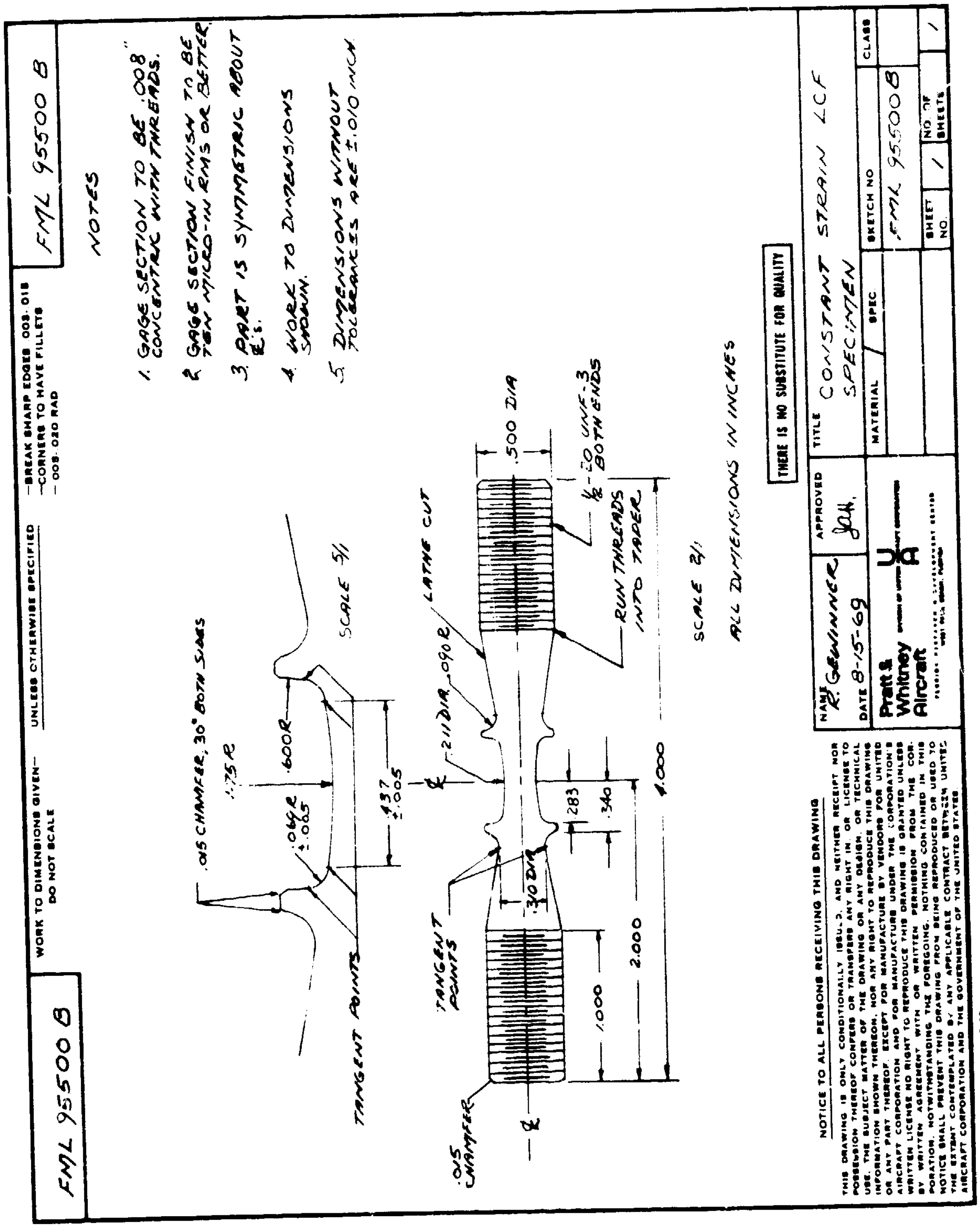
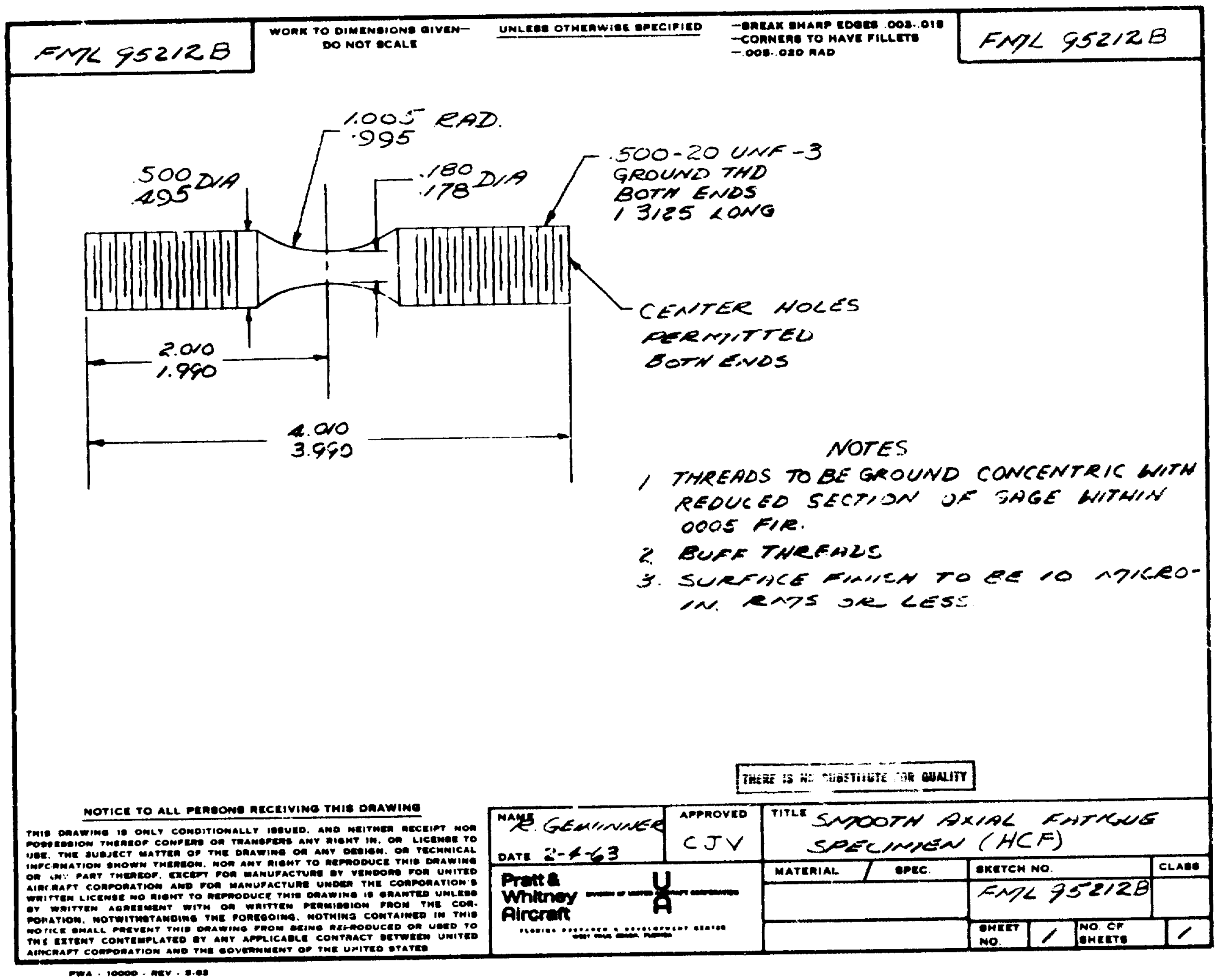


Figure III-3. Constant Strain Low-Cycle Fatigue Specimen



FML 95212B

Figure III-4. Smooth Axial High-Cycle Fatigue Specimen

FML 95559C

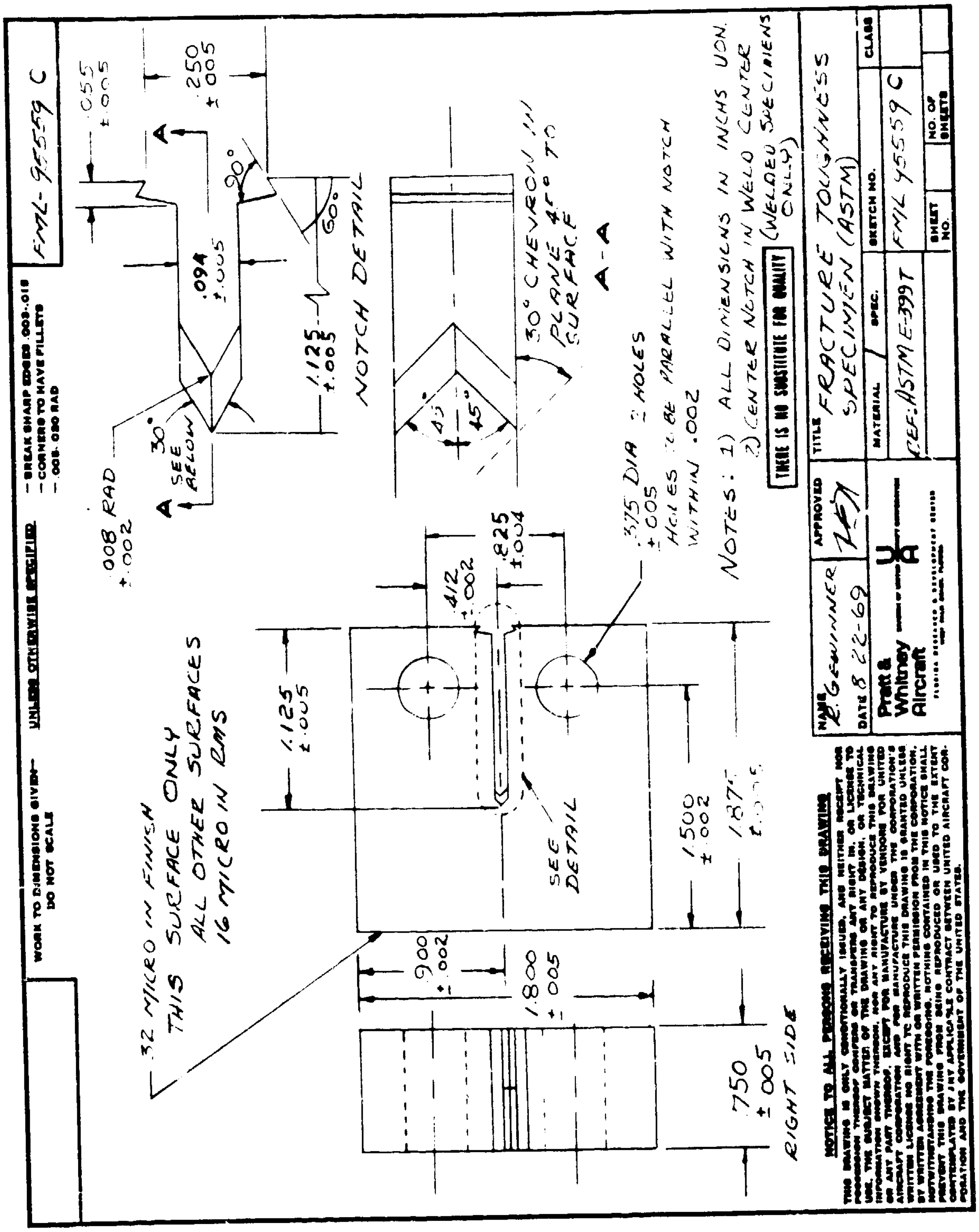


Figure III-5. Compact Tensile Fracture Toughness Specimen (ASTM)

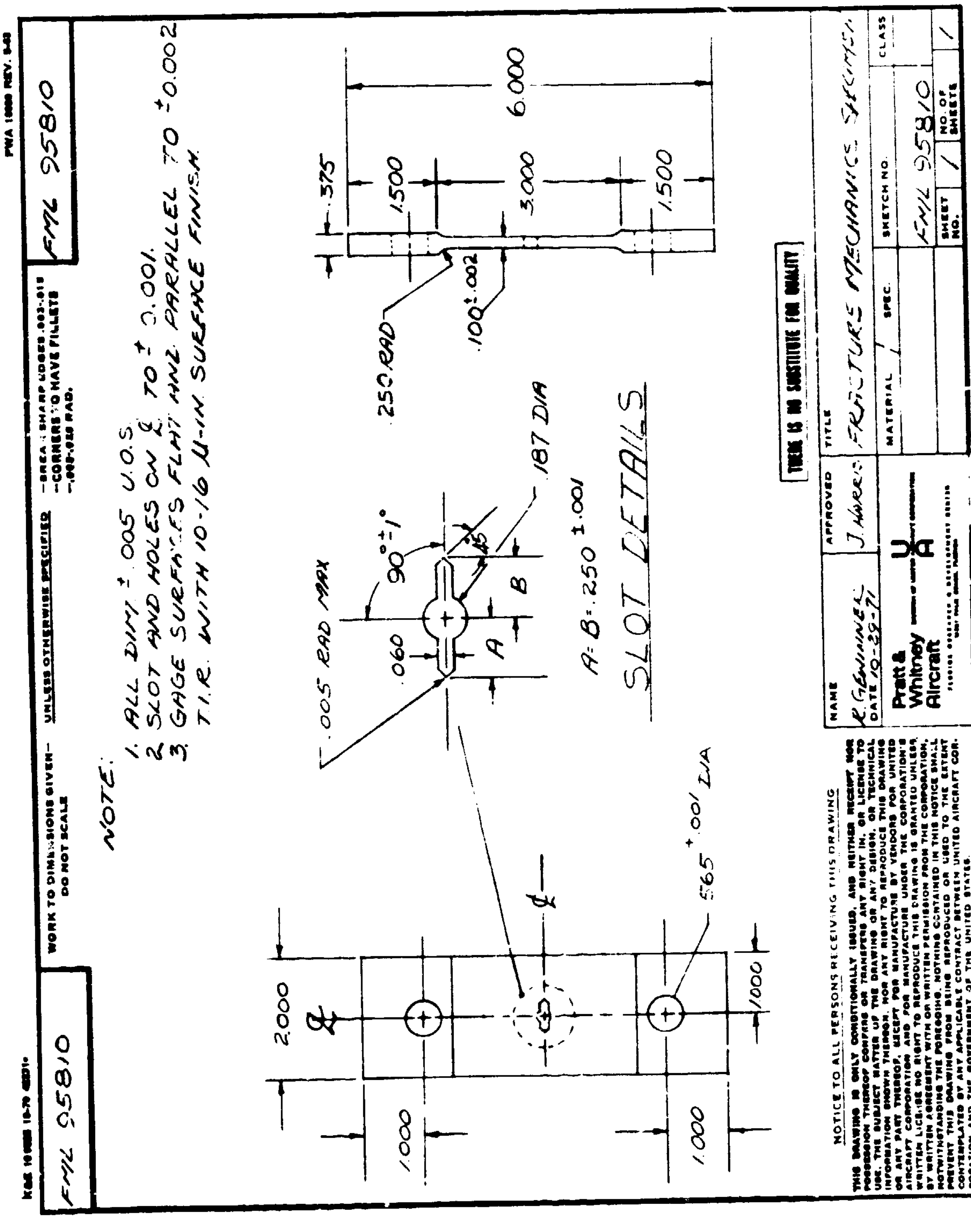


Figure III-6. Center Slot (Notch) Fracture Mechanics Specimen

FML 95810

FML 95623B

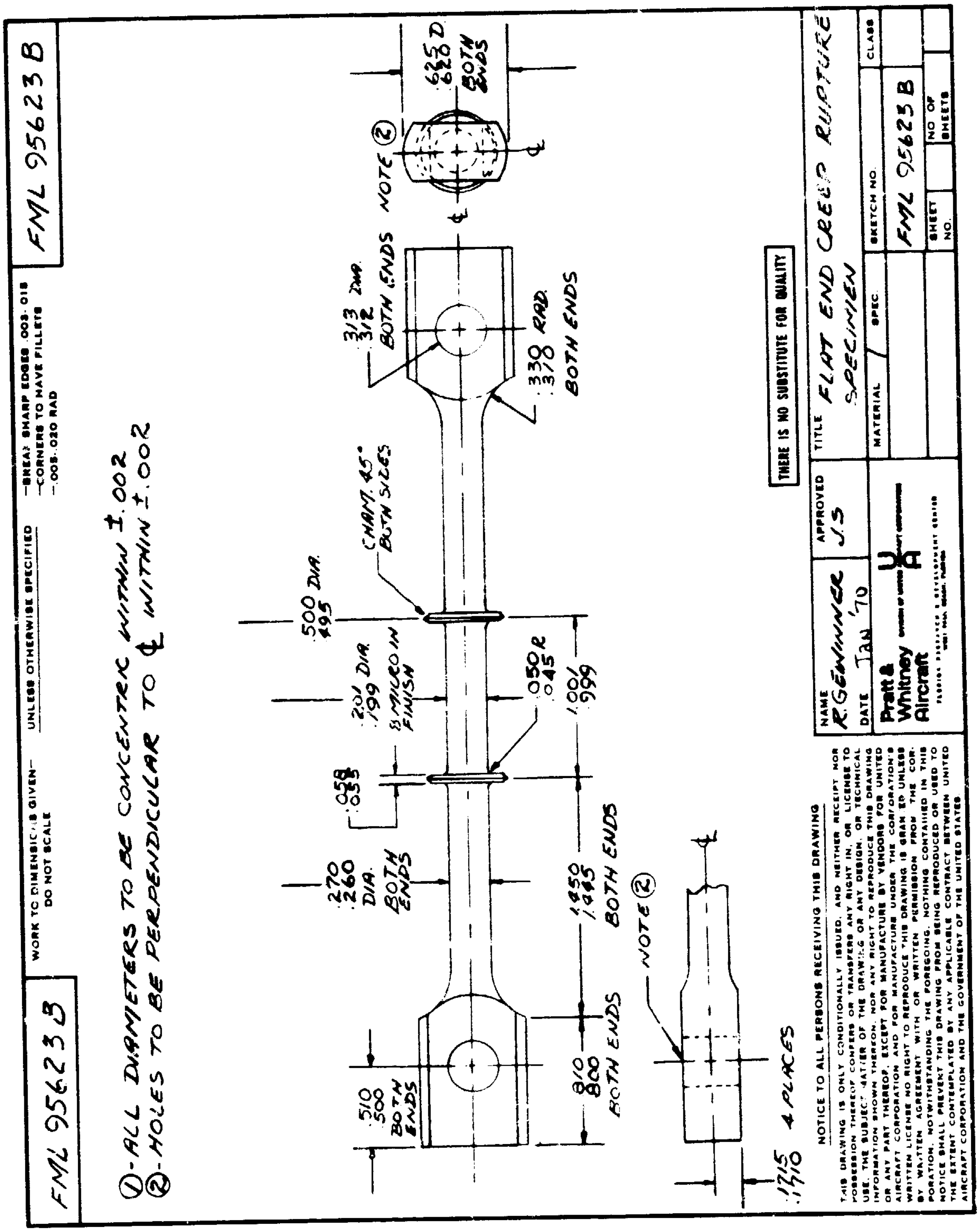
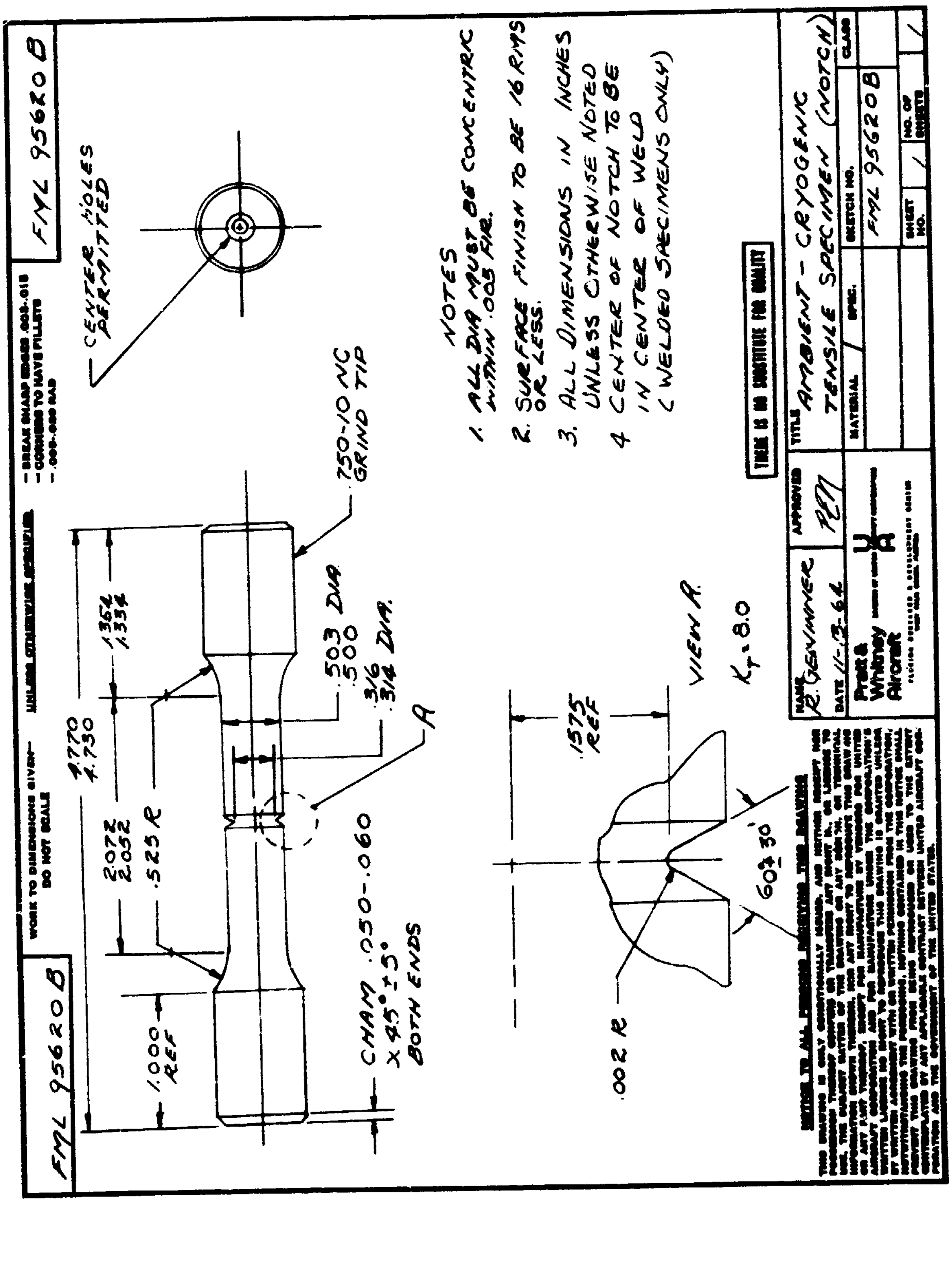


Figure III-7. Flat End Creep-Rupture Specimen



FML 95620B

Figure III-8. Notch Tensile Specimen

FML 95224B

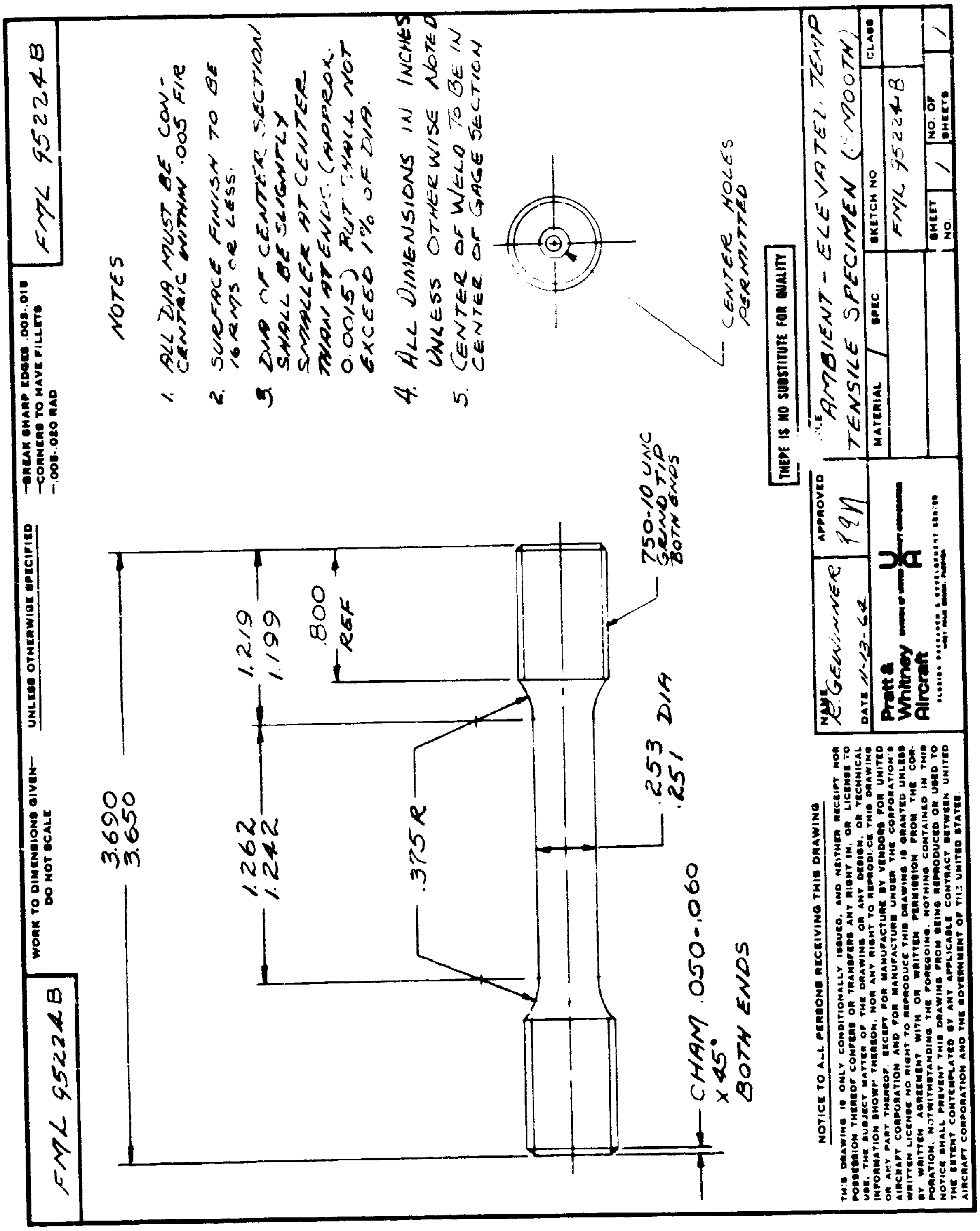


Figure III-9. Smooth Tensile Specimen

SECTION IV
HIGH-CYCLE FATIGUE

SECTION IV
HIGH-CYCLE FATIGUE

A. GENERAL

Load (stress) controlled high-cycle fatigue tests (HCF) were conducted on three nickel-, one iron-, one titanium-, and one cobalt-base alloys to determine the effect of high-pressure hydrogen and/or dissociated hydrazine (anhydrous ammonia) upon the high-cycle fatigue life of these materials at ambient and elevated temperatures and establish the susceptibility of these alloys to hydrogen and/or dissociated hydrazine degradation.

High-cycle fatigue tests were conducted in 34.5-MN/m^2 (5000-psig) gaseous helium and hydrogen at 300°K (80°F) on Inconel 718 (two heat treatments), AISI 347, and Titanium A-110, and at 951°K (1250°F) on Inconel 718 (two heat treatments), IN-100 and MAR M-200 DS. Additional HCF tests were conducted on the MAR M-200 DS material in 3.45-MN/m^2 (500-psig) hydrogen and dissociated hydrazine at 1144°K (1600°F). HCF tests were conducted on Haynes 188 material in 3.45-MN/m^2 (500-psig) dissociated hydrazine at 951°K (1250°F) only. Comparison of the HCF S-N curves established in helium to those established in other test media determined HCF life degradation due to high pressure hydrogen or dissociated hydrazine. Test stress levels for each material were selected to obtain specimen failure within 1000 to 100,000 test cycles.

B. CONCLUSIONS AND DISCUSSION

As a class, the nickel-base alloys were the most susceptible to environmental degradation of HCF life, with all exhibiting degradation. On a qualitative basis, MAR M-200 DS was most susceptible to HCF life degradation, followed by Inconel 718 (both heat treatments showed approximately the same) and IN-100. The iron- and titanium-base alloys, AISI 347 and titanium A-110, were not affected at 300°K (80°F). The cobalt alloy, Haynes 188, was tested in dissociated hydrazine (ammonia) only, and HCF degradation could not be established.

Inconel 718 alloy, with 1227°K (1750°F) and 1313°K (1900°F) solution plus age heat treatments, was tested at 300°K (80°F) and 951°K (1250°F) in helium and hydrogen atmospheres at 34.5 MN/m^2 (5000 psig). Both the 1313°K (1900°F) and 1227°K (1750°F) solutioned material were degraded in hydrogen to approximately the same degree, indicating microstructure did not significantly affect the high-cycle fatigue life in hydrogen of this material. Cyclic stress level had some influence on the degree of degradation as indicated by the slopes of the S-N curves (figures IV-1 and IV-2). However, both heat treatments show approximately the same degradation at both 300°K (80°F) and 951°K (1250°F), indicating HCF life degradation of this material in hydrogen atmosphere was not as dependent upon temperature as some of the other properties obtained in this program.

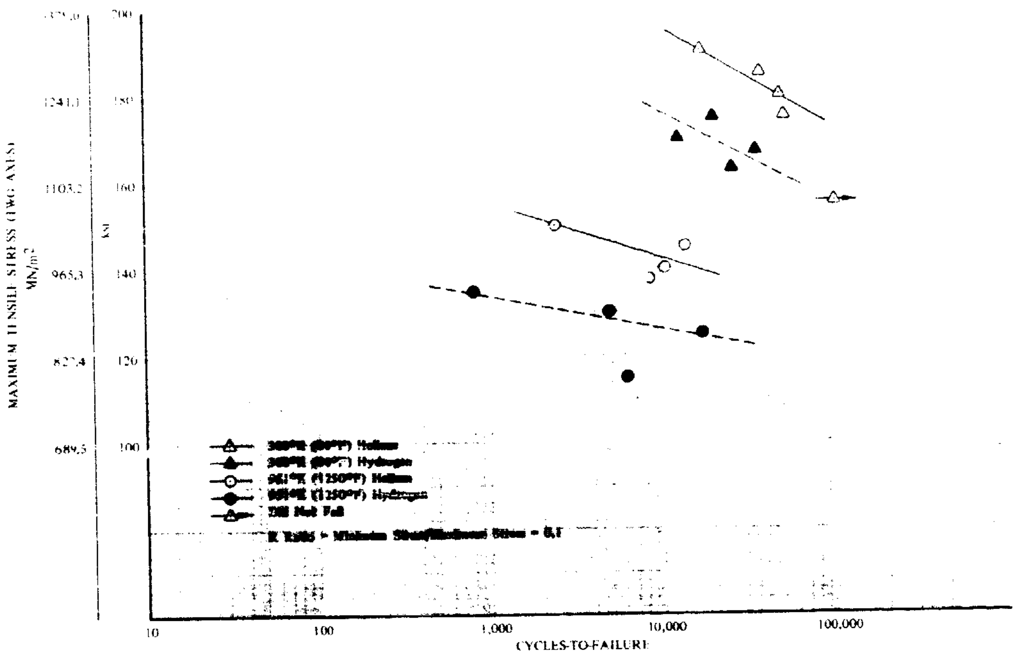


Figure IV-1. High-Cycle Fatigue Life of Inconel 718, 1227°K (1750°F) Solution Plus Age Heat Treatment at 300°K (80°F) and 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure DF 96475

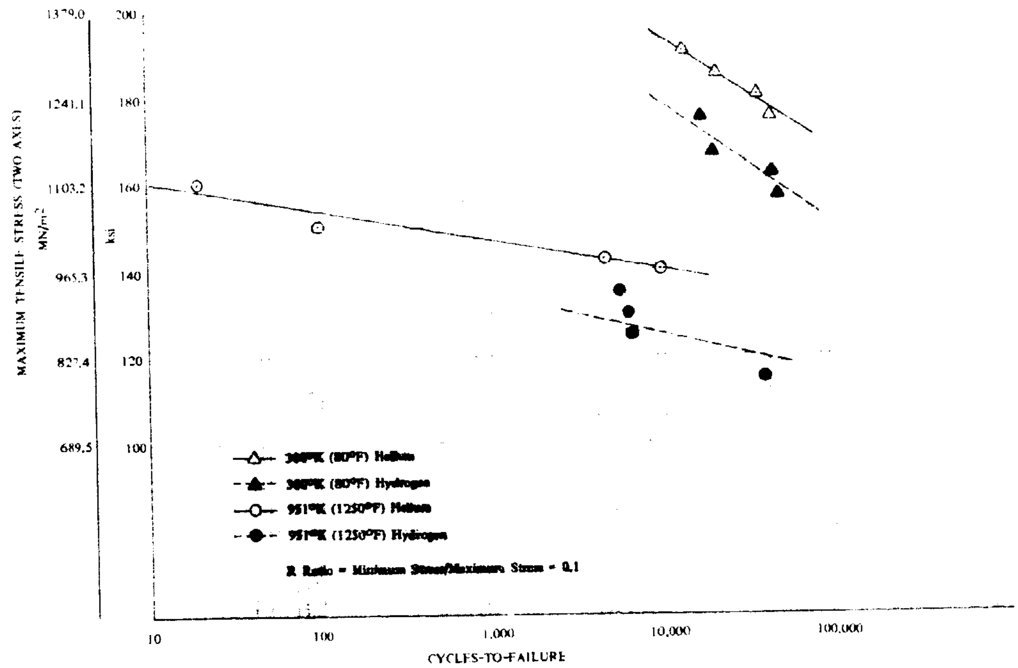


Figure IV-2. High-Cycle Fatigue Life of Inconel 718, 1313°K (1900°F) Solution Plus Age Heat Treatment at 300°K (80°F) and 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure DF 96476

IN-100 material was tested in 34.5-MN/m^2 (5000-psig) hydrogen at 951°K (1250° F) only and exhibited the lowest HCF life degradation of all the nickel-base alloys tested. The slope of the S-N curves in helium and hydrogen indicate the HCF life degradation of this material is not stress-level sensitive since the degree of degradation appears constant with increasing cyclic stress level (figure IV-3). Specimens tested in both helium and hydrogen were examined metallurgically after test, and one specimen tested in the hydrogen atmosphere exhibited excessive microshrinkage (figure IV-4).

MAR M-200 DS material was tested in 34.5-MN/m^2 (5000-psig) helium and hydrogen at 951°K (1250° F) (heat P9108) and in 3.45-MN/m^2 (500-psig) hydrogen and dissociated hydrazine at 1144°K (1600°F) (heat P9199). The HCF life of this alloy was degraded at 951°K (1250°F). Since there was not a helium baseline established at 1144°K (1600° F), nor any testing in 3.45-MN/m^2 (500-psig) hydrogen at 951°K (1250° F), the degradation due to 3.45-MN/m^2 (500-psig) hydrogen or dissociated hydrazine at 1144°K (1600° F) could not be established. Cyclic stress level and temperature influence the degree of degradation of this alloy in a hydrogen atmosphere as indicated by the S-N curves shown in figure IV-5.

Because of the test data scatter, all specimens tested in helium atmosphere were examined metallurgically after failure. The specimens showed numerous small fractures along the gage section, cracks through MC carbides and some incipient melting resulting from the 1477°K (2200° F) solution heat treat cycles. However, all specimens showed satisfactory grain orientation with some, but not excessive, micro-shrinkage evident (figure IV-6). The slope of the helium HCF baseline S-N curve was established by the half-cycle ultimate tensile strength (1208.7 MN/m^2 (175.3 ksi)) at 951°K (1250° F) in helium and the cycles-to-failure for a HCF specimen at the average maximum stress level (1068.7 MN/m^2 (155 ksi) tested.

AISI 347 alloy was tested in 34.5-MN/m^2 (5000-psig) helium and hydrogen atmospheres at 300° K (80° F) only. This material exhibited no HCF life degradation at these conditions (figure IV-7).

Titanium A-110 material was tested in 34.5-MN/m^2 (5000-psig) helium and hydrogen atmospheres at 300° K (80° F) only. Test results indicate no HCF life degradation within the 10,000- to 100,000-cycle test range (figure IV-8). Since all testing was accomplished at one pressure and temperature, no conclusions regarding degradation versus temperature or pressure can be made.

HCF testing of Haynes 188 alloy was accomplished in 3.45-MN/m^2 (500-psig) dissociated hydrazine (obtained by dissociating anhydrous ammonia) at 951°K (1250°F) only (figure IV-9). Since no comparable data are available, conclusions as to degree, if any, degradation of HCF life resulting from this media and temperature can only be speculative.

Test data are listed in table IV-1 for all materials and test conditions.

C. TEST PROCEDURE

Smooth, round specimens were used for the high-cycle fatigue tests discussed in this report. The test specimen is shown in Section III and is detailed by print FML 95212B.

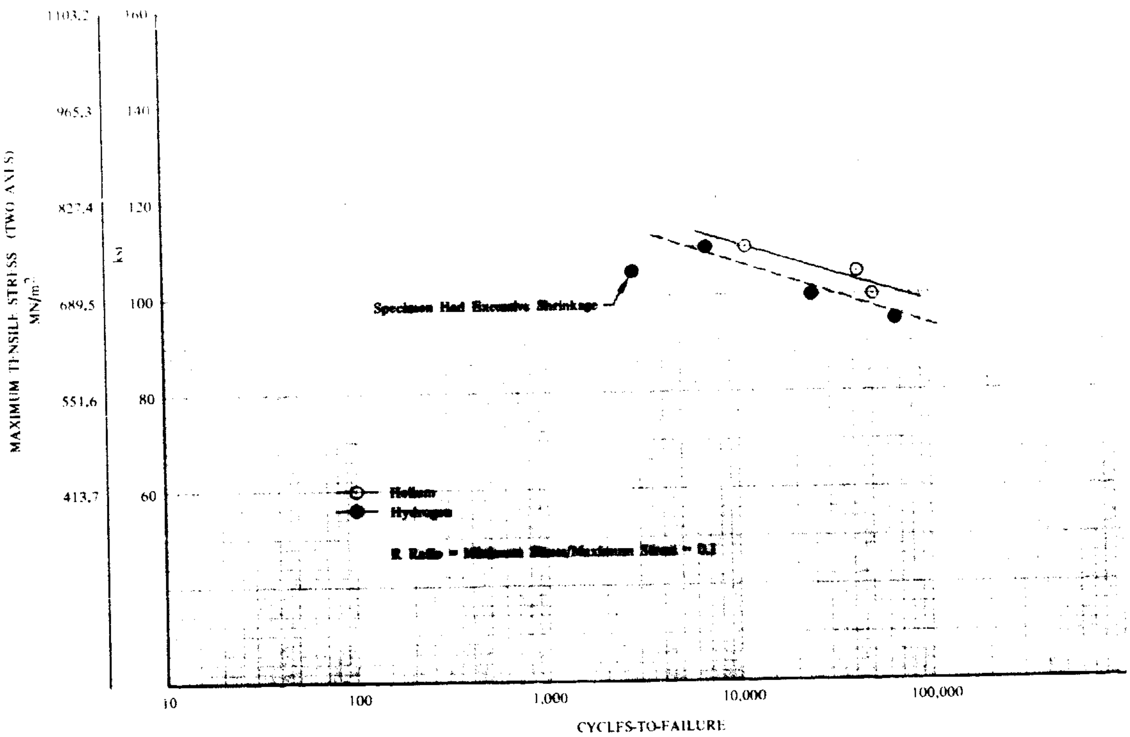


Figure IV-3. High-Cycle Fatigue Life of IN-100 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure

DF 96477

Figure IV-4. Macrograph of IN-100 HCF Test Specimen Showing Excessive Micro Shrinkage at Fracture Face

FD 72601

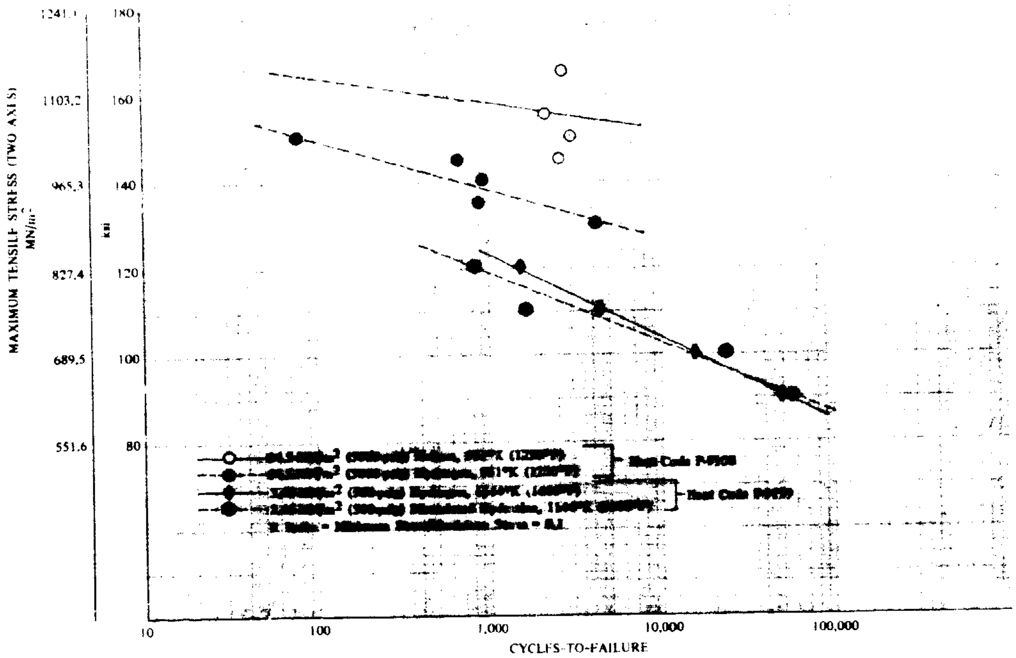


Figure IV-5. High-Cycle Fatigue Life of MAR M-200 DS at 951°K (1250°F) and 34.5-MN/m^2 (5000-psig) Helium and Hydrogen Gas at 1144°K (1600°F) and 3.45 MN/m^2 (500 psig) Hydrogen and Dissociated Hydrazine (Ammonia) Gas — DF 96478

Figure IV-6. Typical MAR M-200 DS Cross Section Adjacent to Fracture Face. Specimen Tested in Helium — FD 72602

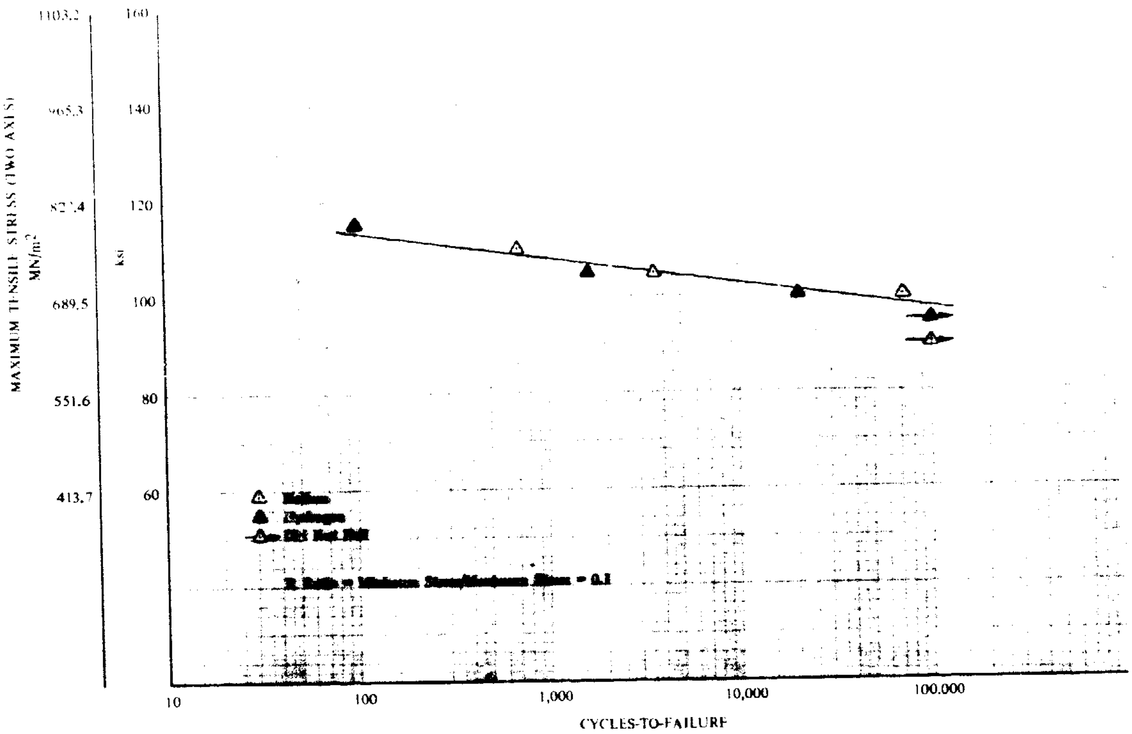


Figure IV-7. High-Cycle Fatigue Life of AISI 347 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96479

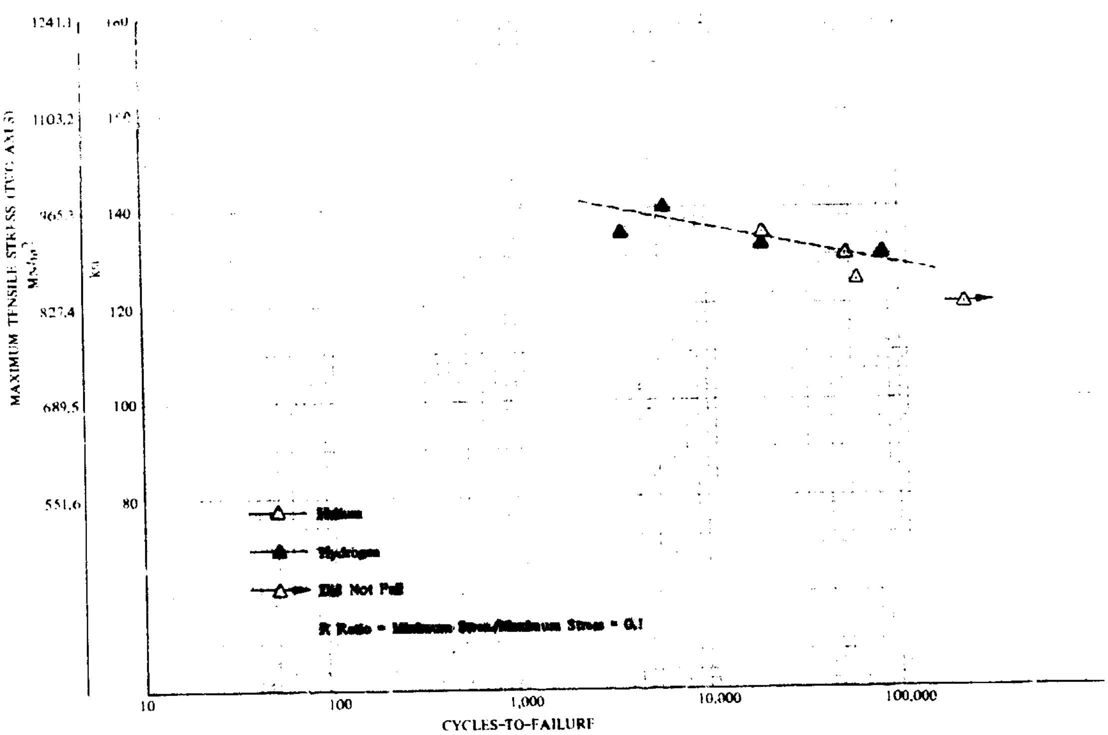


Figure IV-8. High-Cycle Fatigue Life of Titanium A-110 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96480

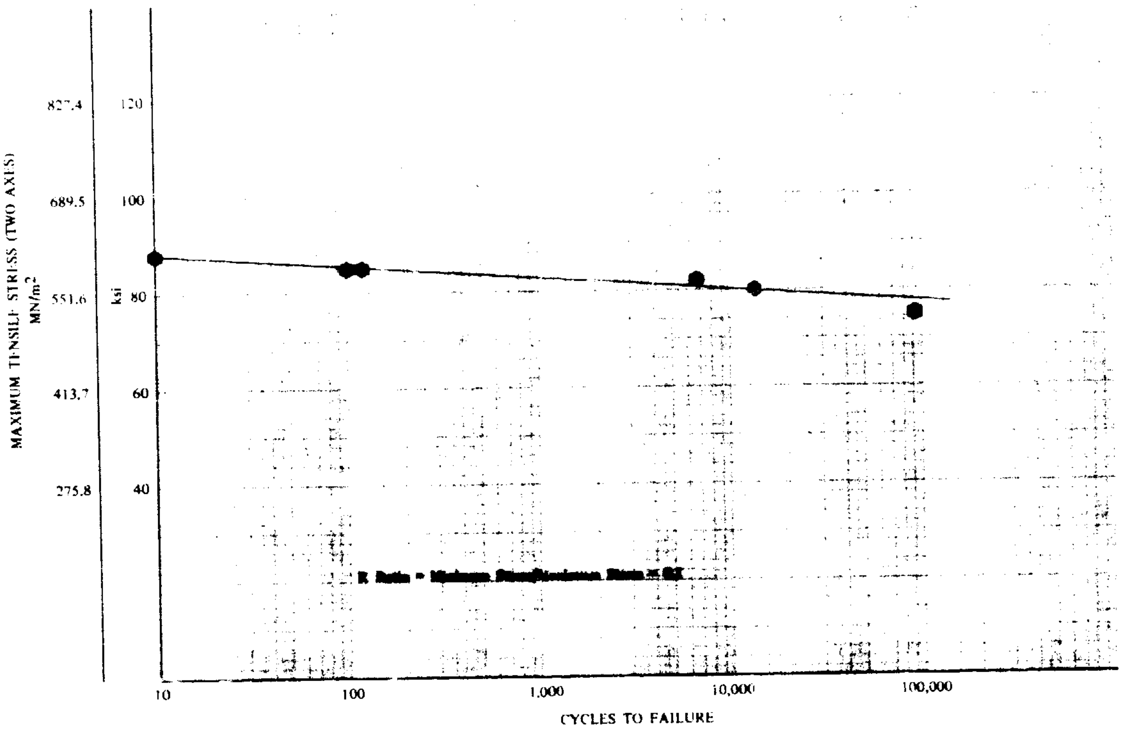


Figure IV-9. High-Cycle Fatigue Life of Haynes 188 at 951°K (1250°F) and 3.45-MN/m^2 (500-psig) Pressure Dissociated Hydrazine (Ammonia) DF 96481

After machining, specimen material was verified, and each specimen was inspected visually for machining discrepancies. Prior to testing, each specimen gage diameter (minimum cross section) was measured to the nearest 0.012 mm (0.0005 in.) with a micrometer and then cleaned with acetone.

The HCF life data were established by an axial load (stress) controlled tension-tension test. The test cycle was a tensile load that varied sinusoidally about a constant tensile preload at a cyclic rate of 20 Hz. All specimens were tested at an R ratio (minimum stress/maximum stress) of 0.1.

Table IV-1. High-Cycle Fatigue Properties of Materials in High Pressure Gaseous Environment

Material	Test Temperature, °K	Test Temperature, °F	Environment	Pressure, MN/m^2	Pressure, psi	Stress Level[(1)] Maximum, MN/m^2	Maximum, ksi	Minimum, MN/m^2	Minimum, ksi	Cycles-to-Failure
Inconel 718	300	80	Helium	34.5	5000	1069.00	155.0	106.90	15.5	100,000 Did Not Fail
1227°K (1750°F) Solution + Age Heat Treatment	300	80	Helium	34.5	5000	1206.59	175.0	120.66	17.5	50,900
	300	80	Helium	34.5	5000	1241.06	180.0	124.11	18.0	48,800
	300	80	Helium	34.5	5000	1275.53	185.0	127.55	18.5	37,800
	300	80	Helium	34.5	5000	1310.01	190.0	131.00	19.0	17,000
	300	80	Hydrogen	34.5	5000	1123.85	163.0	112.39	16.3	25,600
	300	80	Hydrogen	34.5	5000	1151.43	167.0	115.14	16.7	35,500
	300	80	Hydrogen	34.5	5000	1172.11	170.0	117.21	17.0	12,500
	300	80	Hydrogen	34.5	5000	1206.59	175.0	120.66	17.5	20,100
	951	1250	Helium	34.5	5000	965.27	140.0	96.53	14.0	10,200
	951	1250	Helium	34.5	5000	999.74	145.0	99.97	14.5	13,400
	951	1250	Helium	34.5	5000	1034.22	150.0	103.42	15.0	2,400
	951	1250	Helium	34.5	5000	954.93	137.5	95.49	12.75	8,400
	951	1250	Helium	34.5	5000	792.90	115.0	79.29	11.5	6,100
	951	1250	Hydrogen	34.5	5000	861.85	125.0	86.19	12.5	16,800
	951	1250	Hydrogen	34.5	5000	896.32	130.0	89.63	13.0	4,900
	951	1250	Hydrogen	34.5	5000	930.80	135.0	93.08	13.5	800
Inconel 718	300	80	Helium	34.5	5000	1206.59	175.0	120.66	17.5	42,400
1313°K (1900°F) Solution + Age Heat Treatment	300	80	Helium	34.5	5000	1241.06	180.0	124.11	18.0	35,800
	300	80	Helium	34.5	5000	1275.53	185.0	127.55	18.5	20,700
	300	80	Helium	34.5	5000	1310.01	190.0	131.00	19.0	13,200
	300	80	Hydrogen	34.5	5000	1082.48	157.0	108.25	15.7	46,400
	300	80	Hydrogen	34.5	5000	1116.95	162.0	111.70	16.2	43,500

(1) Stress Levels for R ratio $\frac{\text{Minimum Stress}}{\text{Maximum Stress}} = 0.1$

Table IV-1. High-Cycle Fatigue Properties of Materials in High Pressure Gaseous Environment (Continued)

	Test Conditions										
	Test Temperature,		Environ-	Pressure,		Stress Level,(1) Maximum,		Minimum,		Cycles-to-Failure
Material	°K	°F	ment	MN/m²	psi	MN/m²	ksi	MN/m²	ksi	
Inconel 718	300	80	Hydrogen	34.5	5000	1151.43	167.0	115.14	16.7	19,540
1313°K (1900°F)	300	80	Hydrogen	34.5	5000	1206.59	175.0	120.66	17.5	16,820
Solution + Age	951	1250	Helium	34.5	5000	965.27	140.0	96.53	14.0	9,600
Heat Treatment	951	1250	Helium	34.5	5000	982.51	142.5	98.25	14.25	4,530
	951	1250	Helium	34.5	5000	1034.22	150.0	103.42	15.0	100
	951	1250	Helium	34.5	5000	1103.17	160.0	110.32	16.0	20
	951	1250	Hydrogen	34.5	5000	792.90	115.0	79.29	11.5	37,200
	951	1250	Hydrogen	34.5	5000	861.85	125.0	86.19	12.5	6,500
	951	1250	Hydrogen	34.5	5000	930.80	135.0	93.08	13.5	5,560
	951	1250	Hydrogen	34.5	5000	896.32	130.0	89.63	13.0	6,200
IN-100	951	1250	Helium	34.5	5000	689.48	100.0	68.95	10.0	50,600
	951	1250	Helium	34.5	5000	723.95	105.0	72.40	10.5	43,000
	951	1250	Helium	34.5	5000	758.42	110.0	75.84	11.0	11,100
	951	1250	Hydrogen	34.5	5000	655.00	95.0	65.50	9.5	60,700
	951	1250	Hydrogen	34.5	5000	689.48	100.0	68.95	10.0	24,400
	951	1250	Hydrogen	34.5	5000	723.95	105.0	72.40	10.5	2,860
	951	1250	Hydrogen	34.5	5000	758.42	110.0	75.84	11.0	6,900
MAR M-200 DS	951	1250	Helium	34.5	5000	999.74	145.0	99.97	14.5	2,630
Heat Code P-9108	951	1250	Helium	34.5	5000	1034.22	150.0	103.42	15.0	3,100
	951	1250	Helium	34.5	5000	1069.00	155.0	106.90	15.5	2,200
	951	1250	Helium	34.5	5000	1138.63	165.0	113.86	16.5	2,800
	951	1250	Hydrogen	34.5	5000	896.32	130.0	89.63	13.0	4,265
	951	1250	Hydrogen	34.5	5000	930.00	135.0	93.00	13.5	890
	951	1250	Hydrogen	34.5	5000	965.26	140.0	96.53	14.0	935

(1)Stress Levels for R ratio $\frac{\text{Minimum Stress}}{\text{Maximum Stress}}$ 0.1

Table IV-1. High-Cycle Fatigue Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Environment	Test Conditions: Pressure, MN/m²	Pressure, psi	Stress Level(1): Maximum, MN/m²	Maximum, ksi	Maximum, MN/m²	Maximum, ksi	Cycles-to-Failure
MAR M-200 DS	951	1250	Hydrogen	34.5	5000	999.74	145.0	99.97	14.5	680
Heat Code P-9108	951	1250	Hydrogen	34.5	5000	1034.22	150.0	103.42	15.0	80
MAR M-200 DS	1144	1600	Hydrogen	3.45	500	827.38	120.0	82.74	12.0	1,530
Heat Code P-9199	1144	1600	Hydrogen	3.45	500	758.43	110.0	75.84	11.0	4,430
	1144	1600	Hydrogen	3.45	500	689.48	100.0	68.95	10.0	15,800
	1144	1600	Hydrogen	3.45	500	620.53	90.0	62.05	9.0	49,400
	1144	1600	Dissociated Hydrazine(2)	3.45	500	827.38	120.0	82.74	12.0	813
	1144	1600	Dissociated Hydrazine	3.45	500	758.43	110.0	75.84	11.0	1,630
	1144	1600	Dissociated Hydrazine	3.45	500	689.48	100.0	68.95	10.0	23,700
	1144	1600	Dissociated Hydrazine	3.45	500	620.53	90.0	62.05	9.0	53,000
AISI 347	300	80	Helium	34.5	5000	620.53	90.0	62.05	9.0	100,000 + (did not fail)
	300	80	Helium	34.5	5000	689.48	100.0	68.95	10.0	70,800
	300	80	Helium	34.5	5000	723.95	105.0	72.40	10.5	3,640
	300	80	Helium	34.5	5000	758.43	110.0	75.84	11.0	700
	300	80	Hydrogen	34.5	5000	655.00	95.0	65.50	9.5	100,000 + (did not fail)
	300	80	Hydrogen	34.5	5000	689.48	100.0	68.95	10.0	20,300
	300	80	Hydrogen	34.5	5000	723.95	105.0	72.40	10.5	1,620
	300	80	Hydrogen	34.5	5000	792.90	115.0	79.29	11.5	100

(1) Stress Levels for R ratio $\frac{\text{Minimum Stress}}{\text{Maximum Stress}} = 0.1$

(2) Dissociated Anhydrous Ammonia

Table IV-1. High-Cycle Fatigue Properties of Materials in High Pressure Gaseous Environment (Continued)

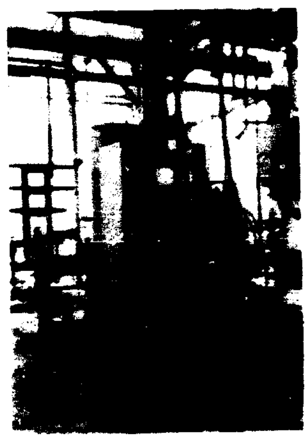
Material	Test Temperature, °K	Test Temperature, °F	Environment	Test Conditions: Pressure, MN/m^2	Pressure, psi	Stress Level,(1) Maximum, MN/m^2	Stress Level,(1) Maximum, ksi	Stress Level,(1) Maximum, MN/m^2	Stress Level,(1) Maximum, ksi	Cycles-to-Failure
A-110	300	80	Helium	34.5	5000	827.38	120.0	82.74	12.0	196,600 (did not fail)
	300	80	Helium	34.5	5000	861.85	125.0	86.19	12.5	51,900
	300	80	Helium	34.5	5000	896.32	130.0	89.63	13.0	46,900
	300	80	Helium	34.5	5000	930.80	135.0	93.08	13.5	17,500
	300	80	Hydrogen	34.5	5000	896.32	130.0	89.63	13.0	73,600
	300	80	Hydrogen	34.5	5000	913.66	132.5	91.37	13.25	17,200
	300	80	Hydrogen	34.5	5000	930.80	135.0	93.08	13.5	3,200
	300	80	Hydrogen	34.5	5000	965.27	140.0	96.53	14.0	5,300
Haynes 188	951	1250	Dissociated Hydrazine(2)	3.45	500	517.11	75.0	51.71	7.5	90,900
	951	1250	Dissociated Hydrazine	3.45	500	551.58	80.0	55.16	8.0	13,300
	951	1250	Dissociated Hydrazine	3.45	500	565.37	82.0	56.54	8.2	6,690
	951	1250	Dissociated Hydrazine	3.45	500	586.05	85.0	58.61	8.5	100
	951	1250	Dissociated Hydrazine	3.45	500	586.05	85.0	58.61	8.5	120
	951	1250	Dissociated Hydrazine	3.45	500	606.74	88.0	60.67	8.8	0.5

All Tests Conducted at a Cyclic Rate of 20 Hz

(1) Stress levels for R ratio $\frac{\text{Minimum Stress}}{\text{Maximum Stress}} = 0.1$

(2) Dissociated Anhydrous Ammonia

Tests were conducted using a closed loop on load, servohydraulic test machine located in an isolated test cell (figure IV-10). The test specimen was mounted in a heavy walled pressure vessel attached to the upper platen of the test machine. Test specimens were mounted in the pressure vessel load frame by threading each end into tapped loading rods (top and bottom) and securing with locknuts. The specimen and the sealed pressure vessel were subjected to a purge cycle, consisting of nitrogen purge, evacuation, two successive pop purges with the test media and final pressurization to test pressure.



FC 23389
Test Machine Located in Isolated Test Cell

Test Vessel Open

Test Vessel Closed

Figure IV-10. High Pressure Gaseous Environment High-Cycle Fatigue Testing Equipment FD 53128

Specimen load was measured by a strain-gage load cell, integral with the specimen load rod and inside the pressure vessel. Before the initial test and periodically throughout the test program, the load cell calibration was checked (using an instrumented and calibrated specimen) at 34.5 MN/m^2 (5000 psig) pressure, so that axial tensile loads on the specimen due to high pressure acting over differential specimen and loading rod areas could be compensated for by the loading system. Since the load cell was adjacent to, and calibrated to give absolute specimen load, friction loss through the loading rod O-ring seals was of no consequence. Electrical connections to all internal strain gages, load cell, thermocouples, and heating devices were made through the bottom of the pressure vessel via an instrumentation manifold and high pressure bulkhead connectors. During testing, the load cycle and number of cycles-to-failure were constantly monitored on a calibrated oscilloscope and electronic counter using the internal load cell output.

Elevated temperature testing was accomplished using a dc power supply and high-power-density, single-zone furnace mounted inside the pressure vessel around the test specimen. Analysis of hydrogen gas samples, before and after the specimen tests, indicated required gas purity was obtained. Thermocouples looped around the specimen minimum cross section were used to monitor and control specimen temperature during each test.

Test system shutdown was provided at the instant of specimen failure by a linear variable differential transformer, which sensed load rod position, in combination with a meter relay. This proved an accurate method of determining the total number of cycles-to-failure.

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SECTION V
LOW-CYCLE FATIGUE

A. INTRODUCTION

Low-cycle fatigue (LCF) tests were conducted to determine degradation due to high pressure gaseous hydrogen of six nickel-base, two iron-base, two titanium-base, and one cobalt-base alloys. Comparison of results of axial strain tests in a high pressure hydrogen environment to results of similar tests in a helium environment established the degradation in cyclic life due to the hydrogen environment. The low-cycle fatigue tests performed under the contract were of the strain-controlled type, with the material cycling through a constant total (elastic plus plastic) strain range (figure V-1) until complete specimen fracture.

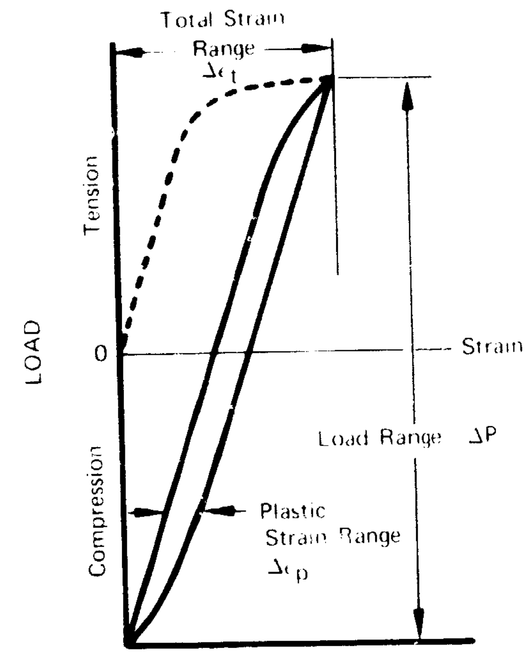

Figure V-1. Typical Load-Strain Hysteresis Curve Obtained During a Specimen Low-Cycle Fatigue Test

FD 48672

B. CONCLUSIONS AND DISCUSSION

Except for AISI 347 and MAR M-200 DS, materials tested exhibited some degradation in LCF life due to high-pressure hydrogen in at least one of the conditions tested. This degradation was dependent upon both temperature and strain range. As a class, the nickel-base alloys were most susceptible to hydrogen effects, followed by the titanium-base and cobalt-base alloys, with the iron-base alloys tested the least susceptible. It must be emphasized that the conclusions made herein were based on limited numbers of tests, particularly in the cases of IN-100, MAR M-200 DS, WASPALOY®, Haynes 188, and on extrapolated data. For these reasons, the conclusions must be viewed in a qualitative rather than quantitative manner.

Two alloys, Inconel 718 at 111°K (-260°F) and MAR M-200 DS at 951°K (1250°F), had greater LCF life in hydrogen than in helium, indicating a beneficial effect of the hydrogen environment at those conditions. Metallurgical and surface evaluations of the failed specimens did not reveal conditions that would significantly contribute to this occurrence. It is believed that additional work to define the environmental degradation mechanism would explain this occurrence; unfortunately that work was beyond the scope of this program.

Inconel 718 material was the most thoroughly investigated. Two heat treatments, differing in solution temperature only, were applied to specimens from the same stock. The base material was fine grained, fully recrystallized. The primary difference in microstructure was the larger grain size produced by the 1313°K (1900°F) solution treatment than by the 1227°K (1750°F) solution treatment.

The 1313°K (1900°F) solutioned material was slightly more degraded in hydrogen than the 1227°K (1750°F) solutioned material, indicating that microstructure affects LCF performance with small grain, recrystallized structures more desirable. (Investigations by Harris and VanWanderham[1] have also described this relationship.) Cyclic strain level and temperature also influenced the degree of degradation (figures V-2 through V-9). The 1227°K (1750°F) solutioned material had increased degradation at increased cyclic strain levels. The 1313°K (1900°F) solutioned material did not exhibit this trend. The most influential effect on LCF life resulted from temperature. Both materials were less susceptible to life degradation at 951°K (1250°F) than at 300°K (80°F). The 1313°K (1900°F) solutioned material was tested at 111, 300, and 951°K (-260, 80, and 1250°F). A plot of degradation vs temperature (figure V-9) indicates that degradation is most severe in the range of temperatures around 300°K (80°F), with decreasing degradation with decreasing temperature. In fact, at a temperature of 111°K (-260°F) LCF life in hydrogen was significantly better than LCF life in helium at that temperature. The reason for this great improvement in LCF life at cryogenic temperature is not presently understood. It is believed that the 1227°K (1750°F) solutioned material and perhaps other nickel-base alloys will also show similar influences of temperature. It is clear, however, that Inconel 718 LCF life is most severely degraded in the room temperature range. Additional testing at temperature ranges between 266 and 589°K (-100 and 600°F) would define the point of inflection in the temperature-degradation curve.

The remaining nickel-base alloys, IN-100, WASPALOY, Inconel 625, Hastelloy X, and MAR M-200 DS, were tested at only one temperature; therefore, no conclusions as to effect of temperature were reached. Inconel 625 and Hastelloy X were tested at 300°K (80°F), and LCF life of both was degraded (figures V-10 through V-13). In fact, Inconel 625 was the most severely degraded of all materials tested at room temperature. IN-100 (cast), WASPALOY, and MAR M-200 DS (cast) (figures V-14 through V-16) were tested at 951°K (1250°F) and were ranked in that order as to degree of degradation. IN-100 was the most severely degraded of all materials tested at 951°K (1250°F).

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(1) VanWanderham, M., and J. A. Harris, Jr., "Low-Cycle Fatigue of Metals in High Pressure Gaseous Hydrogen at Cryogenic, Ambient, and Elevated Temperatures," presented to the 1971 WESTEC Conference, Los Angeles, California.

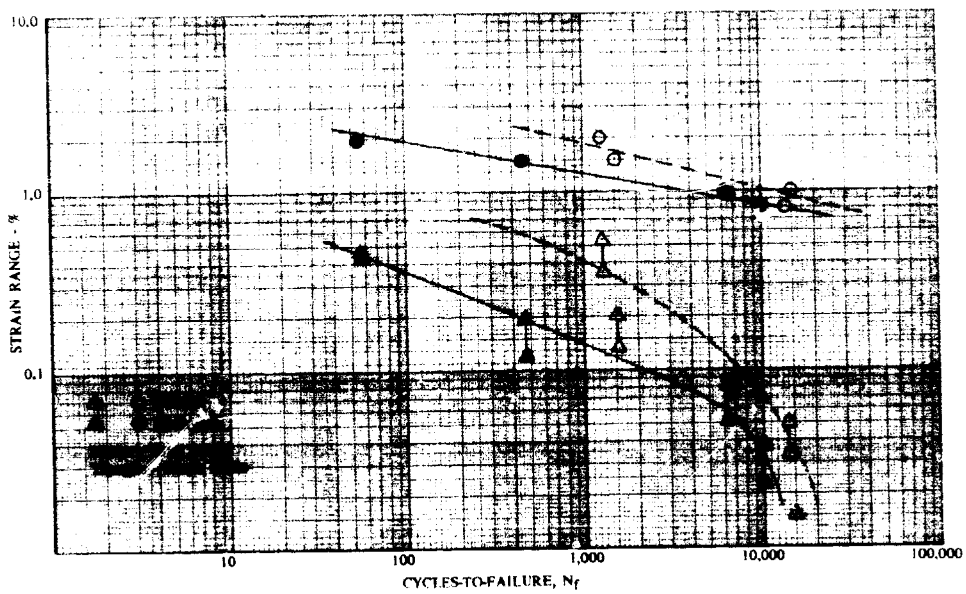


Figure V-2. Low-Cycle Fatigue Life of Inconel 718 With 1227°K (1750°F) Solution Plus Age Heat Treatment at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96401

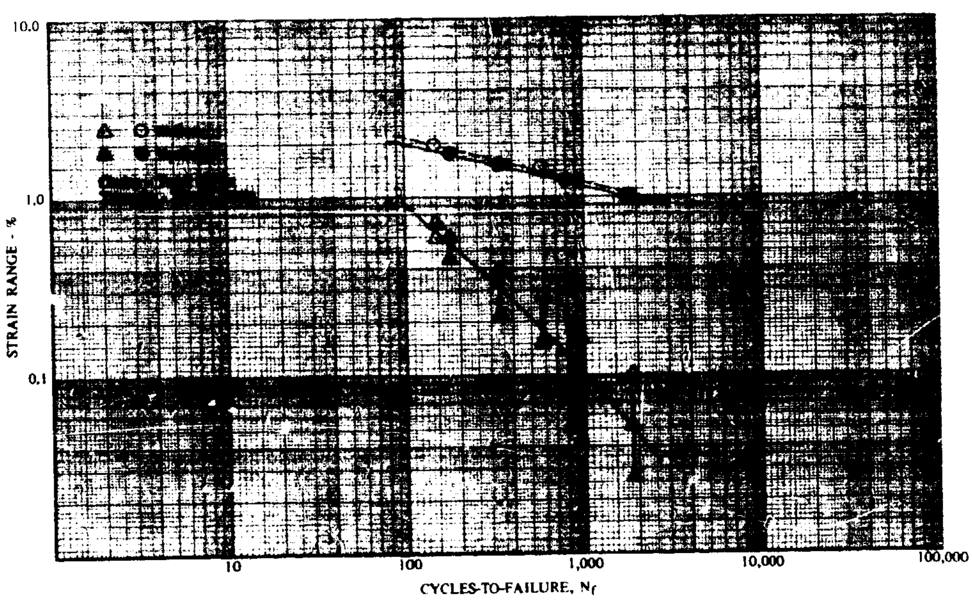


Figure V-3. Low-Cycle Fatigue Life of Inconel 718 With 1227°K (1750°F) Solution Plus Age Heat Treatment at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) pressure — DF 96402

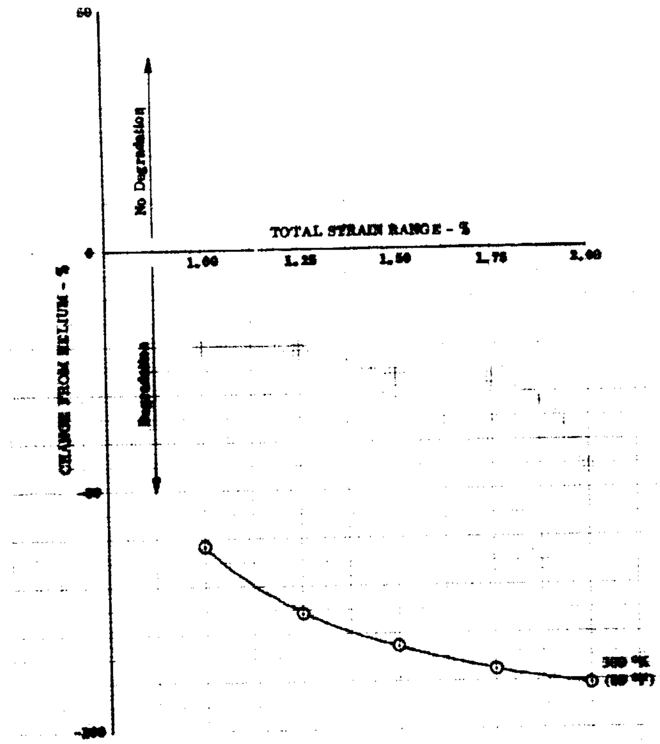


Figure V-4. Effect of Gaseous Hydrogen and Strain Range on Low-Cycle Fatigue Life of Inconel 718 With 1227°K (1750°F) Solution Plus Age Heat Treatment at 34.5 MN/m^2 (5000 psig) Pressure — DF 96403

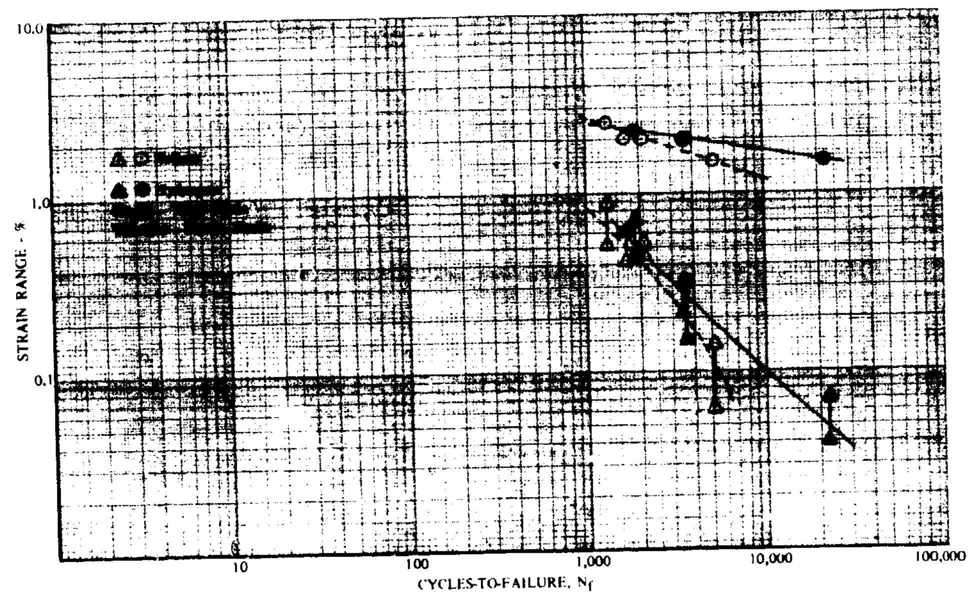


Figure V-5. Low-Cycle Fatigue Life of Inconel 718 With 1313°K (1900°F) Solution Plus Age Heat Treatment at 111°K (-260°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96404

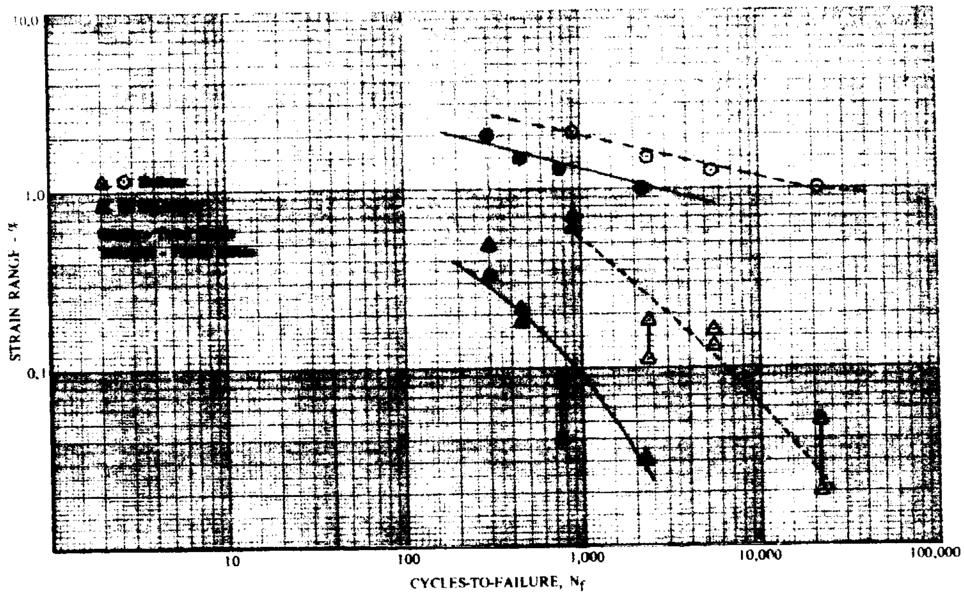


Figure V-6. Low-Cycle Fatigue Life of Inconel 718 With 1313°K (1900°F) Solution Plus Age Heat Treatment at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96405

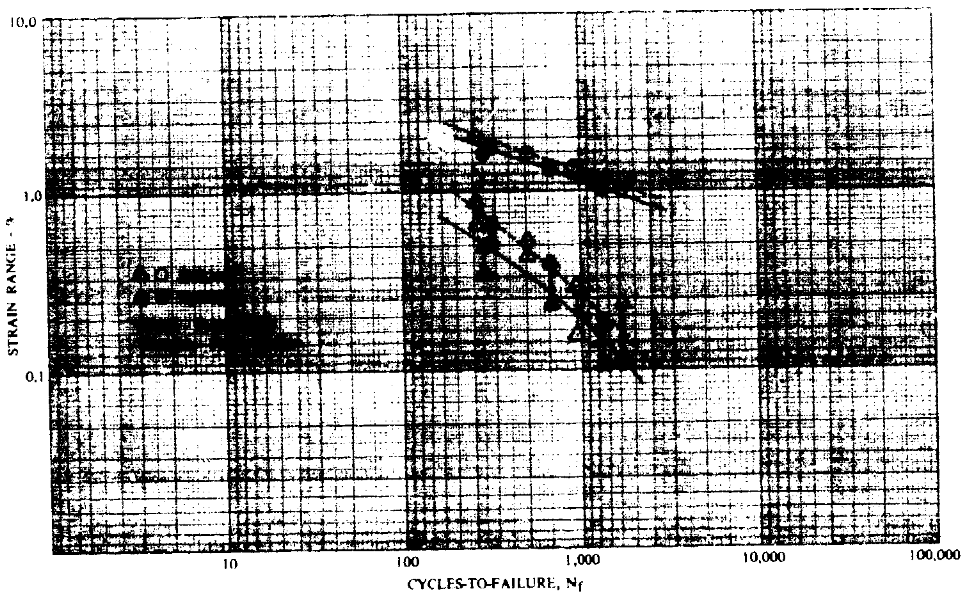


Figure V-7. Low-Cycle Fatigue Life of Inconel 718 With 1313°K (1900°F) Solution Plus Age Heat Treatment at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96406

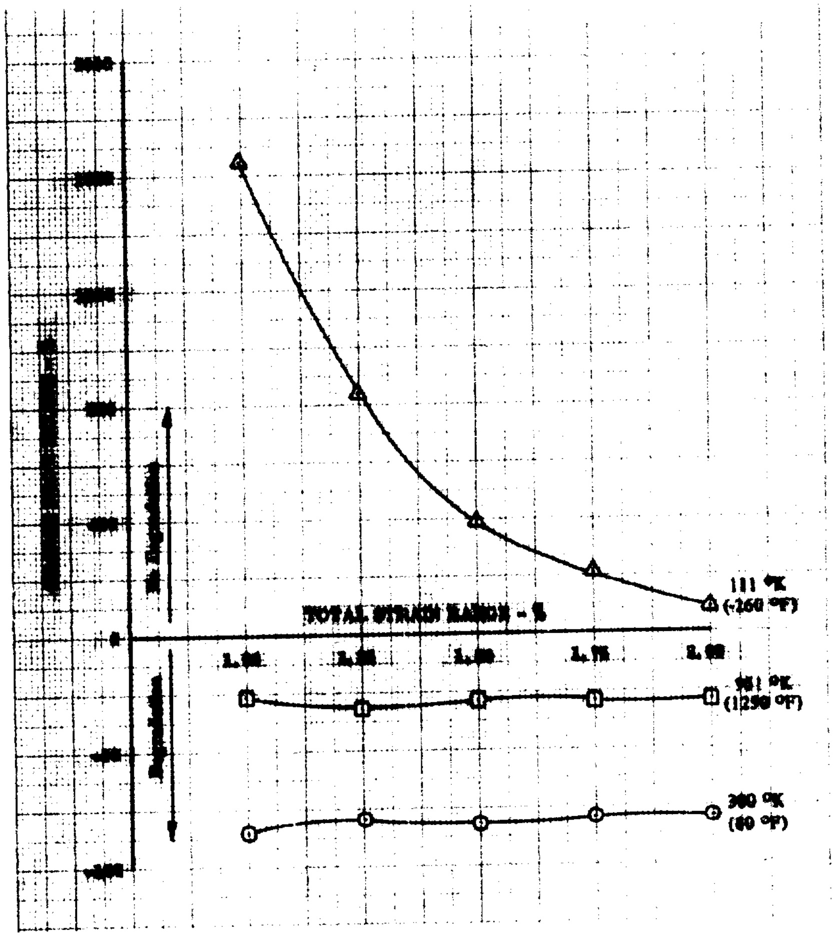


Figure V-8. Effect of Gaseous Hydrogen and Strain Range on Low-Cycle Fatigue Life of Inconel 718 With 1313°K (1900°F) Solution Plus Age Heat Treatment at 34.5 MN/m² (5000 psig) Pressure

DF 96407

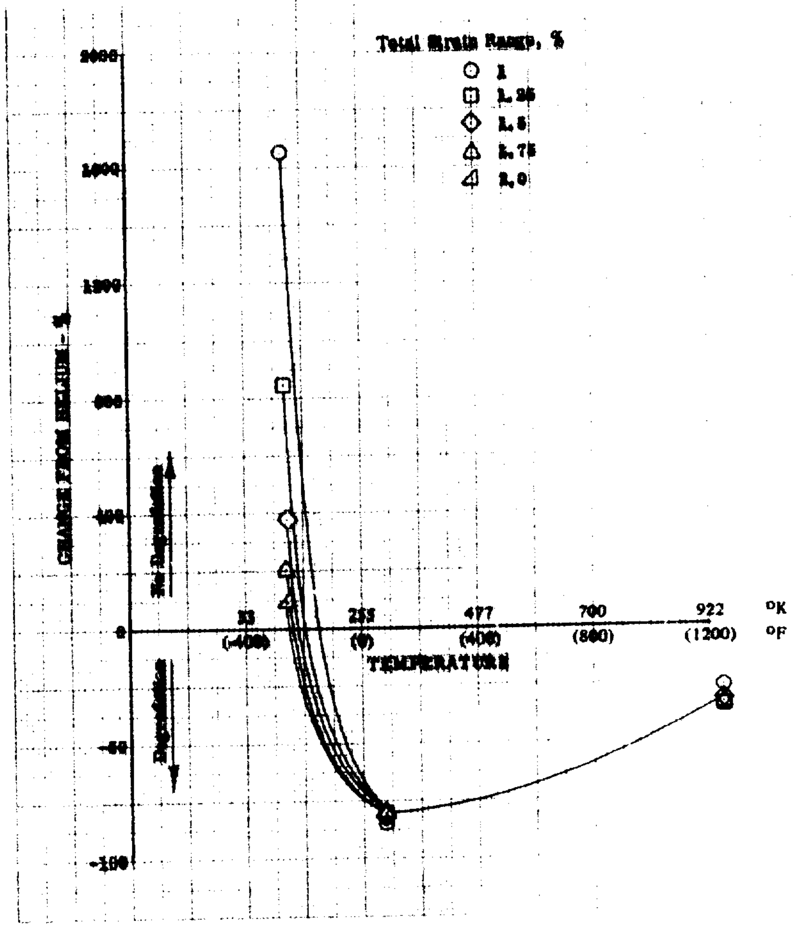


Figure V-9. Effect of Gaseous Hydrogen and Temperature on Low-Cycle Fatigue Life of Inconel 718 With 1313°K (1900°F) Solution Plus Age Heat Treatment at 34.5 MN/m² (5000 psig) Pressure

DF 96408

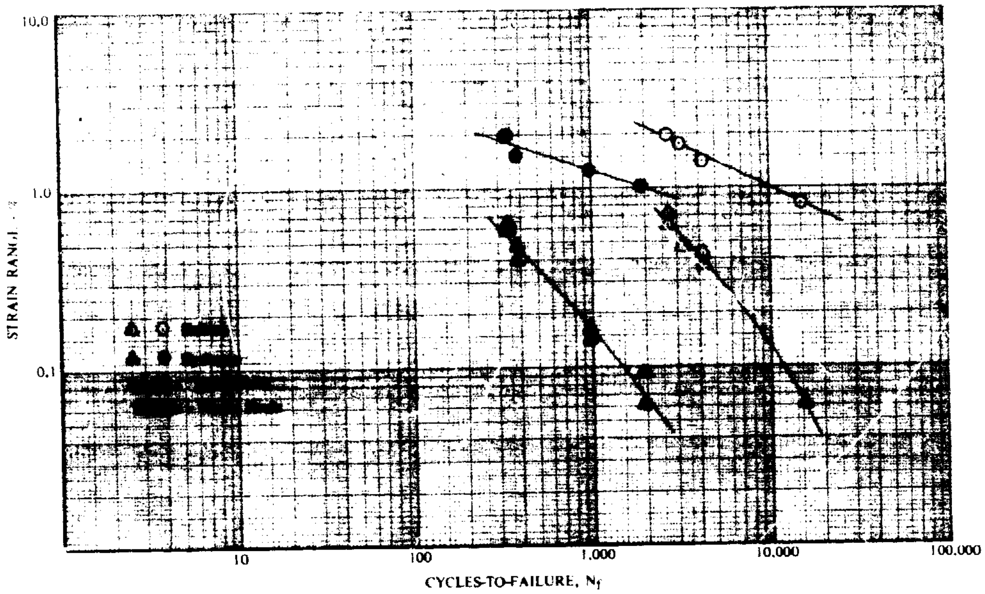


Figure V-10. Low-Cycle Fatigue Life of Inconel 625 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure

DF 96409

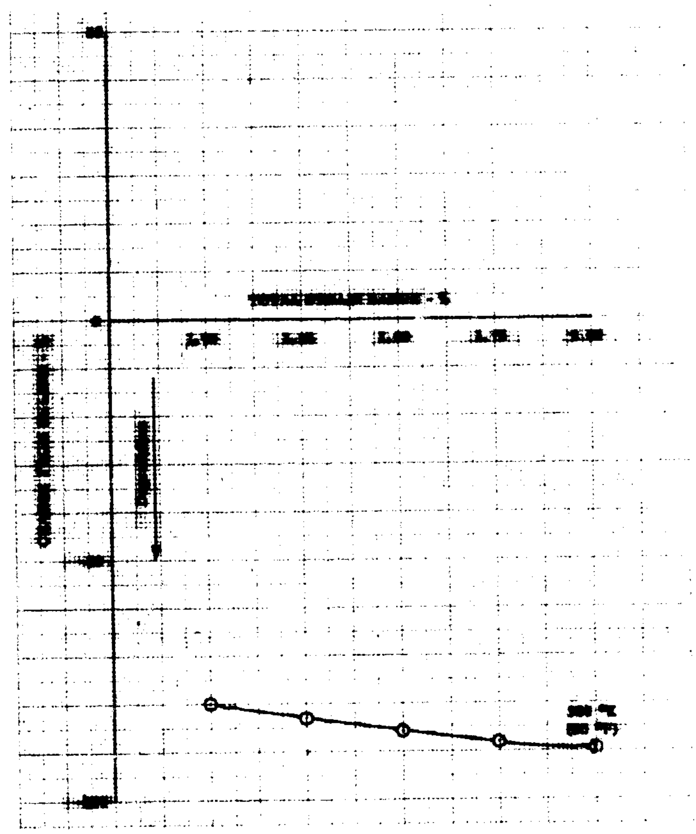


Figure V-11. Effect of Gaseous Hydrogen and Strain Range on Low-Cycle Fatigue Life of Inconel 625 at 34.5 MN/m^2 (5000 psig) Pressure

DF 96410

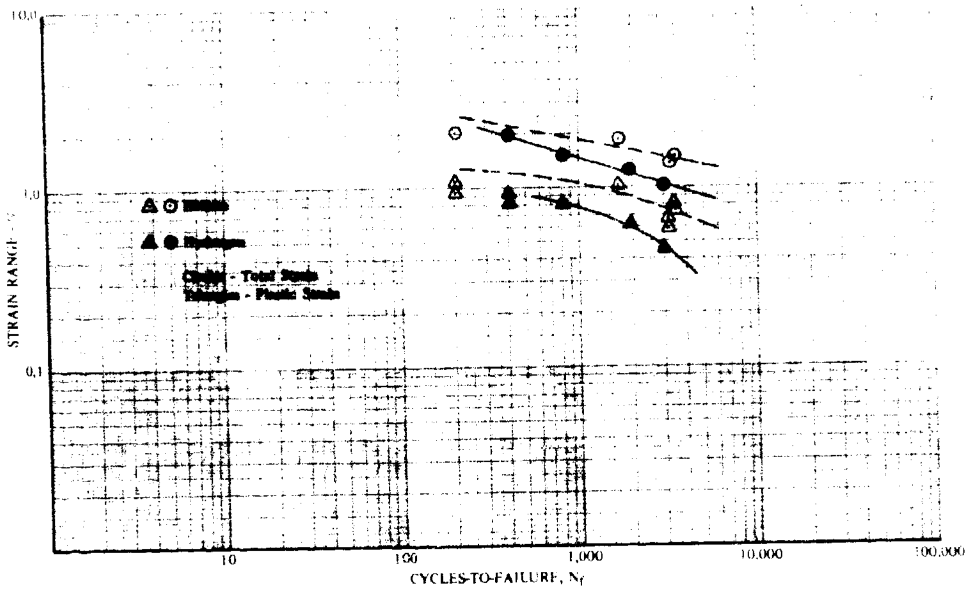


Figure V-12. Low-Cycle Fatigue Life of Hastelloy X at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96411

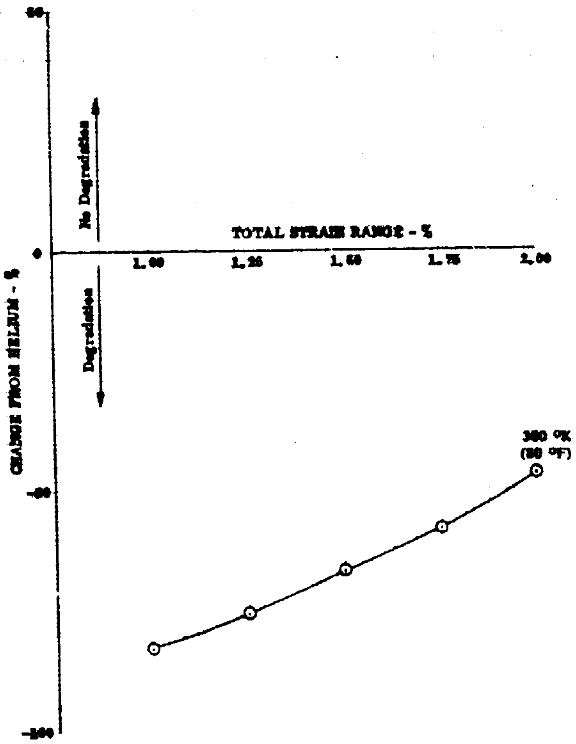


Figure V-13. Effect of Gaseous Hydrogen and Strain Range on Low-Cycle Fatigue Life of Hastelloy X at 34.5 MN/m^2 (5000 psig) Pressure — DF 96412

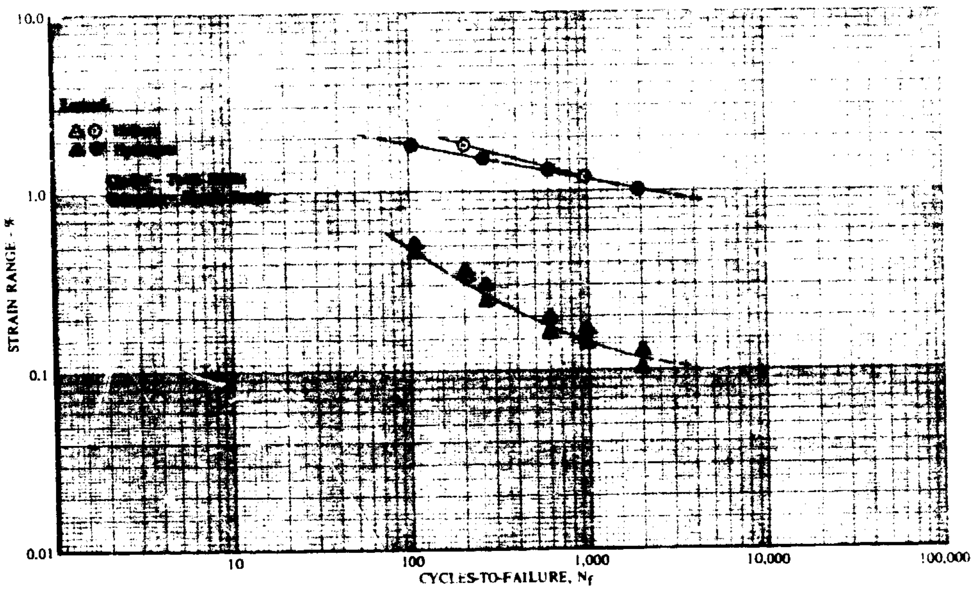


Figure V-14. Low-Cycle Fatigue Life of WASPALOY® at 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure

DF 96413

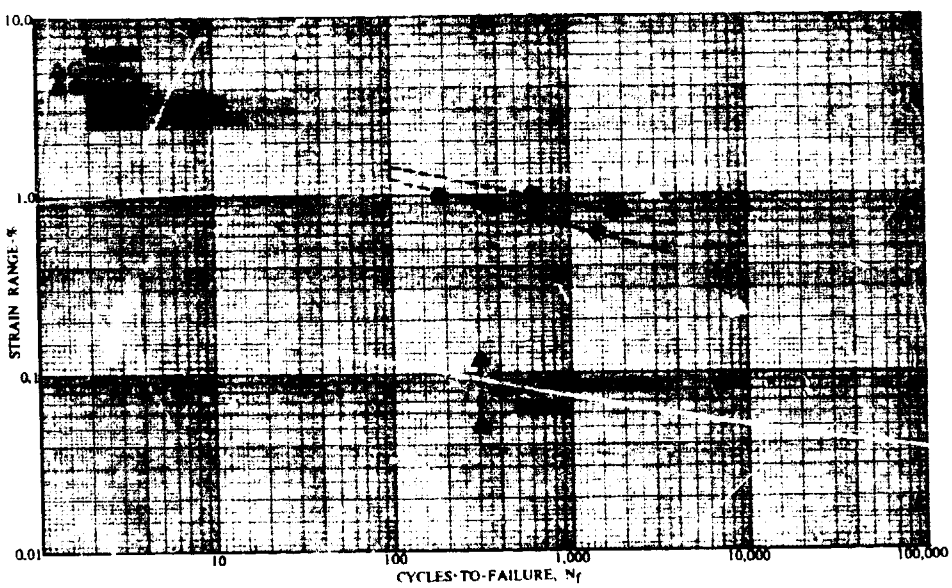


Figure V-15. Low-Cycle Fatigue Life of IN-100 at 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure

DF 96414

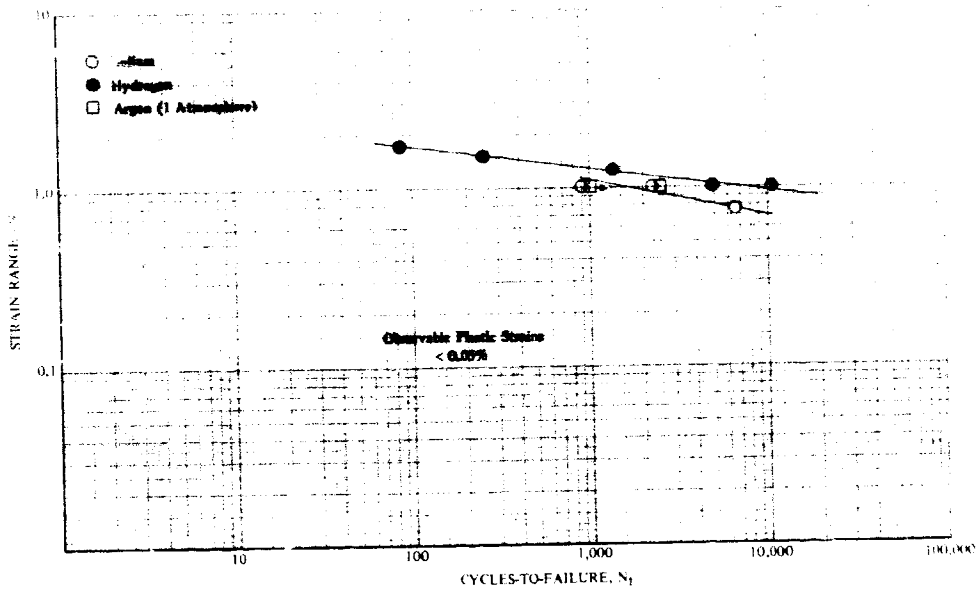


Figure V-16. Low-Cycle Fatigue Life of MAR M-200 DS at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure   DF 96415

The two titanium alloys (titanium 6-4 and A-110) were tested at room temperature and 366°K (200°F). (See figures V-17 through V-20.) There appeared to be little or no degradation of LCF life of titanium 6-4 at either temperature. The titanium A-110 did exhibit severe degradation at 366°K (200°F) with the degree of degradation dependent upon cyclic strain level. There appeared to be no degradation of A-110 at 300°K (80°F). There was, however, considerable data scatter for these alloys at room temperature.

The cobalt-base alloy, Haynes 188, was tested at 951°K (1250°F) only and did not display a marked influence of strain level upon degree of degradation (figure V-21), with severe degradation over the entire cyclic strain range of 1 to 2% at this temperature.

The two iron-base alloys, AISI 347 and A-286, exhibited the least degradation of all alloys tested. Neither alloy was degraded at room temperature 300°K (80°F), and A-286 was only slightly degraded at 951°K (1250°F). (See figures V-22 through V-24.)

Test results are listed in table V-1 for all alloys and conditions tested. Curves of strain range (both total and plastic, where applicable) vs cycles-to-failure are plotted for each material. Plots of percent change from helium vs cyclic strain range were obtained by comparing the helium and hydrogen LCF curves, which were extrapolated, if necessary.

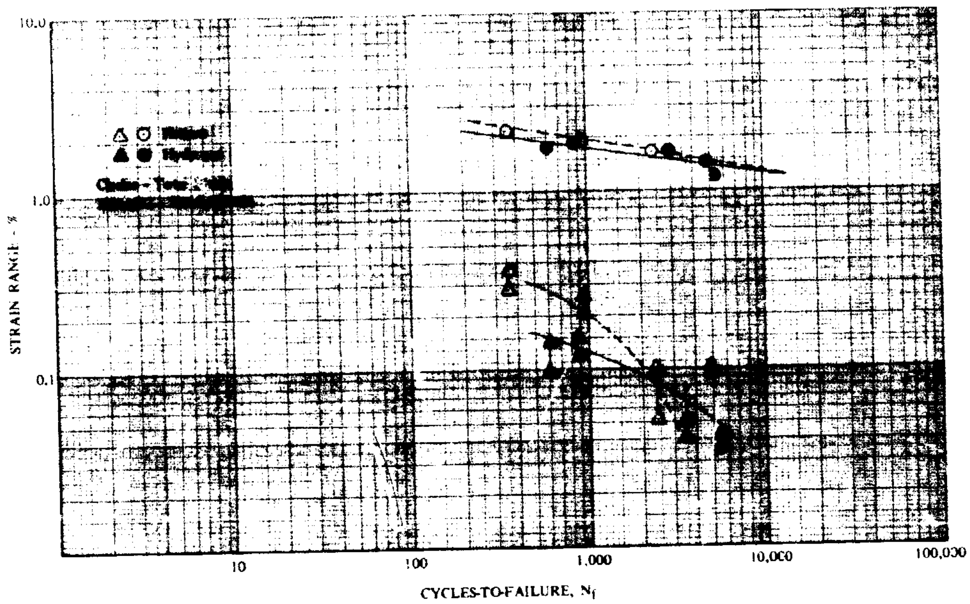


Figure V-17. Low-Cycle Fatigue Life of Titanium 6-4 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96416

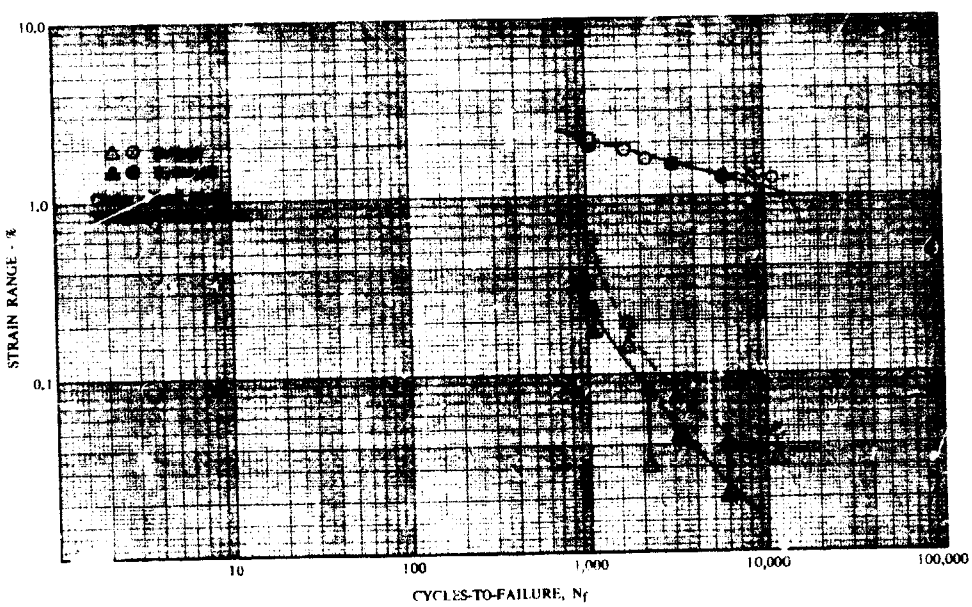


Figure V-18. Low-Cycle Fatigue Life of Titanium 6-4 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96417

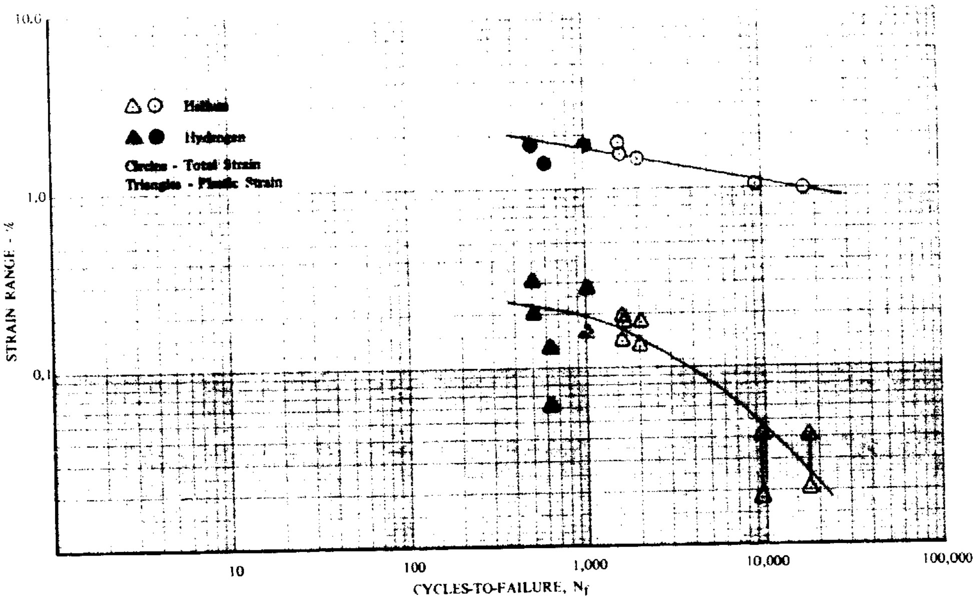


Figure V-19. Low-Cycle Fatigue Life of Titanium A-110 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96418

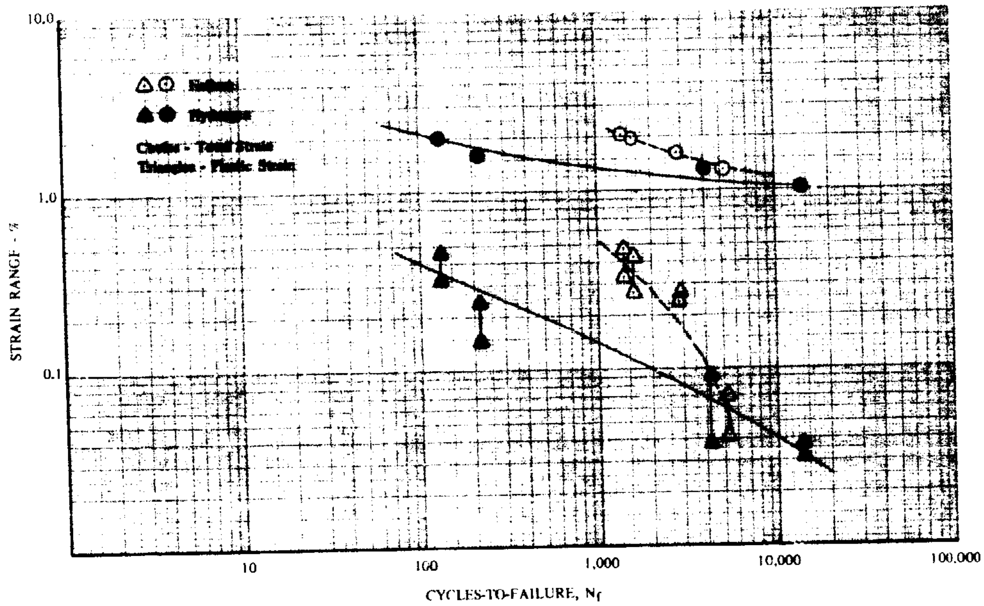


Figure V-20. Low-Cycle Fatigue Life of Titanium A-110 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96419

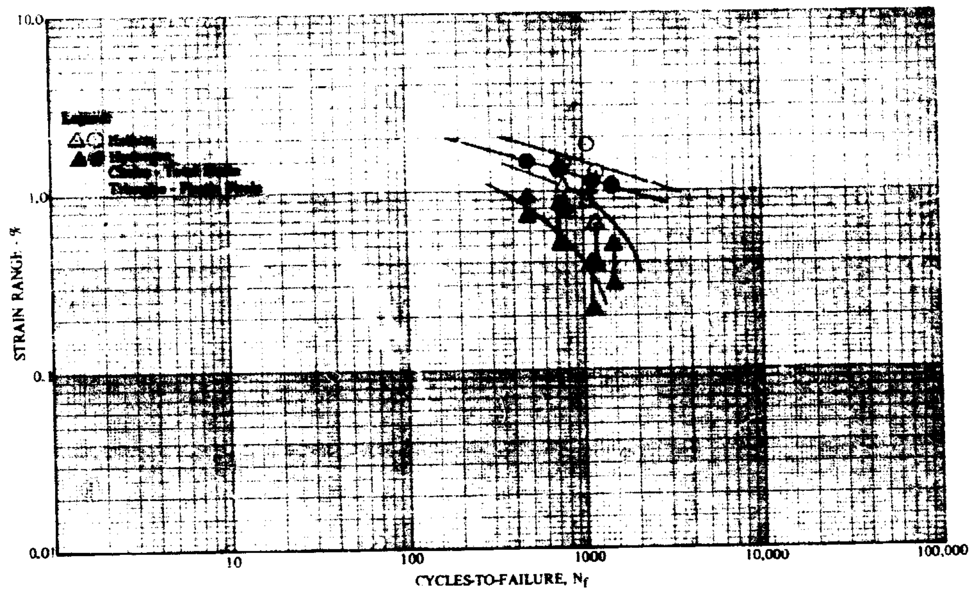


Figure V-21. Low-Cycle Fatigue Life of Haynes 188 at 951°K (1250°F) and 34. 5 MN/m^2 (5000 psig) Pressure

DF 96420

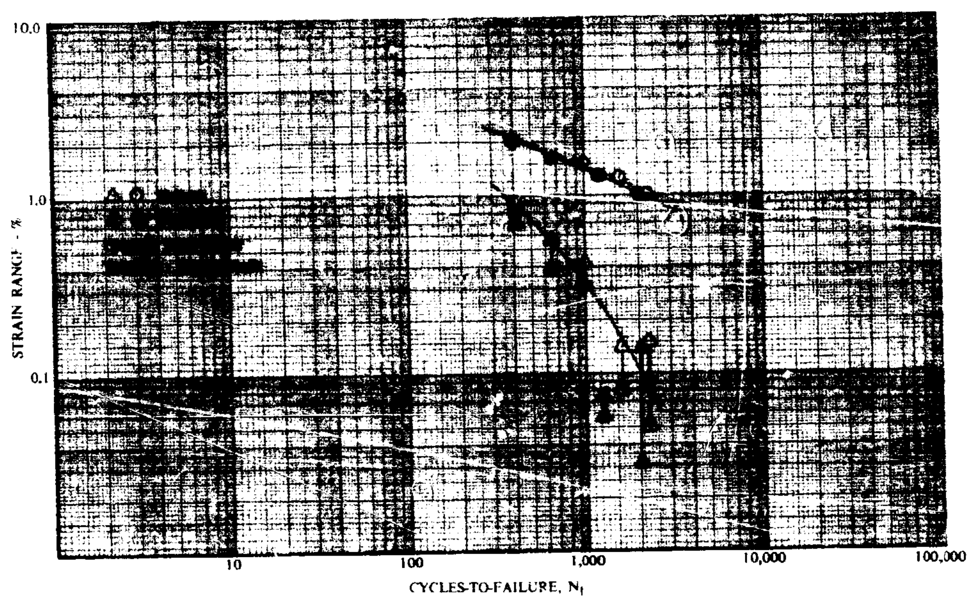


Figure V-22. Low-Cycle Fatigue Life of A-286 at 300°K (80°F) and 34. 5 MN/m^2 (5000 psig) Pressure

DF 96421

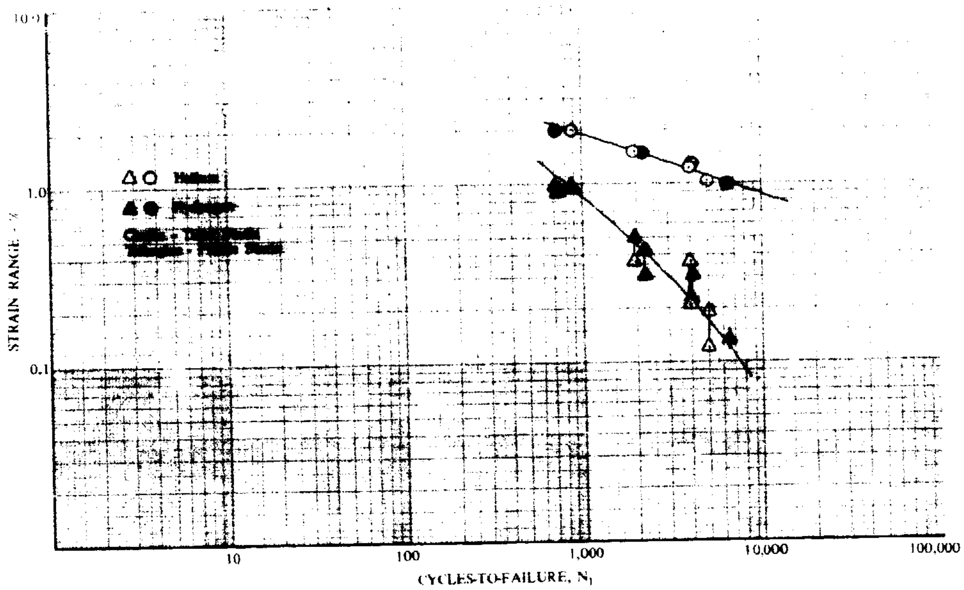


Figure V-23. Low-Cycle Fatigue Life of A-286 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure

DF 96422

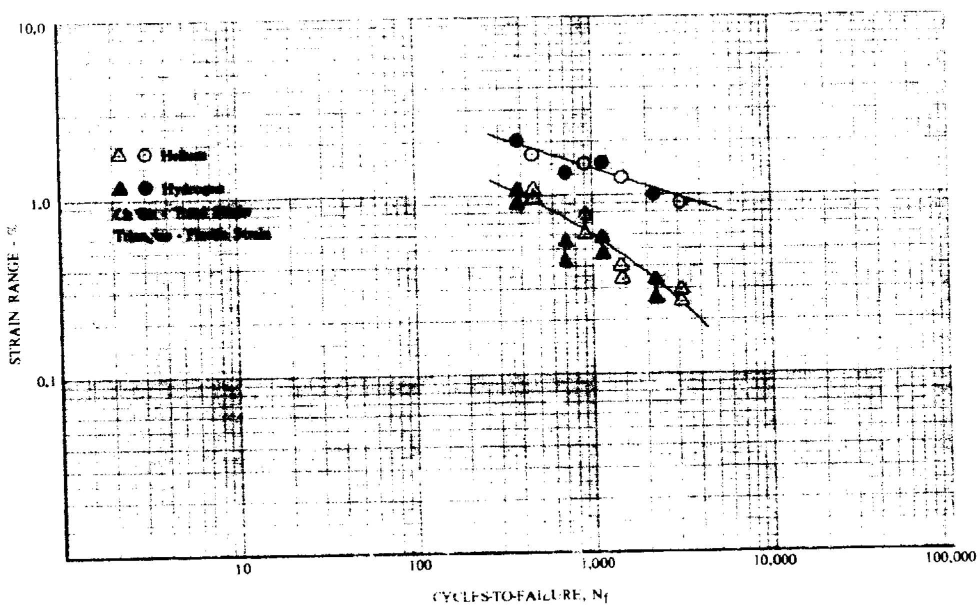


Figure V-24. Low-Cycle Fatigue Life of AISI 347 at 300°K (80°F) and 34.5 MN/m^2 (5000 psig) Pressure

DF 96423

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment

Material	Test Temperature, °K	Test Temperature, °F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, %*(1)	Heat Code
Nickel Base							
Inconel 718	300	80	Helium	1.0	15,000	0.02	BVTO
1227°K (1750°F)	300	80	Helium	0.8	13,650	0.03 to 0.5	
Solution + Age	300	80	Helium	1.5	1,500	0.13 to 0.20	
	300	80	Helium	2.0	1,220	0.35 to 0.54	
	300	80	Hydrogen	1.0	6,310	0.07 to 0.08	
	300	80	Hydrogen	1.0	6,080	0.05 to 0.08	
	300	80	Hydrogen	1.5	450	0.12 to 0.19	
	300	80	Hydrogen	2.0	55	0.41 to 0.46	
	300	80	Hydrogen	0.8	9,950	0.02 to 0.04	
	951	1250	Helium	2.0	145	0.59 to 0.72	
	951	1250	Helium	1.45	570	0.15 to 0.28	
	951	1250	Helium	1.25	940	0.04 to 0.16	
	951	1250	Helium	1.0	1,820	0.03 to 0.10	
	951	1250	Hydrogen	1.75	175	0.45 to 0.59	
	951	1250	Hydrogen	1.50	330	0.21 to 0.39	
	951	1250	Hydrogen	1.25	815	0.05 to 0.18	
	951	1250	Hydrogen	1.0	1,810	0.05	
Inconel 718	111	-260	Helium	1.5	5,140	0.06 to 0.14	BVTO
1313°K (1900°F)	111	-260	Helium	2.0	1,630	0.41 to 0.61	
Solution + Age	111	-260	Helium	2.0	2,080	0.45 to 0.52	
	111	-260	Helium	2.5	1,300	0.51 to 0.89	
	111	-260	Hydrogen	1.5	22,300	0.04 to 0.07	
	111	-260	Hydrogen	2.0	3,460	0.21 to 0.31	
	111	-260	Hydrogen	2.0	3,580	0.15 to 0.32	
	111	-260	Hydrogen	2.25	1,850	0.44 to 0.72	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, %*(1)	Heat Code
	300	80	Helium	1.0	21,500	0.02 to 0.05	
	300	80	Helium	1.25	5,450	0.13 to 0.16	
	300	80	Helium	1.5	2,360	0.11 to 0.18	
	300	80	Helium	2.1	900	0.60 to 0.70	
	300	80	Hydrogen	1.0	2,220	0.03	
	300	80	Hydrogen	1.3	760	0.04 to 0.09	
	300	80	Hydrogen	1.5	460	0.18 to 0.22	
	300	80	Hydrogen	2.0	300	0.33 to 0.49	
	951	1250	Helium	2.0	260	0.64 to 0.85	
	951	1250	Helium	1.6	510	0.43 to 0.52	
	951	1250	Helium	1.35	950	0.15 to 0.28	
	951	1250	Helium	1.0	1,730	0.11 to 0.22	
	951	1250	Hydrogen	1.75	320	0.50 to 0.67	
	951	1250	Hydrogen	1.6	285	0.33 to 0.48	
	951	1250	Hydrogen	1.3	685	0.22 to 0.39	
	951	1250	Hydrogen	1.0	1,350	0.10 to 0.17	
Inconel 625	300	80	Helium	0.8	15,200	0.06	BZCV
	300	80	Helium	1.4	4,160	0.36 to 0.42	
	300	80	Helium	1.75	3,200	0.46	
	300	80	Helium	2.0	2,730	0.62 to 0.72	
	300	80	Hydrogen	1.0	1,890	0.06 to 0.09	
	300	80	Hydrogen	1.25	970	0.14 to 0.16	
	300	80	Hydrogen	1.5	360	0.39 to 0.46	
	300	80	Hydrogen	2.0	340	0.58 to 0.62	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m² (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, % *(1)	Heat Code
Hastelloy X	300	80	Helium	1.5	3,500	0.73 to 0.79	BZCR
	300	80	Helium	1.4	3,300	0.58 to 0.66	
	300	80	Helium	1.85	1,720	1.02 to 1.08	
	300	80	Helium	2.0	200	0.91 to 1.08	
	300	80	Hydrogen	1.0	3,050	0.44 to 0.46	
	300	80	Hydrogen	1.25	1,964	0.61 to 0.62	
	300	80	Hydrogen	1.5	810	0.79 to 0.82	
	300	80	Hydrogen	2.0	405	0.82 to 0.92	
WASPALOY®	951	1250	Helium	1.8	202	0.34 to 0.36	L1288K13
	951	1250	Helium	1.2	980	0.14 to 0.17	
	951	1250	Hydrogen	1.8	105	0.45 to 0.50	
	951	1250	Hydrogen	1.5	260	0.24 to 0.30	
	951	1250	Hydrogen	1.3	600	0.16 to 0.20	
	951	1250	Hydrogen	1.0	1,950	0.10 to 0.13	
IN-100	951	1250	Helium	1.0	630	< 0.05	P-9245
	951	1250	Helium	0.8	1,820	< 0.05	
	951	1250	Hydrogen	1.0	190	< 0.05	
	951	1250	Hydrogen	0.9	315	< 0.05	
	951	1250	Hydrogen	0.8	650	< 0.05	
	951	1250	Hydrogen	0.6	1,450	< 0.05	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	°F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, %*(1)	Heat Code
MAR M-200 DS	951	1250	Helium	1.00	900	<0.05	P-9108
	951	1250	Helium	1.00	2,250	<0.05	
	951	1250	Helium	0.75	6,600	<0.05	
	951	1250	Hydrogen	1.70	85	<0.05	
	951	1250	Hydrogen	1.50	250	<0.05	
	951	1250	Hydrogen	1.25	1,330	<0.05	
	951	1250	Hydrogen	1.00	10,640	<0.05	
	951	1250	Hydrogen	1.00	4,950	<0.05	
(2)	951	1250	Argon	1.00	>1,000	<0.05	
	951	1250	Argon	1.00	2,500	<0.05	
Iron Base							
A-286	300	80	Helium	1.0	4,930	0.12 to 0.19	BZCU
	300	80	Helium	1.25	4,012	0.20 to 0.37	
	300	80	Helium	1.5	1,950	0.36 to 0.50	
	300	80	Helium	2.0	870	0.93 to 0.98	
	300	80	Hydrogen	1.0	6,550	0.12	
	300	80	Hydrogen	1.3	4,030	0.22 to 0.31	
	300	80	Hydrogen	1.5	2,200	0.30 to 0.42	
	300	80	Hydrogen	2.0	700	0.90 to 0.95	
	951	1250	Helium	2.0	405	0.82 to 0.89	
	951	1250	Helium	1.5	1,000	0.31 to 0.42	
	951	1250	Helium	1.25	1,600	0.08 to 0.14	
	951	1250	Helium	1.0	2,290	0.05 to 0.15	
	951	1250	Hydrogen	2.0	410	0.70 to 0.89	
	951	1250	Hydrogen	1.6	650	0.39 to 0.57	
	951	1250	Hydrogen	1.25	1,260	0.57 to 0.06	
	951	1250	Hydrogen	1.0	2,050	0.03 to 0.14	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	°F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, % (1)	Heat Code
AISI 347	300	80	Helium	0.9	3,100	0.25 to 0.29	BZCT
	300	80	Helium	1.25	1,450	0.33 to 0.39	
	300	80	Helium	1.5	905	0.60 to 0.77	
	300	80	Helium	1.7	460	0.96 to 1.05	
	300	80	Hydrogen	1.0	2,200	0.26 to 0.33	
	300	80	Hydrogen	1.5	1,120	0.47 to 0.58	
	300	80	Hydrogen	1.3	700	0.42 to 0.54	
	300	80	Hydrogen	2.0	380	0.90 to 1.10	
Titanium Base Titanium 6-4	300	80	Helium	1.6	2,370	0.05 to 0.10	BZCS
	300	80	Helium	1.9	950	0.21 to 0.26	
	300	80	Helium	2.25	360	0.28 to 0.37	
	300	80	Hydrogen	1.4	4,850	0.09 to 0.10	
	300	80	Hydrogen	1.7	600	0.09 to 0.10	
	300	80	Hydrogen	1.7	2,940	0.04 to 0.05	
	300	80	Hydrogen	1.85	860	0.12 to 0.15	
	300	80	Hydrogen	1.2	5,130	0.03 to 0.04	
	366	200	Helium	1.25	11,270	0.03 to 0.05	
	366	200	Helium	1.6	2,130	0.03 to 0.08	
	366	200	Helium	1.8	1,650	0.14 to 0.19	
	366	200	Helium	2.15	1,050	0.45 to 0.56	
	366	200	Hydrogen	1.25	5,880	0.02 to 0.04	
	366	200	Hydrogen	1.5	3,020	0.04	
	366	200	Hydrogen	1.9	1,050	0.17 to 0.25	
	366	200	Hydrogen	2.1	970	0.24 to 0.32	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, % (1)	Heat Code
Titanium A-110	300	80	Helium	1.8	1,600	0.14 to 0.19	BZCW
	300	80	Helium	1.6	1,620	0.18	
	300	80	Helium	1.5	2,000	0.13 to 0.18	
	300	80	Hydrogen	1.0	9,050	0.015 to 0.04	
	300	80	Hydrogen	1.4	610	0.06 to 0.13	
	300	80	Hydrogen	1.85	500	0.20 to 0.31	
	300	80	Hydrogen	1.8	1,020	0.16 to 0.27	
	300	80	Hydrogen	1.0	17,500	0.02 to 0.04	
	366	200	Helium	1.25	5,300	0.04 to 0.07	
	366	200	Helium	1.6	2,900	0.23 to 0.26	
	366	200	Helium	1.9	1,620	0.26 to 0.42	
	366	200	Helium	2.05	1,400	0.32 to 0.46	
	366	200	Hydrogen	1.0	14,200	0.03 to 0.036	
	366	200	Hydrogen	1.3	4,100	0.035 to 0.09	
	366	200	Hydrogen	1.6	215	0.14 to 0.24	
	366	200	Hydrogen	2.0	130	0.32 to 0.45	

Table V-1. Low-Cycle Fatigue Properties in 34.5-MN/m^2 (5000-psig) Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Environment	Total Strain Range, %	Cycles-to-Failure	Plastic Strain Range, %(1)	Heat Code
Cobalt Base							
Haynes 188	951	1250	Helium	1.8	1,050	0.88 to 1.00	YFYR
	951	1250	Helium	1.25	1,151	0.33 to 0.65	
	951	1250	Helium	1.45	765	0.79 to 1.09	
	951	1250	Hydrogen	1.45	470	0.71 to 0.95	
	951	1250	Hydrogen	1.3	720	0.50 to 0.84	
	951	1250	Hydrogen	1.15	1,106	0.22 to 0.41	
	951	1250	Hydrogen	1.1	1,423	0.31 to 0.53	

(1) Includes Strain Hardening-Softening Effects

(2) Additional Tests Conducted at Atmospheric Pressure for Comparison Purposes.

Cyclic Rates for Tests Were: Less Than 1.0% Total Strain Range - 5 Cycles/min
1.0% to <1.5% Total Strain Range - 4 Cycles/min
1.5% and Higher Total Strain Range - 3 Cycles/min

C. TEST PROCEDURES

Smooth, round, solid specimens were used for the strain-controlled LCF tests conducted under this contract. The test specimen used is described in Section III and detailed in figure III-3. The specimen configuration incorporated integral machined extensometer collars. A calibration procedure was established for each material to relate the maximum strain-to-collar-deflection during both the elastic and plastic portion of the strain cycle. The specimen design and calibration procedure were verified both experimentally and analytically.

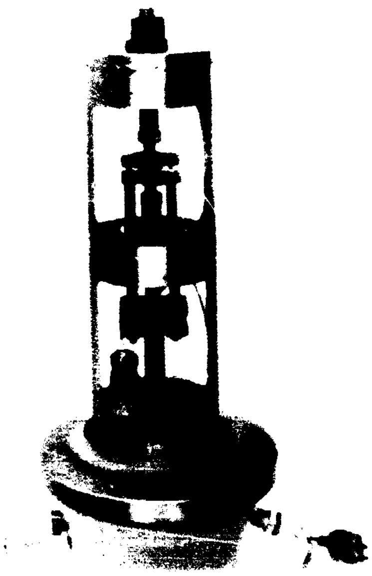
After machining, specimen gage sections were polished and dimensions measured. Prior to installation in the test rig, specimens were cleaned with a nonchlorinated solvent.

Tests were conducted on a P&WA™-designed and built, closed-loop-type, hydraulically actuated test machine, located in an isolated test cell, utilizing the strain control mode. Specimen axial strain was measured and controlled by means of a proximity probe extensometer. A heavy walled pressure vessel made of AISI-type 347 stainless steel was mounted on the upper platen of the test machine. This vessel (shown in figure 25) incorporated a Grayloc-type flange and seal because of the relative ease of assembly and the reliability of the seal in high pressure. The base of the vessel included a pressure-compensating device to eliminate the axial tensile load acting over the differential specimen and adapter areas. Both internal (to the pressure vessel) and external load cells were used; thus the effect of friction at the seals, where the load rods enter the vessel, was known and accounted for. During testing, load strain hysteresis curves were plotted using the extensometer and internal load cell outputs. 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. Cryogenic temperatures were obtained by surrounding the test chamber with a liquid nitrogen bath. The test gas passed through a heat exchanger coil submersed in the liquid nitrogen bath and into the test chamber. Thermocouples attached to the specimen were used to monitor temperature during test.

Elevated temperatures were obtained with a resistance furnace surrounding the specimen internal to the pressure vessel. This furnace is also shown in figure V-25. A dc power supply and controller were used to drive the furnace. Thermocouples attached to the specimen were used to monitor and control temperature during test. Because of the short specimen gage section, a single-zone furnace was adequate to maintain a uniform gage section temperature. The high thermal conductivities of high-pressure helium and hydrogen gases enabled the load cell and strain measuring transducer of the extensometer to operate at safe temperatures because of their location in the bottom of the vessel.

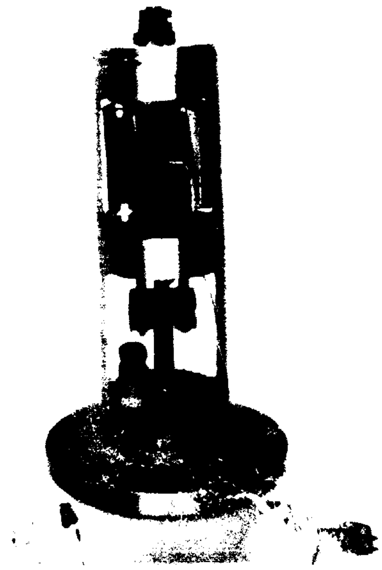
After inserting the prepared specimen in the test vessel and attaching the extensometer, the vessel was sealed and subjected to a purge cycle consisting of a nitrogen purge, evacuation, test gas (helium or hydrogen) purge, and finally a pressurized test gas pop purge. The pop purge consisted of rapidly pressurizing the vessel to a low pressure and releasing while maintaining a minimum positive gas pressure. The pop purge was found to result in significant increases in the purity of the gaseous environment in the vessel over that obtained by normal flow purging. In fact, analysis of gas samples taken during rig checkout early in the program indicated that the pop purge was as effective in reducing gaseous environment contamination as evacuating the test vessel prior to normal purging. For this testing, however, both an evacuation and pop purge were used.

After purging, high-pressure gas was introduced and maintained in the vessel until specimen temperature and gas pressure were stabilized at the desired level and testing conducted. The test machine control provided automatic system shutdown upon specimen fracture. Test gas was then vented (and vessel purged with nitrogen in the case of hydrogen tests), vessel opened, and specimen removed. The test vessel and test procedure used for the low-cycle fatigue testing were similar to those used for all types of testing under this contract and are also discussed in the sections of this report dealing with tensile, fracture mechanics, high-cycle fatigue, and creep-rupture.



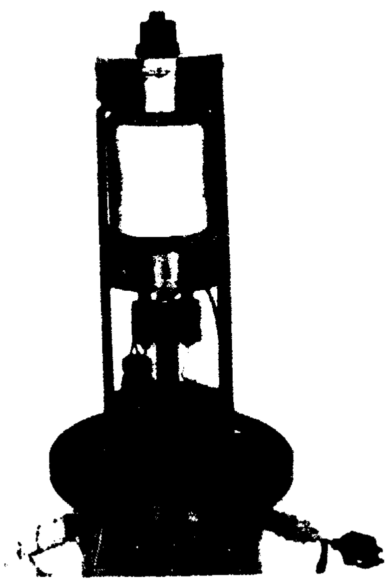
FE 100028

Specimen and Extensometer in Place



FE 100027

Specimen, Extensometer and Half Furnace in Place



FE 100026

Specimen, Extensometer and Furnace in Place

FE 100025

Closed Pressure Vessel, Cooling Jacket Not Shown

Figure V-25. High Pressure Gaseous Environment, Low-Cycle Fatigue Test Vessel FD 53147

SECTION VI
FRACTURE MECHANICS TESTING

SECTION VI
FRACTURE MECHANICS TESTING

A. INTRODUCTION

Fracture mechanics tests were conducted in 34.5-MN/m^2 (5000-psig) hydrogen and helium on three nickel-, two iron-, and two titanium-base wrought alloys at a temperature of 300°K (80°F). The materials tested included Inconel 718, Inconel 625, WASPALOY®, A-286, AISI 347, Titanium 6-4, and Titanium A-110. Two of the nickel-base alloys, Inconel 718 and Inconel 625, were also tested in the welded condition.

The fracture mechanics tests used two types of specimens. Materials evaluated in the first year of this program to determine fracture toughness were tested using the ASTM compact tensile specimen. The WASPALOY tests of the second year were conducted using a center notch (flaw) type specimen. The compact tensile specimen supplied fracture toughness information, either K_{IC} or K_{IE} for precracks parallel to grain flow (longitudinal) and transverse to the grain flow. The center slot specimen supplied fracture toughness K_{IE}, threshold stress intensity, K_{TH}, and cyclic stress intensity, K_I/K_{IC}, information for WASPALOY.

B. CONCLUSIONS AND DISCUSSION

Only one alloy, WASPALOY, indicated a significant effect due to the hydrogen environment. Fracture toughness values of 63 to 74 MN/m^2 $\sqrt{m}$ (57 to 67 ksi $\sqrt{in.}$) occurred in hydrogen compared to 103 MN/m^2 $\sqrt{m}$ (93 ksi $\sqrt{in.}$) in helium. This material was the only one tested using the center flaw specimen. The other alloys were tested using the ASTM compact tensile specimen, and there was no severe hydrogen degradation in any of the materials tested. Only slight sensitivity was evident in Inconel 718, as indicated by slightly lower fracture toughness values and the smoother fracture face in hydrogen than in helium.

Although there was no evidence of hydrogen degradation in fracture toughness of Titanium 6-4 and Inconel 625, the metallographic examination indicated some sensitivity to hydrogen in both materials. In fact, from the metallographic examination, it was concluded that Inconel 625 was the most severely hydrogen-embrittled alloy. This was not consistent with the compact tension fracture toughness values obtained, as, if anything, they showed a slight increase in hydrogen. An R_{SC} analysis, discussed on the next page, does tend to show hydrogen degradation.

A comparison of welded Inconel 718 and Inconel 625 to parent material revealed a general decrease in magnitude of the fracture toughness values, but no susceptibility to hydrogen degradation. GTA-welded Inconel 718 with the 1313°K (1900°F) solution was less ductile than the parent material, as was evidenced by the appearance of the fracture face and the attained K_{IE} values. This was not the case with Inconel 625, in that it remained very ductile and retained approximately the same K_{IE} values. Comparing longitudinal to transverse fracture toughness values, there was generally no difference, although there was a slight increase in magnitude in the transverse direction in both titanium materials tested. There was a significant increase in R_{SC} of welded Inconel 625 over the parent material. This increase is mostly due to the decrease in yield strength. This increased R_{SC} indicates the welded Inconel 625 is significantly tougher than parent material.

Cyclic flaw growth data and sustained load flaw growth data for WASPALOY center flaw specimens are presented in figures VI-1 and VI-2, respectively. An initial stress intensity value of approximately 27.9 $MN/m^2 \sqrt{m}$ (25 ksi $\sqrt{in.}$) would be required for 100 cycle life in 34.5-MN/m^2 (5000-psig) hydrogen at 300°K (80°F). This is a ratio of initial stress intensity to critical stress intensity (K_I/K_C) of approximately 0.45. Sustained-load threshold stress intensity, K_{TH}, of approximately 33 $MN/m^2 \sqrt{m}$ (30 ksi $\sqrt{in.}$) would yield no flaw growth in 100 hr at 300°K (80°F) and 34.5-MN/m^2 (5000-psig) hydrogen pressure.

Fracture toughness values are reported in table VI-1 for all materials (except WASPALOY) that were tested using the ASTM compact tensile specimen. Originally, the tests were conducted per ASTM E-399-70T, and results were reported accordingly in progress reports. These data have been re-evaluated according to ASTM E-399-72, and most K_{IC} values downgraded to K_{IE} (an engineering estimate of K_{IC}) since they did not meet the more stringent requirements. The new specification adds the evaluation factor, R_{SC}, which is the ratio of stress at the crack tip to yield stress.

In the analysis and reporting of fracture toughness test results, fracture appearance is valuable supplementary information. The present ASTM standard for fracture toughness testing assumes that all crack front constraint, due to the free surfaces, will be evidenced by the formation of shear lips (illustrated in figure VI-3 as type A). This is not always the case, as was found in very ductile materials, such as Inconel 625 and AISI 347. In these cases, there was such a large amount of plastic flow constraining the crack front along the free surfaces that the result was only a reduction in thickness of the specimen (type B), instead of the formation of shear lips. Therefore, reporting the fracture-appearance in terms of percent oblique (implying the percentage of shear lip) is misleading when dealing with very ductile materials.

Following testing, metallographic investigations of the fracture characteristics of the materials were conducted. It was reported that rough fibrous, transgranular fractures indicated a tough material, whereas brittle failures were characterized by intergranular and cleavage fractures. Investigations also disclosed that the material orientation was quite evident in all of the specimens except the welds. The fibrous appearance of the fracture was oriented parallel to the longitudinal direction (grains parallel to direction of forging) of the material.

C. TEST PROCEDURE

Two different specimen configurations were used; compact tensile to determine fracture toughness values and center slot (notch) to determine fracture toughness values, threshold stress intensity, and cyclic stress intensity.

For the compact tensile specimens, longitudinal and transverse blanks were cut from round bar stock, heat treated, and machined per ASTM specifications for compact tensile specimens. The specimens were 19.05 mm (0.750 in.) thick and are detailed in Section III, figure III-5.

For the center slot (notch) specimens, blanks were cut from rectangular bar stock, heat treated, and machined into center-flawed specimens. The specimens were 2.45 mm (0.100 in.) thick in the gage section, and are detailed in Section III, figure III-6.

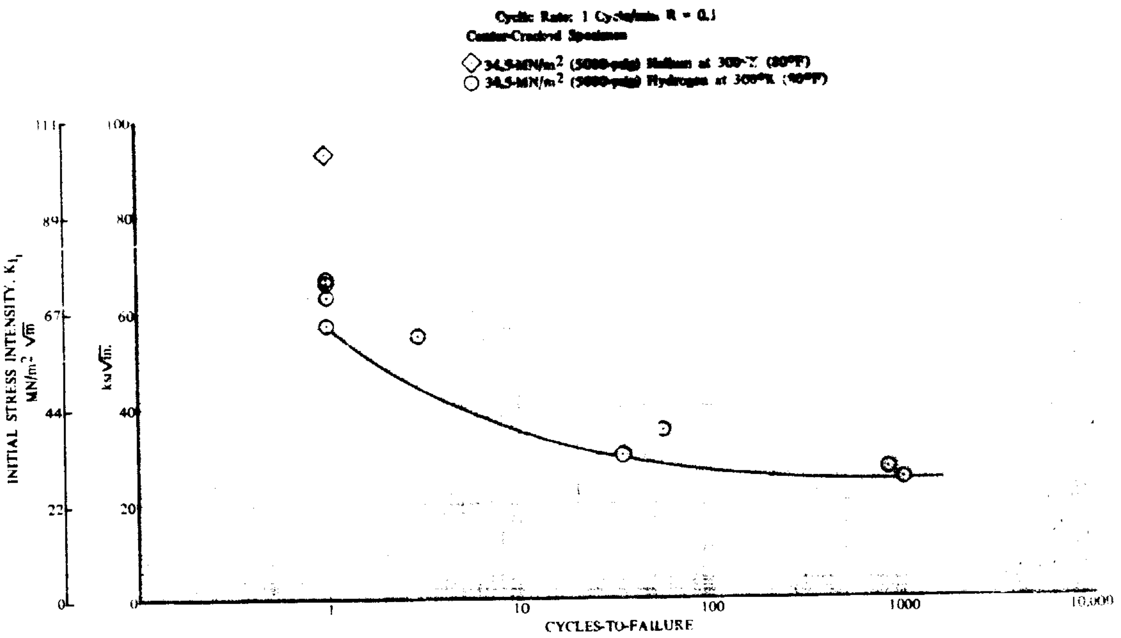


Figure VI-1. Cyclic Flaw Growth (Cyclic Stress Intensity) Data for WASPALOY® DF 91090

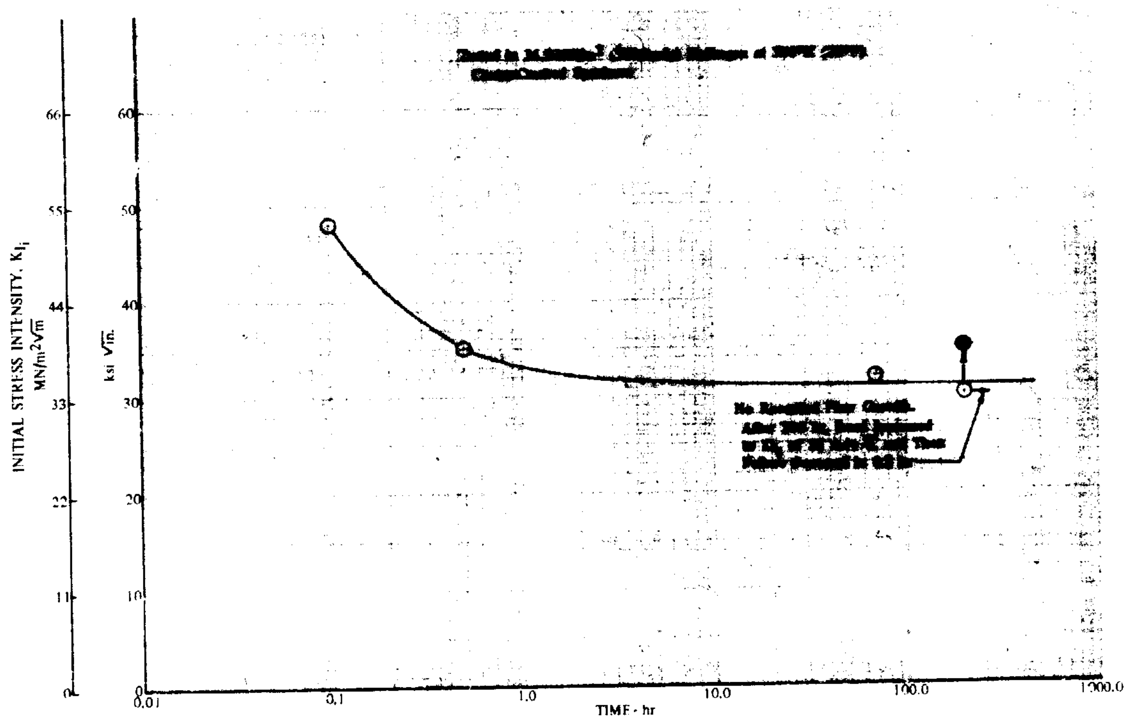


Figure VI-2. Sustained-Load Flaw Growth Data for WASPALOY® DF 91091

Table VI-1. Fracture Toughness of Materials in 34.5 MN/m^2 (5000-psig) Gaseous Environment at 300°K (80°F)

Material	Test Conditions: Notch Orientation	Test Conditions: Environment	Specimen Parameters: Thickness "B" mm	Thickness "B" in.	Depth "W" mm	Depth "W" in.	Crack Length mm	Crack Length in.	Fracture Appearance (% Oblique)	$2.5\left(\frac{K_Q}{\sigma_{YS}}\right)^2$	Test Results: Fracture Toughness K_{IF} (1) $MN/m^2\sqrt{m}$	K_{IF} (1) ksi $\sqrt{in.}$	K_{IC} $MN/m^2\sqrt{m}$	K_{IC} ksi $\sqrt{in.}$	R_{SC}	Heat Code
Wrought Nickel Base																
Inconel 718 1227°K (1750°F) Solution + Age	Long	Helium	19.050	0.750	38.125	1.501	21.640	0.852	3	0.686	93.2	84.0			1.02	BZMK
	Trans	Helium	18.920	0.745	38.150	1.502	19.940	0.785	3	0.881	106.0	95.7			1.08	
	Long	Hydrogen	19.075	0.751	38.100	1.500	21.720	0.855	3	0.613	84.5	76.2			1.09	
	Trans	Hydrogen	18.800	0.740	38.000	1.496	21.539	0.845	3	0.620	85.1	76.7			1.19	
Inconel 718 1313°K (1900°F) Solution + Age	Long	Helium	18.974	0.747	38.125	1.501	21.967	0.865	4	1.270	123.8	111.5			1.42	BZMK
	Trans	Helium	19.075	0.751	38.100	1.500	22.047	0.868	6	1.181	119.4	107.6			1.44	
	Long	Hydrogen	19.050	0.750	38.100	1.500	21.641	0.852		1.029	112.1	101.0			1.39	
	Trans	Hydrogen	18.999	0.748	38.100	1.500	22.149	0.872	5	0.916	105.7	95.2			1.49	
Welded Inconel 718 1313°K (1900°F) Solution + Age	Long	Helium	19.100	0.752	38.175	1.503	21.640	0.852	3	0.660	96.3	86.8			1.02	BZMK
	Trans	Helium	19.125	0.753	38.075	1.499	22.987	0.905	2	0.503	81.9	73.8			1.02	
	Long	Hydrogen	18.999	0.748	38.050	1.498	22.047	0.868	2	0.550	84.1	76.0			1.07	
	Trans	Hydrogen	19.075	0.751	38.175	1.503	22.479	0.885	3	0.531	82.8	74.6			0.99	
Inconel 625	Long	Helium	19.075	0.751	38.325	1.509	22.047	0.868	15	0.954	59.2	53.3			1.99	BYAP
	Trans	Helium	19.025	0.749	38.200	1.504	24.765	0.975	14	1.142	62.8	56.6			(2)	
	Long	Hydrogen	18.999	0.748	38.100	1.500	22.479	0.885	10	0.888	66.3	59.7			1.47	
	Trans	Hydrogen	18.974	0.747	38.100	1.500	21.895	0.862	8	0.731	60.2	54.2			1.41	
Welded Inconel 625	Long	Helium	19.050	0.750	38.150	1.502	23.165	0.912	16	2.260	56.6	51.0			(2)	BYAP
	Trans	Helium	19.100	0.752	38.175	1.503	22.657	0.892	15	2.180	55.3	49.8			3.25	
	Long	Hydrogen	19.025	0.749	38.200	1.504	23.165	0.912	13	2.730	61.1	55.0			3.46	
	Trans	Hydrogen	19.075	0.751	38.200	1.504	24.333	0.958	12	2.560	59.3	53.4			3.51	
Wrought Iron Base																
A-286	Long	Helium	19.100	0.752	38.225	1.505	21.894	0.862	6	1.740	99.1	89.3			1.82	BXGY
	Trans	Helium	19.025	0.749	38.050	1.498	22.555	0.888	5	2.000	106.1	95.6			2.17	
	Long	Hydrogen	19.025	0.749	38.100	1.500	21.539	0.848	4	1.485	94.7	85.3			1.73	
	Trans	Hydrogen	19.025	0.749	37.800	1.488	22.301	0.878	4	1.403	92.0	82.9			2.01	
AISI 347	Long	Helium	19.025	0.749	38.075	1.499	22.149	0.872	12	0.925	45.2	40.7			3.95	BUJH
	Trans	Helium	19.050	0.750	38.100	1.500	23.495	0.925	13	0.582	35.7	32.2			4.30	
	Long	Hydrogen	19.050	0.750	38.025	1.497	22.911	0.902	9	0.559	34.6	31.2			3.86	
	Trans	Hydrogen	18.974	0.747	38.050	1.498	22.047	0.868	10	0.815	41.9	37.7			3.89	
Wrought Titanium Base																
Titanium 6-4	Long	Helium	18.847	0.742	38.125	1.501	20.371	0.802	16	0.368			62.2	56.0	0.74	BZAX
	Trans	Helium	18.390	0.724	38.050	1.498	20.117	0.792	13	0.515			73.7	66.4	0.88	
	Long	Hydrogen	19.000	0.748	38.075	1.499	22.809	0.898	17	0.681	83.0	75.7			1.10	
	Trans	Hydrogen	18.796	0.740	38.075	1.499	18.999	.748	16	0.522	80.4	72.4			(2)	
Titanium A-110	Long	Helium	18.974	0.747	37.973	1.495	20.955	0.825	8	0.433			56.8	51.2	0.84	BZMP
	Trans	Helium	19.075	0.751	38.075	1.499	22.047	0.868	4	0.665	70.5	63.5			1.08	
	Long	Hydrogen	18.920	0.745	38.075	1.497	21.387	0.842	2	0.343	52.2	47.0			0.77	
	Trans	Hydrogen	18.847	0.742	37.948	1.494	21.437	0.844	3	0.605	69.5	62.6			0.95	

(1) Does not Meet ASTM Designation E399-72.

(2) R_{SC} not calculated due to absence of maximum load which was not required by ASTM 399-70T under which tests were conducted.

All Fatigue Precracking Accomplished per ASTM Designation E399-72.
Specimens Tested at 88.8 $MN/m^2\sqrt{m}$ (80.0 ksi $\sqrt{in.}$)/min Load Rate

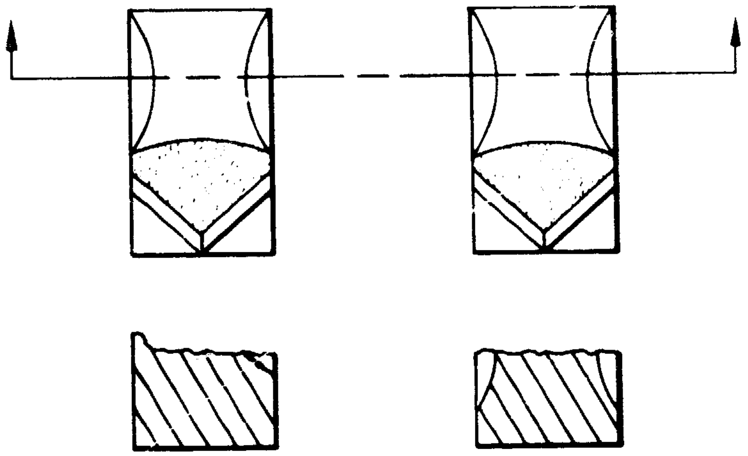


Figure VI-3. Fracture Appearances FD 51883

The thicknesses were chosen to stay within the load limits of the high pressure tensile testing rig. All specimens were precracked in axial tension-tension fatigue using a Sonntag fatigue machine, which operates at 30 Hz (1800 cpm). Precracking was conducted in air at approximately 300°K (80°F) at load levels (K_F), which later were verified not to exceed 60% of K_Q. Precracking was per ASTM Designation E-399.

Compact tensile and center slot fracture toughness tests were conducted on a 266.8 KN (60,000 lb) Tinius Olsen testing machine. The basic procedure and pressure vessel described in Section VIII were used. An internal load cell was used to eliminate the effects of friction on the load rod. The vessel is shown mounted in the tensile machine load frame in figure VI-4. The compact tensile specimen used a clip gage (figure VI-5) in conjunction with the load cell to record a load-displacement curve for each test. The center slot (notch) specimen used the load cell to record a load-time curve for each test.

Fracture mechanics testing was conducted on a P&WA™-designed test machine. Dead-weight loading was attained by constant gas pressure applied to an actuator from a large reservoir of 11.6-MN/m^2 (1700-psig) nitrogen. Both internal and external load cells were used to compensate for the effects of friction on the load rod. Crack propagation with time was monitored during K_{TH} testing by following the crack front with crack propagation continuity gages.

Fracture toughness values for the compact tensile specimens were calculated from the load (P_Q) established by a 5% deviation from the linear portion of the recorded load-displacement curve, the specimen thickness (B), width (W), and crack length (a) after fracture by the equation:

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

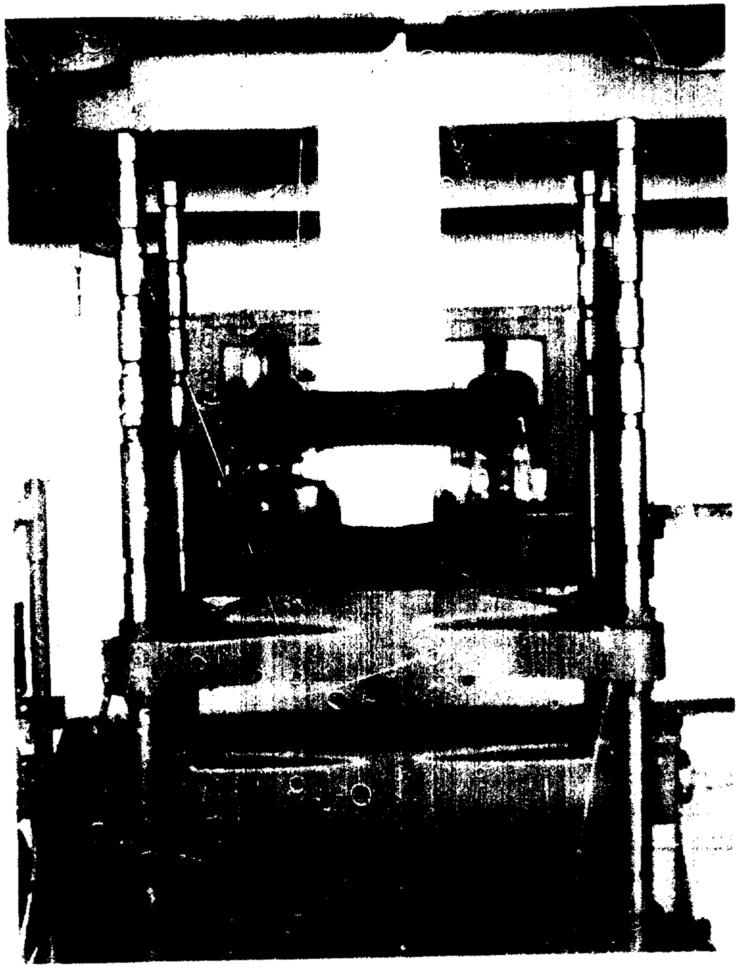


Figure VI-4. High Pressure Gaseous Environment Fracture Mechanics Test Vessel Installed on Tensile Machine in the Test Cell FC 21268

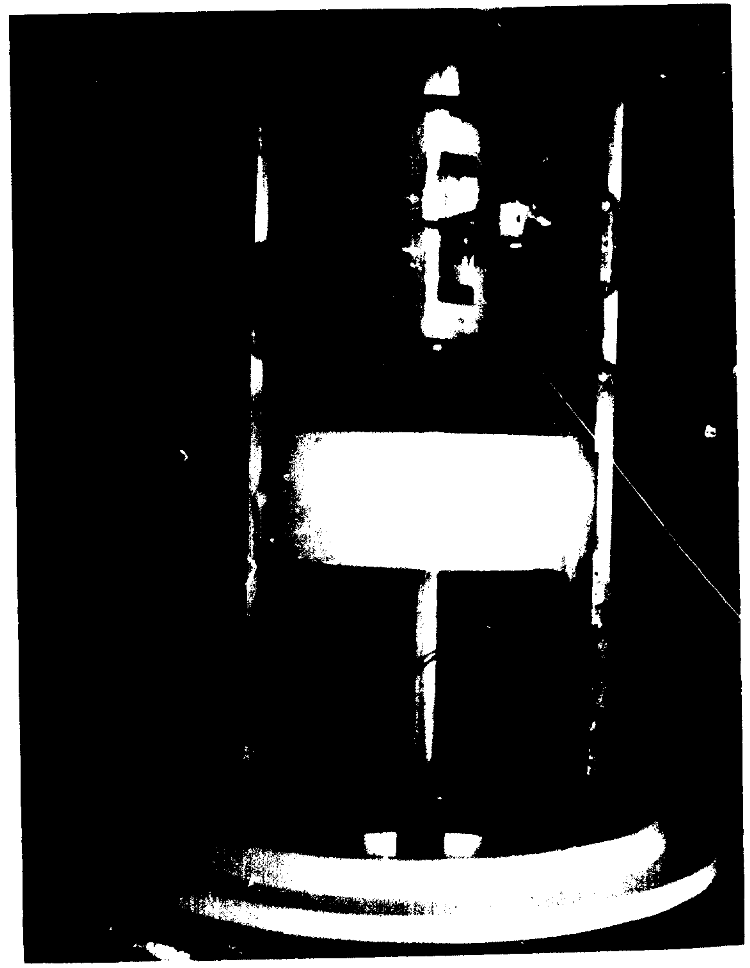


Figure VI-5. High Pressure Gaseous Environment Fracture Mechanics Test Vessel With Outer Chamber Removed and Fracture Toughness Specimen With COD Gage Attached FE 107942

A typical chart record of a fracture toughness test in high pressure hydrogen is shown in figure VI-6.

Fracture toughness values and initial stress intensities for the center slot (notch) specimens were calculated using the Brown and Srawley equation, reported in ASTM STP-410, from the load (P), the specimen thickness (B), width (W), and total crack length (2a) by the equation:

$$K_I = \frac{Pa^{1/2}}{BW} \left[1.77 + 0.277\,(2a/W) - 0.510\,(2a/W)^2 + 2.7\,(2a/W)^3\right]$$

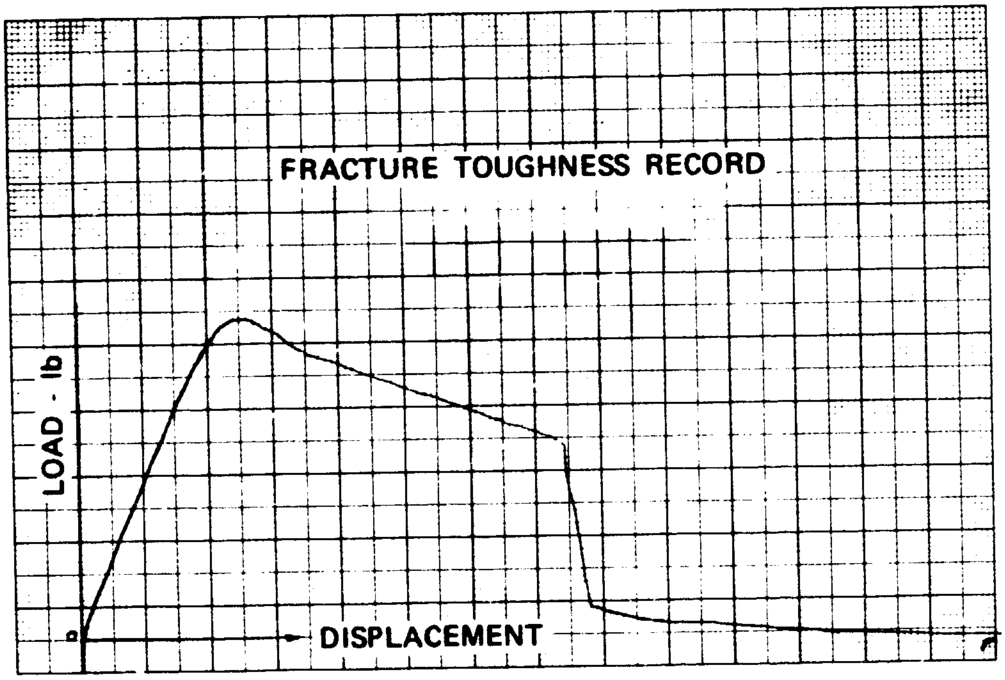


Figure VI-6. Actual Load-Displacement Record for a Fracture Toughness Test Conducted in a High Pressure Gaseous Environment (Titanium 6-4, 34.5-MN/m^2 5000-psig) Hydrogen at 300°K (80°F)) FD 51884A

SECTION VII
CREEP-RUPTURE

SECTION VII
CREEP-RUPTURE

A. INTRODUCTION

Creep-rupture properties were evaluated in gaseous environments of helium, hydrogen, hydrogen/water vapor, and dissociated hydrazine (anhydrous ammonia) under pressures of 3.45 MN/m^2 (500 psig) and 34.5 MN/m^2 (5000 psig) at 366°K (200°F), 951°K (1250°F), and 1144°K (1600°F). Creep rate, rupture life, percent elongation, and percent reduction of area were determined for four wrought nickel-, two cast nickel-, two wrought iron-, two wrought titanium-, and one wrought cobalt-base alloys.

B. RESULTS AND CONCLUSIONS

Degradation was determined from the percentage reduction in stress for the hydrogen environments, compared to the helium environment, to obtain a given stress-rupture life. Degradation percentages are listed in table VII-1, for all alloys tested. The influence of hydrogen environments was not consistent upon the different heats of the Astroloy, MAR M-200 DS, A-286, and Hayes 188 tested, indicating the need for additional testing before an alloy can be completely classified.

All the wrought nickel-base alloys were degraded by the hydrogen environment. Inconel 718 and Inconel 625 materials were severely degraded while WASPALOY® and Astroloy materials were only slightly degraded. Hydrogen degradation was reduced for WASPALOY when water vapor was mixed with the hydrogen. Astroloy appeared to have slightly better life in dissociated hydrazine than in hydrogen. However, this was comparing two heats of Astroloy material at two different pressures; therefore, a definite conclusion cannot be stated as to the effect of the dissociated hydrazine environment upon this material.

The cast nickel-base alloys, IN-100 and MAR M-200 DS, were degraded appreciably by the hydrogen environment. However, degree of degradation was inconsistent between the two heats of MAR M-200 DS material. One heat exhibited greater rupture life in hydrogen than in helium. Metallurgical investigation did not reveal any material anomalies. Hydrogen and water vapor mixture and dissociated hydrazine environments both gave greater life than that obtained when these materials were tested in helium.

The wrought iron-base AISI 347 material was not degraded by the hydrogen environment. The A-286 material was degraded by the hydrogen environment at 34.5 MN/m^2 (5000 psig) and 951°K (1250°F), but a second heat of this material exhibited no degradation at 3.45 MN/m^2 (500 psig) at the same temperature.

The results of the wrought titanium-base alloys were inconsistent because of the narrow margin between the test stress level and the ultimate tensile strength. It was necessary to test at high stress levels to obtain rupture lives in the 10- to 100-hr range. Hydrogen influence was evident by surface flaking of the material. Metallurgical investigation identified this as hydriding.

Table VII-1. Degradation Based on Stress Required for a Given Stress-Rupture Life

Material	Heat Code	Environment Temperature, °K (°F)	Environment Pressure, MN/m^2 (psig)	Degradation (%) for Life (hr) of: 10 hr H_2	10 hr H_2/H_2O	10 hr N_2H_4	100 hr H_2	100 hr H_2/H_2O	100 hr N_2H_4
Wrought Nickel-Base Alloys									
Inconel 718 1227°K (1750°F) Solution + Age	BVTO	951 (1250)	34.5 (5000)	31			33		
Inconel 718 1313°K (1900°F) Solution + Age	BVTO	951 (1250)	34.5 (5000)	27			28		
Inconel 625	BZCV	951 (1250)	34.5 (5000)	28			32		
WASPALOY®	L1288K13	951 (1250)	34.5 (5000)	ND(1)	(2)		14	(2)	
Astroloy	LKKC	951 (1250)	34.5 (5000)	ND			ND		
Astroloy	BYQO	951 (1250) 1144 (1600)	3.45 (500) 3.45 (500	See Section VII, Paragraph B, Results and Conclusions					
Cast Nickel-Base Alloys									
IN-100	P-9245	951 (1250)	34.5 (5000)	20	ND		26	ND	
MAR M-200 DS	P-9108	951 (1250)	34.5 (5000)	See Section VII, Paragraph B, Results and Conclusions					
MAR M-200	P-9199	1144 (1600)	3.45 (500)	11		ND	17		ND
Wrought Iron-Base Alloys									
A286	BZCU	951 (1250)	34.5 (5000)	11			23		
A286	BXOY	951 (1250)	3.45 (500) 34.5 (5000)	ND (2)		ND	ND (2)		0

Table VII-1. Degradation Based on Stress Required for a Given Stress-Rupture Life (Continued)

Material	Heat Code	Environment Temperature, °K (°F)	Environment Pressure, MN/m^2 (psig)	Degradation (%) for Life (hr) of: 10 hr H_2	10 hr H_2/H_2O	10 hr N_2H_4	100 hr H_2	100 hr H_2/H_2O	100 hr N_2H_4
AISI 347	BZCT	951 (1250)	34.5 (5000)	ND			0		
Wrought Titanium-Base Alloys									
Titanium 6-4	BZCS	366 (200)	34.5 (5000)	(2)			(2)		
A-110	BZCW	366 (200)	34.5 (5000)	(2)			(2)		
Wrought Cobalt-Base Alloy									
Haynes 188	YFYR	951 (1250)	34.5 (5000)	ND	ND		ND	ND	
Haynes 188	YGDM	951 (1250)	3.45 (500)	See Section VII, Paragraph B, Results and Conclusions					ND
		1144 (1600)	3.45 (500)	ND		ND	ND		

(1)ND Indicates no Effect or Degradation Less Than 10%

(2)Results Inconclusive for Limited Tests Conducted.

$$\text{Degradation} = \frac{\text{He Stress} - \text{H2, H2} \cdot \text{H2O or NH3 Stress}}{\text{He Stress}} \times 100$$

The wrought cobalt-base alloy, Haynes 188, was slightly degraded in hydrogen at 34.5 MN/m2 (5000 psig). The addition of water vapor to the hydrogen environment lessened this influence. A second heat of the material showed no degradation at 3.45 MN/m2 (500 psig) in either hydrogen or dissociated hydrazine. Several tests of specimens from the second heat of material ran out at approximately 100 hr, with no rupture and minimal creep. To obtain rupture life in the 10- to 100-hr range required operating beyond the yield point that produced initial elongation beyond the range of the creep measuring extensometry. Tests of the second heat of material were to obtain creep data only, with the knowledge that rupture would not occur. To obtain supplementary stress-rupture data in hydrogen, three specimens were prestrained and then tested in hydrogen to obtain both creep and rupture life.

The creep measuring extensometry was later modified for the dissociated hydrazine environment, which allowed the system to be reset after initial loading. This made it possible to operate at the high stress levels without prestraining the specimen. Results compared favorably with the prestrained specimens tested in hydrogen.

The influence of the hydrogen environment was also evidenced by increased creep rates, compared to the helium environment. A comparison of times required to obtain 1 and 2% creep is listed in table VII-2.

Test results are listed in table VII-3. Stress-rupture life is plotted in figures VII-1 through VII-16. Creep-to-rupture data are plotted in figures VII-17 through VII-34.

C. TEST PROCEDURE

Creep-rupture tests were conducted on a modified 53.4-KN (12,000-lb) capacity Arcweld Model JE creep-rupture machine. The test machine was explosion-proofed and located in a test cell open to the atmosphere. Controls and data recording equipment were located in an adjacent blockhouse. A high pressure test vessel (figures VII-35 and VII-36) contained the test specimen, furnace, and extensometry. The pressure vessel was suspended in the creep-rupture machine and counter-balanced to maintain the load lever arm in a level position.

The design of the test specimen included integral collars for positive location and gripping of creep-measuring extensometer heads. The ends of the specimen were flat pin joints, rather than conventionally threaded joints, and acted as part of a two pin joint. Load rods and adapters also incorporated pin joints, which, in effect, formed universal joints at the ends of the specimen to eliminate alignment errors and resulting bending stresses upon the specimen.

The extensometer was a dual proximity probe averaging type and was contained inside the pressure vessel. The extensometer output was recorded in the adjacent blockhouse as elongation vs time. An elapsed time meter validated the rupture life indicated on the strip chart.

The specimen heater was a resistance-wire, two-zone, split-clamshell configuration. Independent zone control provided even temperature over specimen gage length. Temperature was monitored and controlled by three chromel-alumel thermocouples looped around the gage section. The entire system was contained inside the pressure vessel.

The pressure vessel used a Grayloc high pressure hub connector to facilitate assembly/disassembly. The assembled pressure vessel was mounted in the creep-rupture machine with load rod adapters and bulkhead connections for the furnace, thermocouples, and extensometer. Test stand plumbing and pressure vessel were purged and leak checked. The system was evacuated to 100μ mercury.

The procedure at this point varied, depending on the environment required. Helium or hydrogen test gas was introduced directly. The hydrogen and water vapor environment was obtained by injecting water from a separately purged system prior to pressurization with hydrogen. Water concentration was 3% by weight. Ammonia was introduced as a gas-liquid mixture, and the pressure vessel heated above the critical temperature to completely vaporize the ammonia. The ammonia environment required some substitutions in system components to avoid material reactions. It was also necessary to relocate the extensometer probe system out of the chamber, as the sensing element of the probe was not compatible with the environment. Creep information was obtained for these tests by measuring differential movement between the specimen upper adapter and the vessel base.

The specimen was heated while pressure increase, due to temperature increase, was monitored and vented as necessary. Stable temperature and pressure were obtained in 1-1/2 to 2 hr. An initial room temperature calibration with a strain gaged specimen had established the load induced in the specimen due to the gas pressure acting over the differential areas. The test load applied was adjusted for this induced load and for seal frictional losses. The test system was secured for automatic control and monitoring. When the specimen failed, all control equipment was automatically deactivated. The specimen was removed, and final gage length and diameter were measured and recorded to determine the percent elongation and percent reduction of area.

Table VII-2. Degradation Based on Time Required for a Given Creep

Material	Heat Code	Environment Temperature, °K (°F)	Environment Pressure, MN/m² (psig)	Stress, MN/m² (ksi)	Time (hr) to Creep (%) of: 1.0% He	1.0% H_2	1.0% H_2/H_2O	1.0% N_2H_4	2.0% He	2.0% H_2	2.0% H_2/H_2O	2.0% N_2H_4
Wrought Nickel-Base Alloys												
Inconel 718 1227°K (1750°F) Solution + Age	BVTO	951 (1250)	34.5 (5000)	758.4 (110)	4.0				5.3			
				558.5 (81)		1.6				2.2		
Inconel 718 1313°K (1900°F) Solution + Age	BVTO	951 (1250)	34.5 (5000)		Insufficient Data							
Inconel 625	BZCV	951 (1250)	34.5 (5000)	434.4 (63)	2.0				3.5			
				286.1 (41.5)		1.3				3		
WASPALOY®	L1288K13	951 (1250)	34.5 (5000)	606.7 (88)	21.0	12.5			36.3	19.0		
Astroloy	LKKC	951 (1250)	34.5 (5000)	792.9 (115)	17.5	18.0			49.0	[illegible]		
Astroloy	BYQO	951 (1250)	3.45 (500)		See Section VII, Paragraph B, Results and Conclusions							
		1144 (1600)	3.45 (500)									
Cast Nickel-Base Alloys												
IN-100	P-9245	951 (1250)	34.5 (5000)		0.5% Total Creep							
MAR M-200 DS	P-9108	951 (1250)	34.5 (5000)	792.9 (115)	See Section VII, Paragraph B, Results and Conclusions							
MAR M-200 DS	P-9199	1144 (1600)	3.45 (500)	482.6 (70)	3.6	1.9			10.6	3.7		
				399.9 (58)	41.5		121.0		55.5		>140	
Wrought Iron-Base Alloys												
A286	BZCU	951 (1250)	34.5 (5000)	368.2 (53.4)	90.0				120.0			
				362 (52.5)		15.0				22.0		
A286	BXOY	951 (1250)	3.45 (500)	534.4 (77.5)	0.7				1.0			
				444.7 (64.5)	40.0				51.3			
			34.5 (5000)		Insufficient Data							
AISI 347	BZCT	951 (1250)	34.5 (5000)	133.8 (19.4)	9.0				16.5			
				136.5 (19.8)		14.0				21.0		
Wrought Titanium-Base Alloys												
Titanium 6-4	BZCS	366 (200)	34.5 (5000)	930.8 (135)	45.0	0.6			< 140.0	1.7		
A-110	BZCW	366 (200)	34.5 (5000)	792.9 (115)	3.0	0.35			130.0	3.6		
Wrought Cobalt-Base Alloy												
Haynes 188	YFYR	951 (1250)	34.5 (5000)	482.6 (70)	0.7	0.4	0.9		1.5	0.9	2.4	
Haynes 188	YGDM	951 (1250)	3.45 (500)	395.1 (57.3)		2.0		2.7		5.0		8.0
		1144 (1600)	3.45 (500)	110.3 (16)	2.0	0.9		2.45	4.9	2.0		5.4
				131 (19)	0.95	0.9		0.35	2.3	1.9		1.0

Table VII-3. Creep-Rupture Properties of Materials in Gaseous Environment

Heat Code	Material	Test Temperature, °K	Test Temperature, °F	Environment	Pressure, MN/m²	Pressure, psig	Stress Level, MN/m²	Stress Level, ksi	Time to Creep, hr, 0.5%	Time to Creep, hr, 1.0%	Time to Creep, hr, 2.0%	Time to Rupture, hr	El, %	RA, %
BVTO	Inconel 718	951	1250	Helium	34.5	5000	636.4	92.3	<0.2%	Total Creep		100.0(1)		
		951	1250	Helium	34.5	5000	758.4	110.0	2.5	4.0	5.3	7.2	9.5	38.2
	1227°K (1750°F)	951	1250	Hydrogen	34.5	5000	450.9	65.4	53.0	79.0	103.0	118.6	5.3	36.4
	Solution - Age	951	1250	Hydrogen	34.5	5000	587.4	85.2	0.1	0.15	0.25	0.4	7.8	48.8
	Heat Treat	951	1250	Hydrogen	34.5	5000	558.5	81.0	1.0	1.6	2.2	3.1	10.2	52.3
BVTO	Inconel 718	951	1250	Helium	34.5	5000	624.0	90.5	10.5	17.5	24.5	32.2	6.4	38.8
	1313°K (1900°F)	951	1250	Helium	34.5	5000	782.6	113.5	<0.3%	Total Creep		0.3	(2)	
	Solution - Age	951	1250	Hydrogen	34.5	5000	382.0	55.4	<0.1%	Total Creep		100.0(1)	(2)	
	Heat Treat	951	1250	Hydrogen	34.5	5000	486.1	70.4	<0.5%	Total Creep		6.2	1.2	4.7
		951	1250	Hydrogen	34.5	5000	589.5	85.5	Failed on Loading					
BZCV	Inconel 625	951	1250	Helium	34.5	5000	368.9	53.5	0.6	1.0	1.6	4.0(3)	7.0	51.5
		951	1250	Helium	34.5	5000	434.4	63.0	1.2	2.0	3.5	21.7	14.7	52.5
		951	1250	Helium	34.5	5000	434.4	63.0	0.45	0.7	1.2	25.7	39.2	52.4
		951	1250	Hydrogen	34.5	5000	313.0	45.4	Failed on Loading				18.9	76.7
		951	1250	Hydrogen	34.5	5000	314.4	45.6	Failed on Loading			86.5(1)	17.6	78.4
		951	1250	Hydrogen	34.5	5000	262.0	38.0	< 0.25% Total Creep					
		951	1250	Hydrogen	34.5	5000	286.1	41.5(5)	0.6	1.3	3	49.2	19.5	47.5
L1288K13	WASPALOY®	951	1250	Helium	34.5	5000	758.4	110.0	1.0	2.0	3.9	5.6	4.5	8.7
		951	1250	Helium	34.5	5000	606.7	88.0	9.0	21.0	36.3	58.2	7.5	13.1
		951	1250	Hydrogen	34.5	5000	658.4	95.5	6.4	10.5		13.8	3.7	10.6
		951	1250	Hydrogen	34.5	5000	606.7	88.0	7.5	12.5	19.0	25.1	5.4	12.1
		951	1250	Hydrogen/ Water Vapor	34.5	5000	658.4	95.5	< 0.2% Total Creep			0.3(5)	1.8	6.8
		951	1250	Hydrogen/ Water Vapor	34.5	5000	606.7	88.0	< 0.2% Total Creep			41.8	2.1	9.8
LKKC	Astroloy	951	1250	Helium	34.5	5000	792.9	115.0	3.8	17.5	49.0	87.8	5.5	13.0
		951	1250	Helium	34.5	5000	930.8	135.0	No Measurement			3.8	5.1	11.5
		951	1250	Hydrogen	34.5	5000	896.3	130.0	0.8	1.5	3.3	4.5	3.9	7.7
		951	1250	Hydrogen	34.5	5000	792.9	115.0	3.5	18.0	37.0	59.6	6.5	13.0
BYQO	Astroloy	951	1250	Dissociated Hydrazine	3.45	500	930.8	135.0	0.15	0.4	1.15	5.6	13.7	25.2
		951	1250	Dissociated Hydrazine	3.45	500	868.7	126.0	3.5	7.7	16.3	34.7	6.0	12.7
		951	1250	Dissociated Hydrazine	3.45	500	896.3	130.0	1.4	6.9	18.2	31.4	10.4	16.2
		1144	1600	Dissociated Hydrazine	3.45	500	379.2	55.0	0.25	0.85	2.5	5.2	10.7	31.2
		1144	1600	Dissociated Hydrazine	3.45	500	331.0	50.0	0.75	1.65	3.0	5.0	8.8	26.8
		1144	1600	Dissociated Hydrazine	3.45	500	275.8	40.0	12.5	24.0	40.0	57.2	9.0	30.5
P-9245	IN-100	951	1250	Helium	34.5	5000	551.6	80.0	<0.2%	Total Creep		140.0(1)	(2)	(2)
		951	1250	Helium	34.5	5000	620.5	90.0	<0.3%	Total Creep		7.6	(2)	(2)
		951	1250	Hydrogen	34.5	5000	620.5	90.0	<0.2%	Total Creep		0.2	0.4	1.4
		951	1250	Hydrogen	34.5	5000	551.6	80.0	<0.5%	Total Creep		1.6		
		951	1250	Hydrogen/ Water Vapor	34.5	5000	551.6	80.0	No Measurement			162.0(1)		
		951	1250	Hydrogen/ Water Vapor	34.5	5000	620.5	90.0	No Measurement			3	0.8	3.0

Table VII-3. Creep-Rupture Properties of Materials in Gaseous Environment (Continued)

Heat Code	Material	Test Temperature, °K	Test Temperature, °F	Environment	Pressure, MN/m^2	Pressure, psig	Stress Level, MN/m^2	Stress Level, ksi	Time to Creep, hr, 0.5%	Time to Creep, hr, 1.0%	Time to Creep, hr, 2.0%	Time to Rupture hr	El, %	RA, %
P-9108	MAR M-200 DS	951	1250	Helium	34.5	5000	861.8	125.0	No Measurement			5.6[5]	1.5	6.3
		951	1250	Helium	34.5	5000	861.8	125.0	6.0	8.3		9.6	2.5	4.8
		951	1250	Helium	34.5	5000	792.9	115.0	27.0			36.7	1.7	8.6
		951	1250	Hydrogen	34.5	5000	861.8	125.0	6.5	8.8	11.1	35.6	6.7	6.9
		951	1250	Hydrogen	34.5	5000	861.8	125.0	No Measurement			56.2[5]	5.0	3.9
		951	1250	Hydrogen	34.5	5000	792.9	115.0	22.0	24.6	31.8	49.9	3.9	7.3
		951	1250	Hydrogen and Water Vapor	34.5	5000	861.8	125.0	7.5	13.5		15.4	1.5	6.0
		951	1250	Hydrogen and Water Vapor	34.5	5000	792.9	115.0	29.6	45.0		64.5	2.8	5.9
P-9199	MAR M-200 DS	1144	1600	Helium	3.45	500	482.6	70.0	0.8	3.6	10.6	18.4	9.9	25.1
		1144	1600	Helium	3.45	500	399.9	58.0	24.0	41.5	55.5	67.2	9.1	21.4
		1144	1600	Hydrogen	3.45	500	482.6	70.0	0.8	1.9	3.7	7.9	10.4	20.5
		1144	1600	Hydrogen	3.45	500	310.3	45.0	2.9	28.0	70.0	113.0	10.8	28.9
		1144	1600	Hydrogen	3.45	500	324.1	47.0	15.0	39.5	59.0	97.5	12.2	30.5
		1144	1600	Dissociated Hydrazine	3.45	500	482.6	70.0	4.75	11.0	18.35	30.7	8.0	22.0
		1144	1600	Dissociated Hydrazine	3.45	500	399.9	58.0	47.4	121.0	< 2.0% Total	141.0[1]	1.2	2.0
		1144	1600	Dissociated Hydrazine	3.45	500	324.1	47.0	113.5	182.5	< 2.0% Total	238.4[1]	1.6	3.2
BZCU	A-286	951	1250	Helium	34.5	5000	368.2	53.4	68.0	90.0	120.0	159.8	8.6	36.6
		951	1250	Helium	34.5	5000	444.0	64.4	10.4	16.6	22.8	31.7	9.3	44.2
		951	1250	Helium	34.5	5000	444.0	64.4	3.5	6.5	8.8	22.8	27.5	27.8
		951	1250	Hydrogen	34.5	5000	362.0	52.5	9.0	15.0	22.0	35.6	7.4	26.0
		951	1250	Hydrogen	34.5	5000	444.0	64.4	1.8	2.8	4.2	8.6	12.1	40.0
BXOY	A-286	951	1250	Helium	3.45	500	534.4	77.5	0.4	0.7	1.0	2.4	18.5	36.8
		951	1250	Helium	3.45	500	444.7	64.5	31.0	40.0	51.3	69.7	19.2	60.8
		951	1250	Hydrogen	3.45	500	534.4	77.5	No Measurement			7.0	22.8	53.2
		951	1250	Hydrogen	3.45	500	477.8	69.3	No Measurement			33.5	18.2	47.0
		951	1250	Hydrogen	3.45	500	426.1	61.8	No Measurement			145.8	13.3	48.7
		951	1250	Hydrogen	34.5	5000	[illegible]4.7	64.5	No Measurement			15.2	22.3	39.3
		951	1250	Hydrogen	34.5	5000	[illegible].0	52.5	No Measurement			137.5[1]	1.5	1.0
		951	1250	Hydrogen	34.5	5000	310.3	45.0	No Measurement			216.0[1]	0	0
		951	1250	Dissociated Hydrazine	3.45	500	534.4	7[illegible]5	2.5	3.55	4.85	7.7	12.7	47.0
		951	1250	Dissociated Hydrazine	3.45	500	477.8	69.3	6.25	[illegible].15	10.5	16.8	11.7	37.4
		951	1250	Dissociated Hydrazine	3.45	500	444.7	64.5	30.5	43.0	56.7	75.1	13.2	50.0
BZCT	AISI 347	951	1250	Helium	34.5	5000	278.6	40.4	< 0.1	0.15	0.2	0.3	8.7	48.3
		951	1250	Helium	34.5	5000	133.8	19.4	3.0	9.0	16.5	24.5	8.5	43.7
		951	1250	Hydrogen	34.5	5000	81.4	11.8	60.0	115.0	187.0	187.0[1]		
		951	1250	Hydrogen	34.5	5000	268.2	38.9	Failed on Loading				8.5	[illegible]2.4
		951	1250	Hydrogen	34.5	5000	136.5	19.8	8.0	14.0	21.0	23.9	2.6	14.3
BZCS	Titanium 6-4	366	200	Helium	34.5	5000	965.3	140.0	Failed on Loading			140.3[1]		
		366	200	Helium	34.5	5000	930.8	135.0	2.75	45.0	1.13% Total			
		366	200	Hydrogen	34.5	5000	965.3	140.0	< 0.1	0.3	0.67	0.67	4.2[4]	17.9
		366	200	Hydrogen	34.5	5000	930.8	135.0	< 0.1	0.6	1.7	1.7	3.2[4]	11.8

Table VII-3. Creep-Rupture Properties of Materials in Gaseous Environment (Continued)

Heat Code	Material	Test Temperature, °K	Test Temperature, °F	Environment	Pressure, MN/m^2	Pressure, psig	Stress Level, MN/m^2	Stress Level, ksi	Time to Creep, hr, 0.5%	Time to Creep, hr, 1.0%	Time to Creep, hr, 2.0%	Time to Rupture, hr	El, %	RA, %
BZCW	Titanium A-110	366	200	Helium	34.5	5000	813.6	118.0	Failed on Loading			130.0(1)		
		366	200	Helium	34.5	5000	792.9	115.0	0.18	3.0	130.0	45.3	5.1(4)	10.0
		366	200	Hydrogen	34.5	5000	792.9	115.0	< 0.1	0.35	3.6	21.7	6.5(4)	20.9
		366	200	Hydrogen	34.5	5000	813.6	118.0	< 0.1	0.25	2.75			
YFYR	Haynes 188	951	1250	Helium	34.5	5000	482.6	70.0	0.3	0.7	1.5	9.4	33.8(2)	33.2
		951	1250	Helium	34.5	5000	365.4	53.0	3.0	4.5	9.0	80.3	20.7(2)	23.1
		951	1250	Hydrogen	34.5	5000	365.4	53.0	0.6	1.4	4.0	43.2	18.5(2)	20.5
		951	1250	Hydrogen	34.5	5000	482.6	70.0	0.15	0.4	0.9	5.4	30.9(2)	32.8
		951	1250	Hydrogen and Water Vapor	34.5	5000	365.4	53.0	No Measurement			55.5	10.2(2)	23.2
		951	1250	Hydrogen and Water Vapor	34.5	5000	482.6	70.0	0.4	0.9	2.4	6.35	21.2(2)	32.8
YGDM	Haynes 188	951	1250	Helium	3.45	500	193.1	28.0	< 0.2%	Total Creep		89.0(1)	0.21	0
		951	1250	Helium	3.45	500	244.8	35.5	< 0.4%	Total Creep		88.4(1)	0.37	0
		951	1250	Hydrogen	3.45	500	244.8	35.5	< 0.3%	Total Creep		94.2(1)	0.75	2.0
		951	1250	Hydrogen	3.45	500	275.8	40.0	30.0	70.0		118.3(1)	3.4	3.8
		951	1250	Hydrogen	3.45	500	193.1	28.0	< 0.2%	Total Creep		89.5(1)	0.16	0
		951	1250	Hydrogen	3.45	500	464.0(6)	67.3	No Measurement			14.6	25.7	28.6
		951	1250	Hydrogen	3.45	500	395.1(6)	57.3	0.9	2.0	5.0	41.4	20.0	24.8
		951	1250	Hydrogen	3.45	500	375.8(6)	54.5	1.25	2.25	8.25	72.9	17.6	22.0
		951	1250	Dissociated Hydrazine	3.45	500	468.8	68.0	0.4	1.3	2.9	16.4	22.8	25.1
		951	1250	Dissociated Hydrazine	3.45	500	395.1	57.3	1.0	2.75	8.0	79.8	17.5	24.2
		951	1250	Dissociated Hydrazine	3.45	500	244.8	35.5	73.0	< 1.0% Creep		112.2(1)	1.3	1.9
		1144	1600	Helium	3.45	500	131.0	19.0	0.4	0.95	2.3	29.7	44.5	58.0
		1144	1600	Helium	3.45	500	110.3	16.0	0.9	2.05	4.9	66.2	34.6	50.4
		1144	1600	Hydrogen	3.45	500	131.0	19.0	0.45	0.9	1.9	25.4	41.5	55.3
		1144	1600	Hydrogen	3.45	500	110.3	16.0	0.4	0.9	2.05	39.4	37.1	51.0
		1144	1600	Hydrogen	3.45	500	82.7	12.0	4.3	19.3	225.0	256.0(1)	2.3	1.9
		1144	1600	Dissociated Hydrazine	3.45	500	151.7	22.0	0.05	0.25	0.6	12.7	36.0	60.0
		1144	1600	Dissociated Hydrazine	3.45	500	131.0	19.0	0.1	0.35	1.0	22.0	33.1	55.5
		1144	1600	Dissociated Hydrazine	3.45	500	110.3	16.0	1.25	2.45	5.4	54.6	30.8	50.5

Elongation Measured Over 25.4 mm (1 in.) Original Gage Length

Notes: (1) Did not Fail; Test Discontinued

(2) Includes Yielding Upon Loading

(3) Rig Shutdown Due to Temperature Loss Immediately Prior to Failure

(4) Failure Mode Precludes Accurate Measurement

(5) Failed in Radius

(6) Prestrained in Argon at Temperature and Stress Prior to Test

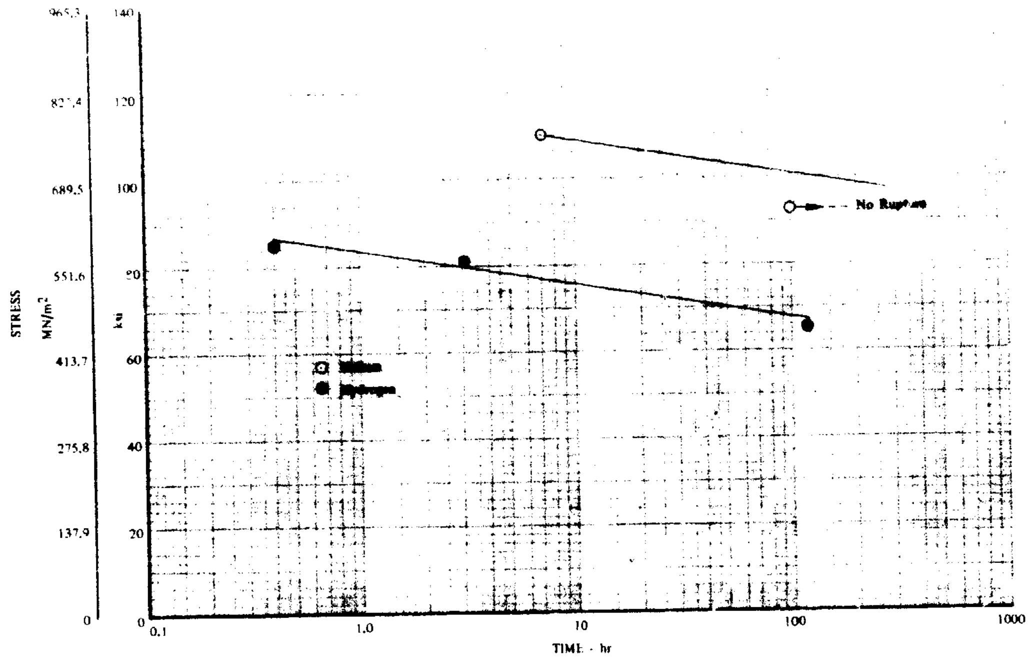


Figure VII-1. Stress-Rupture of Inconel 718, 1227°K (1750°F) Solution at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96651

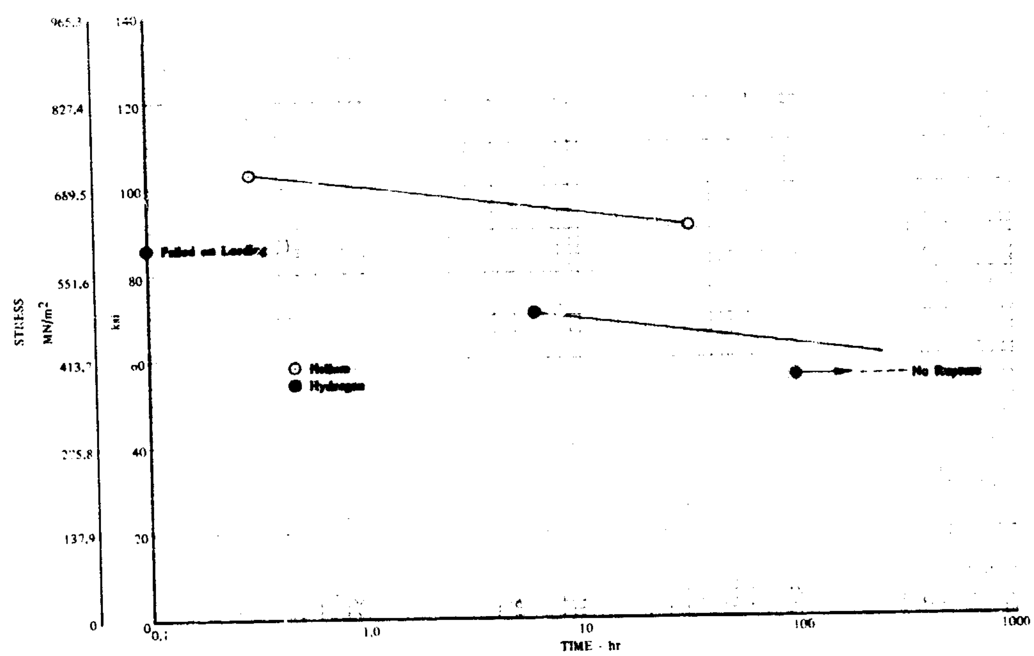


Figure VII-2. Stress-Rupture of Inconel 718, 1313°K (1900°F) Solution at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96652

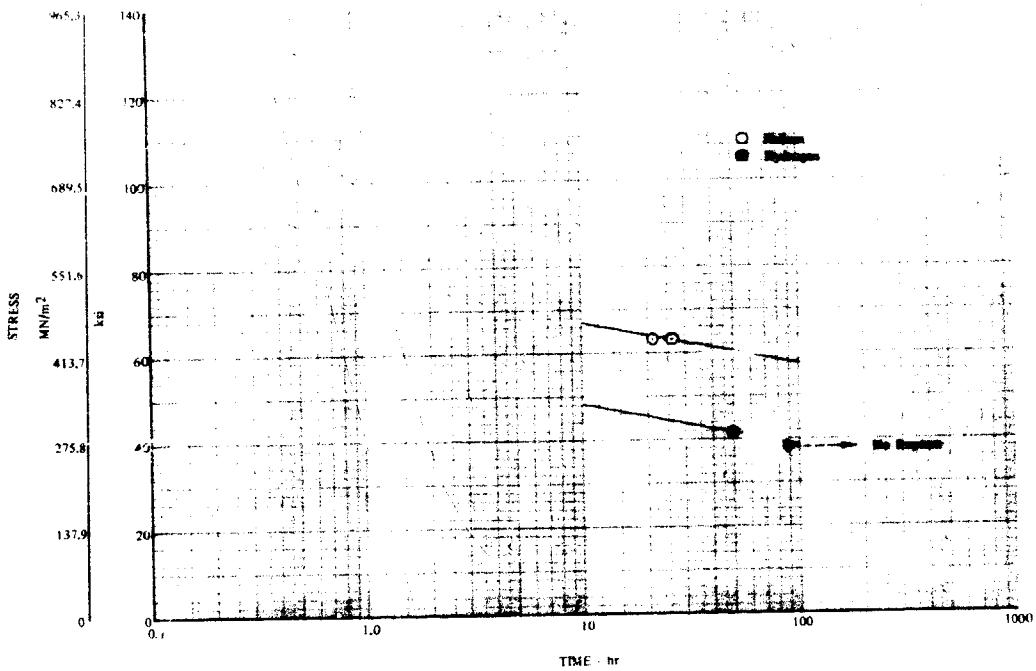


Figure VII-3. Stress-Rupture of Inconel 625 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96653

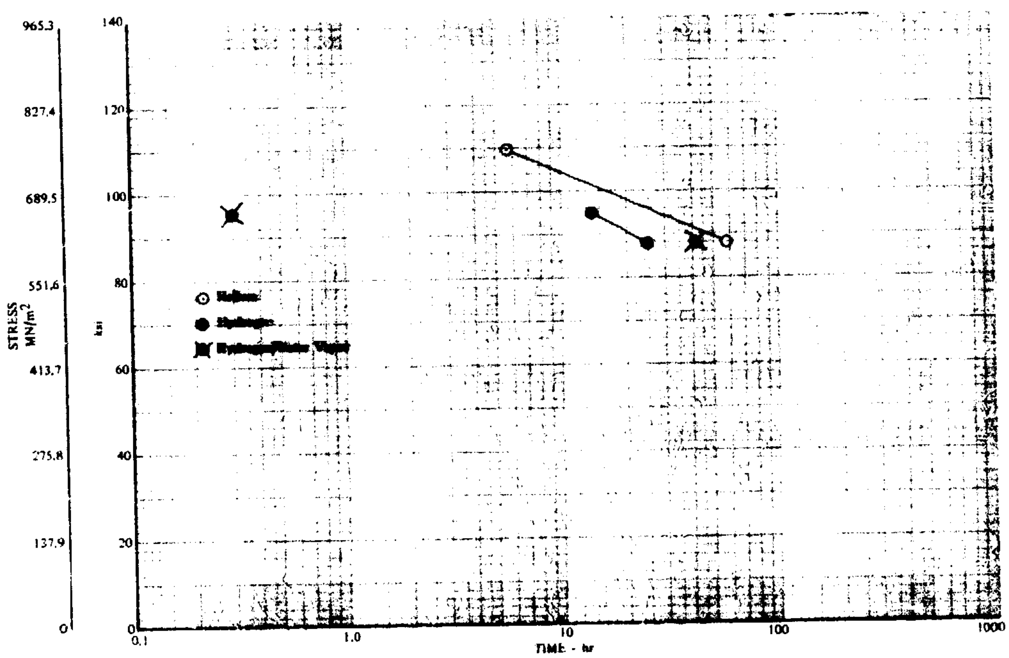


Figure VII-4. Stress-Rupture of WASPALOY® at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96655

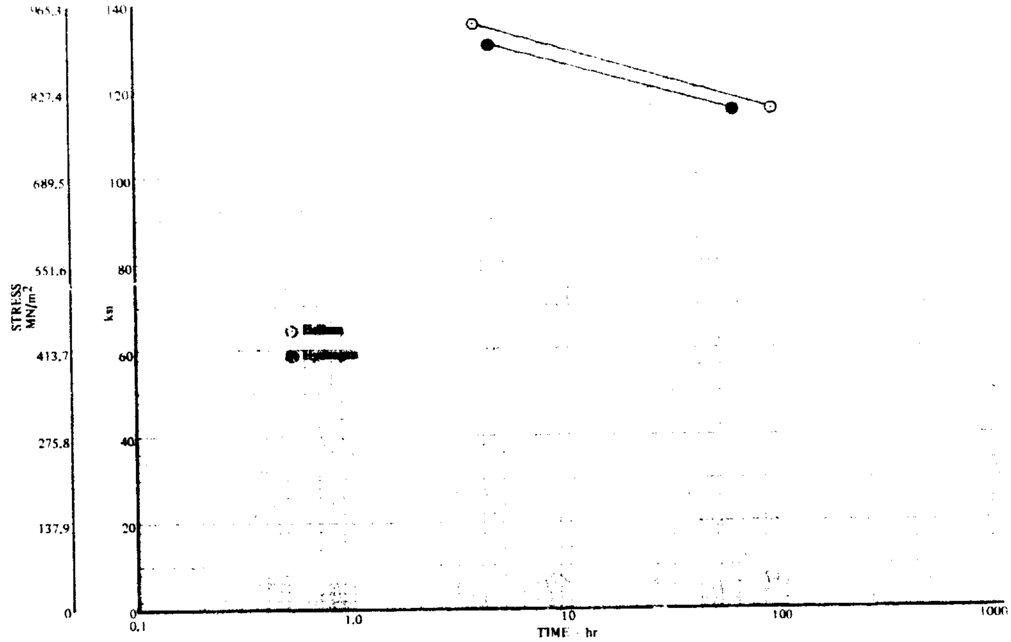


Figure VII-5. Stress-Rupture of Astroloy at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat LKKC DF 96654

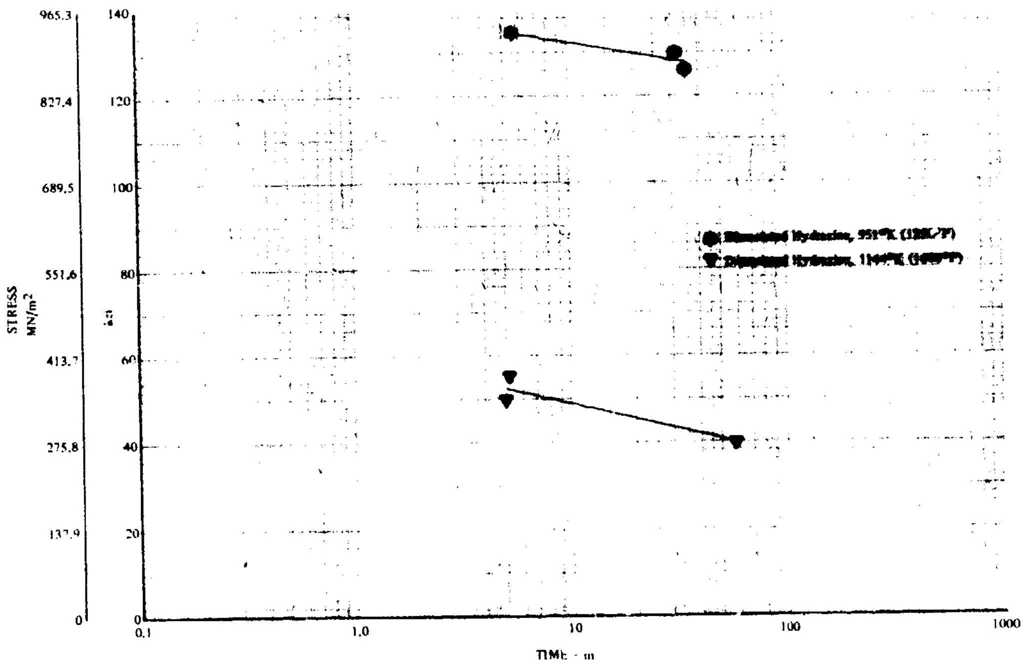


Figure VII-6. Stress-Rupture of Astroloy in Dissociated Hydrazine at 951°K (1250°F) and 1144°K (1600°F) at 3.45 MN/m^2 (500 psig) Pressure; Heat BYQO DF 96656

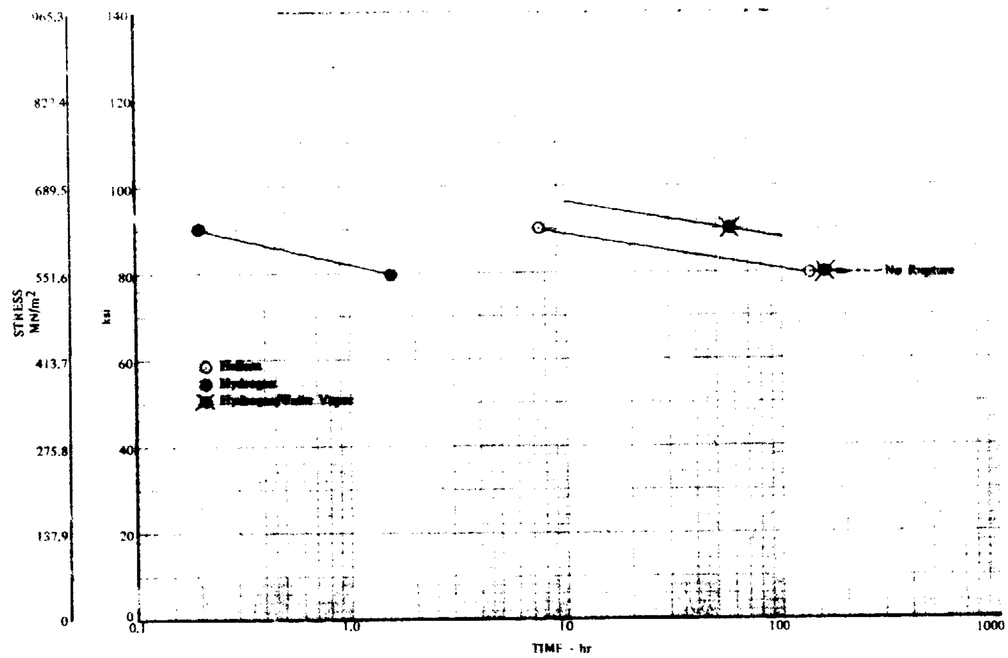


Figure VII-7. Stress-Rupture of IN-100 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96657

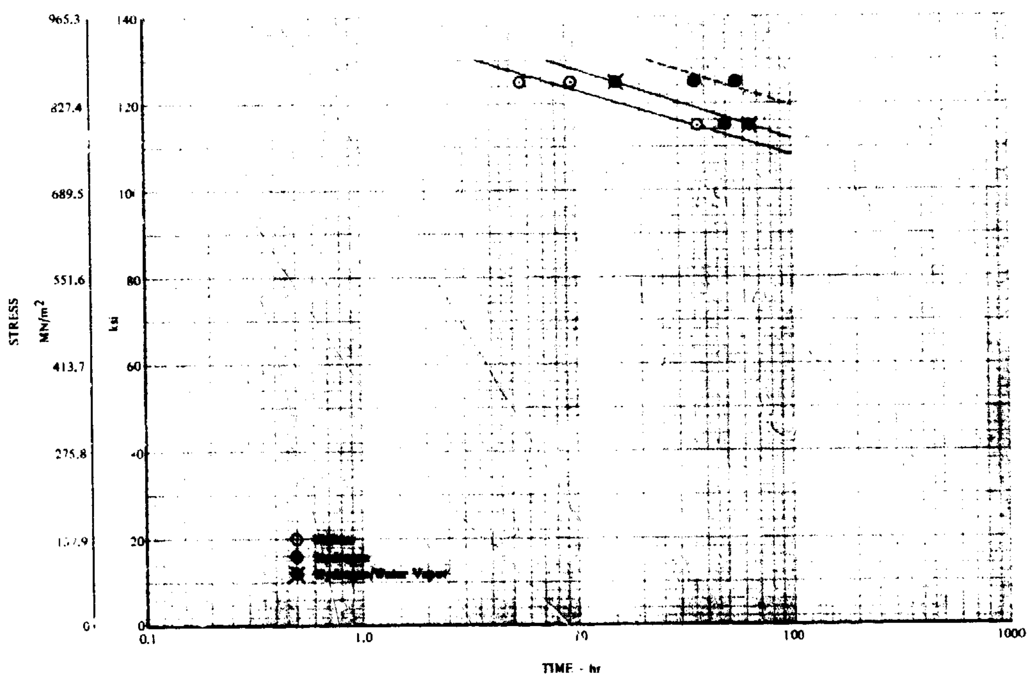


Figure VII-8. Stress-Rupture of MAR M-200 DS at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat P-9108 DF 96658

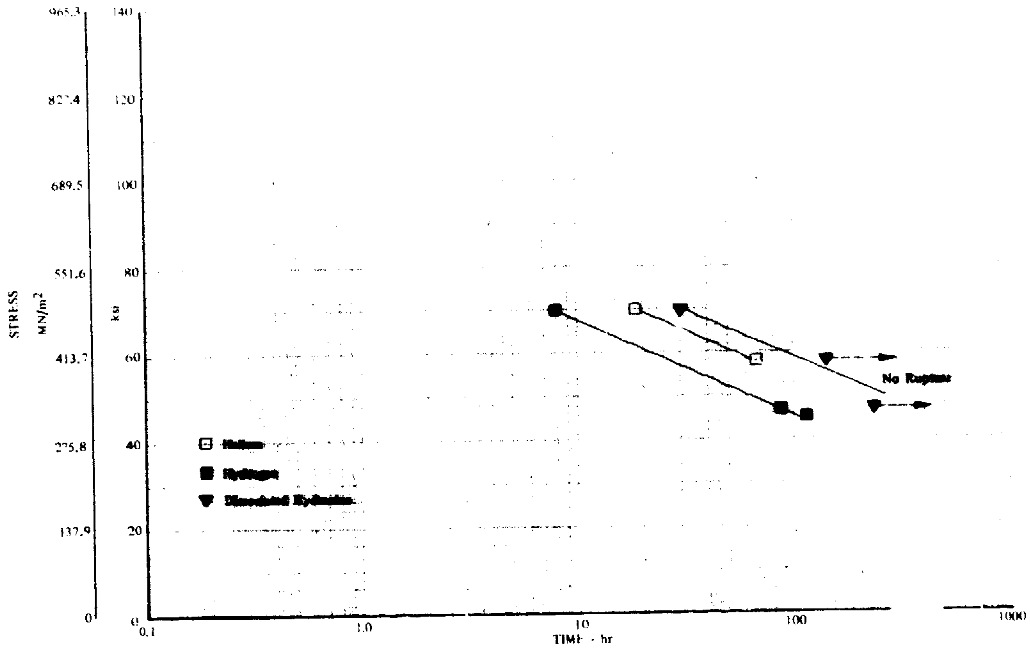


Figure VII-9. Stress-Rupture of MAR M-200 DS at 1144°K (1600°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat P-9199 DF 96659

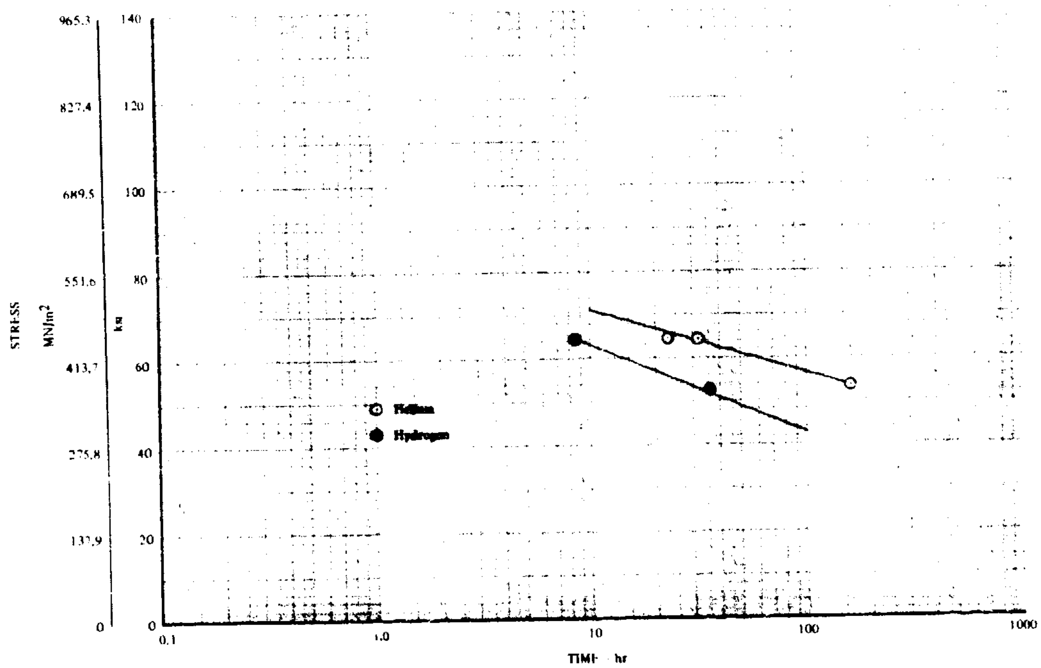


Figure VII-10. Stress-Rupture of A-286 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat BZCU DF 96660

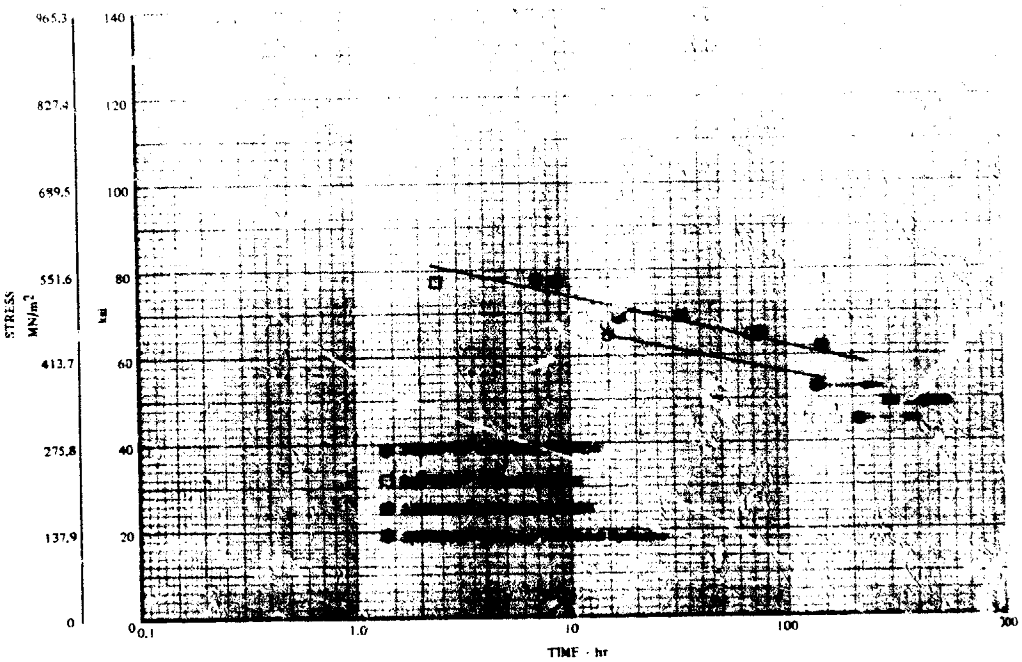


Figure VII-11. Stress-Rupture of A-286 at 951°K (1250°F) and 3.45 or 34.5 MN/m^2 (500 or 5000 psig) Pressure; Heat BXOY   DF 96661

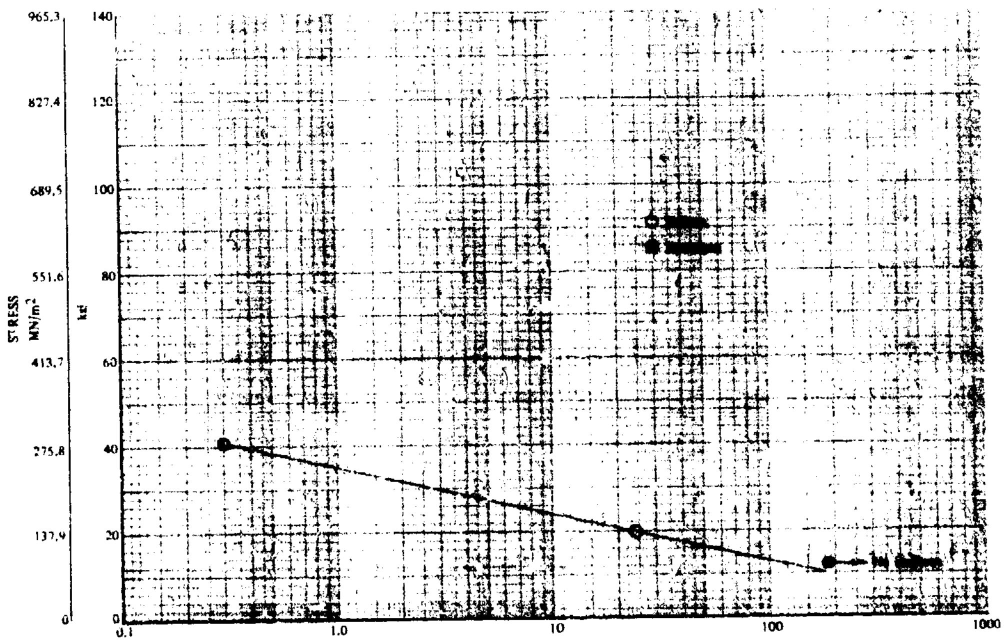


Figure VII-12. Stress-Rupture of AISI 347 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig)   DF 96662

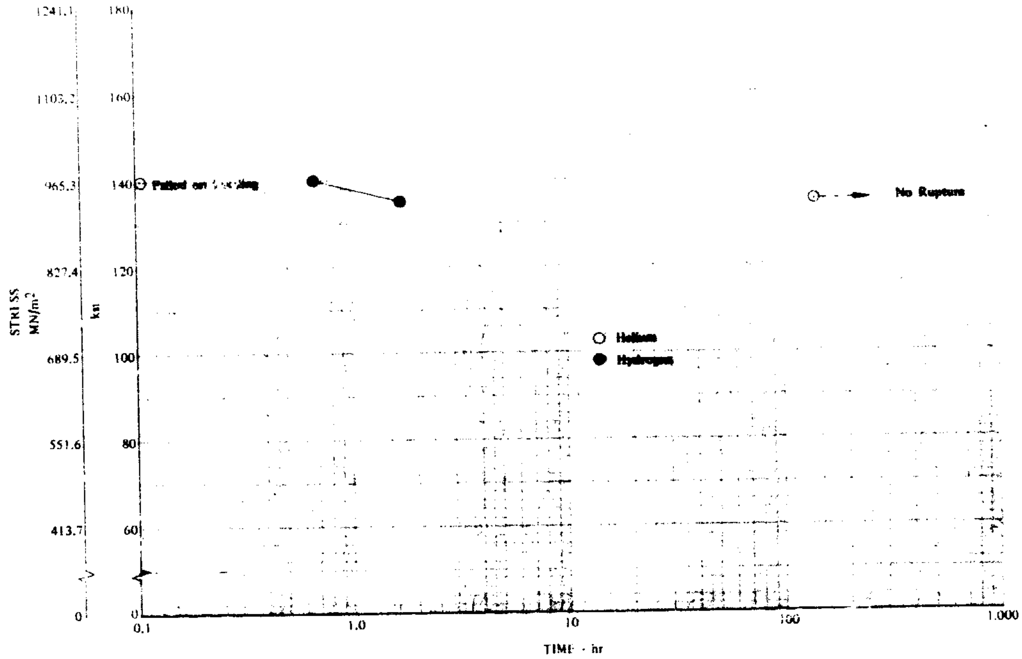


Figure VII-13. Stress-Rupture of Titanium 6-4 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96663

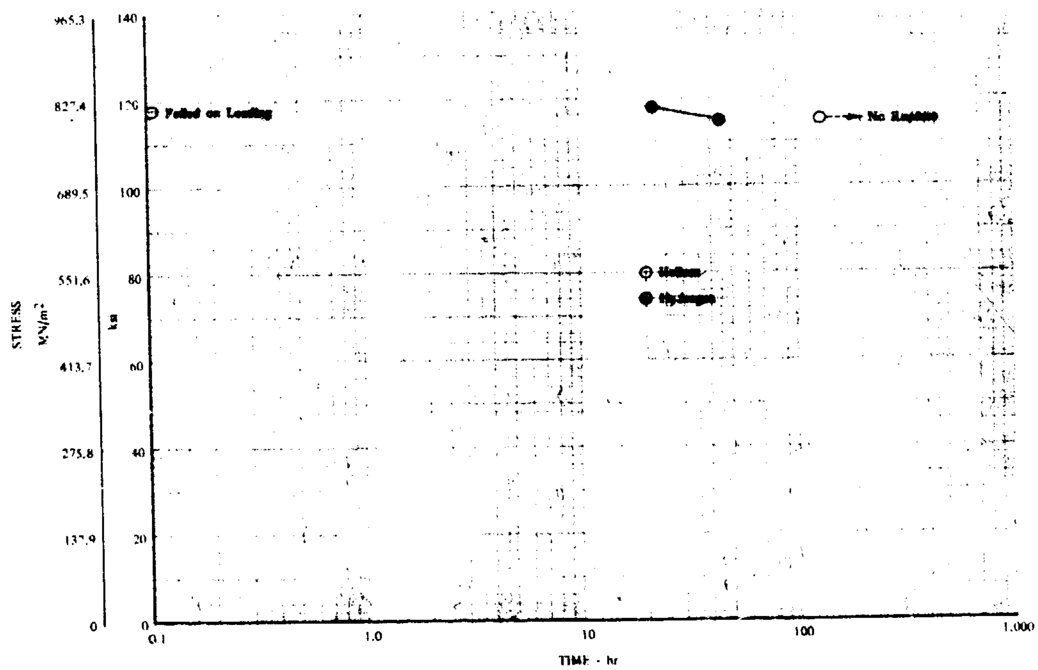


Figure VII-14. Stress-Rupture of Titanium A-110 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96664

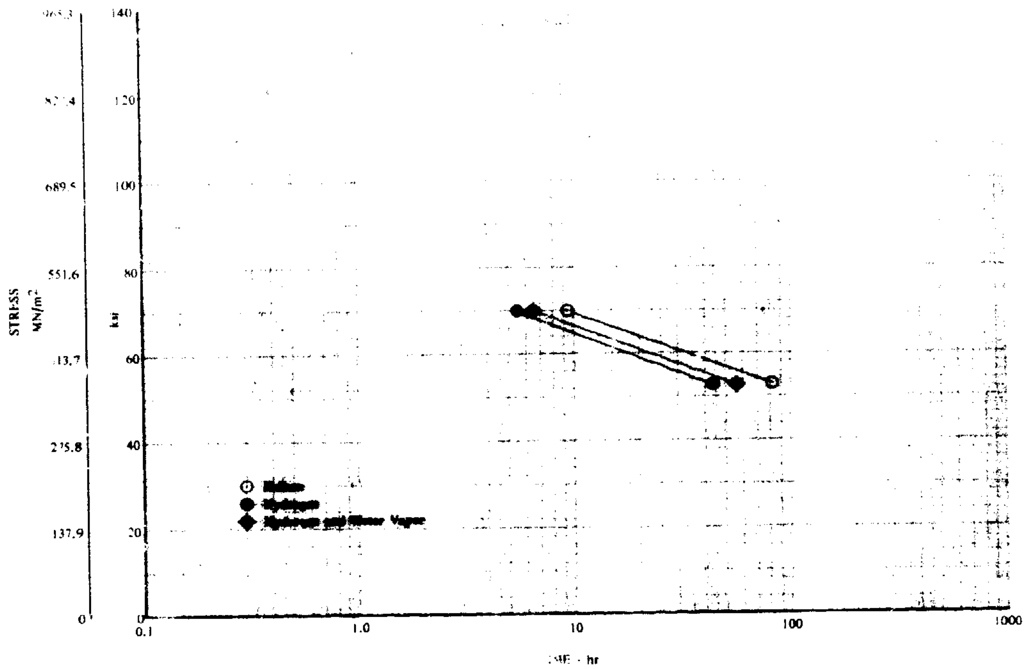


Figure VII-15. Stress-Rupture of Haynes 188 at 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure; Heat YFYR — DF 96665

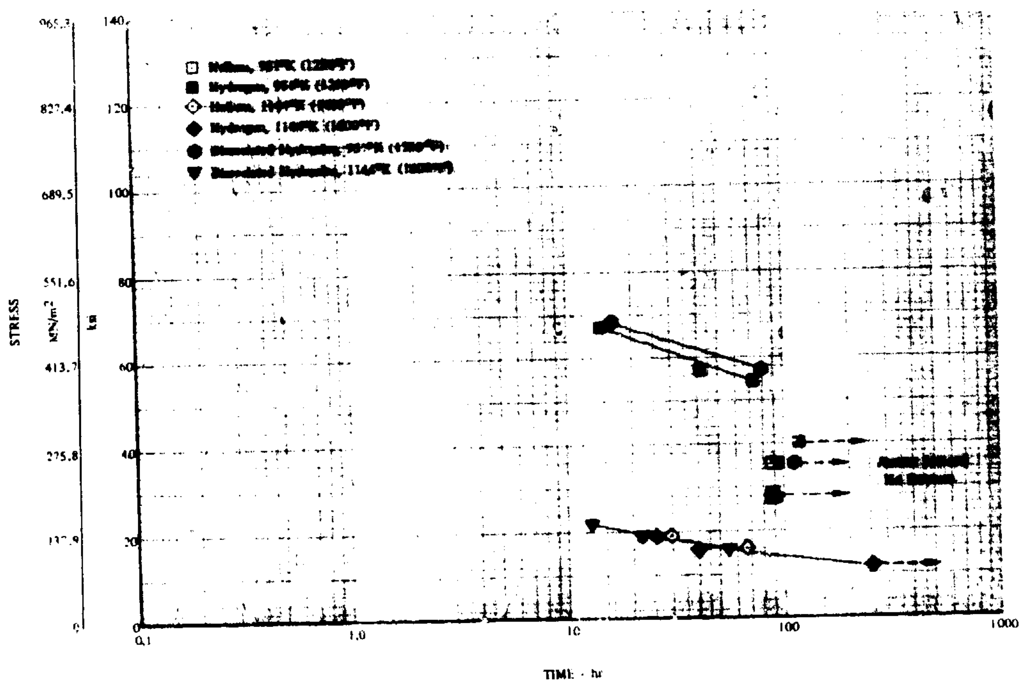


Figure VII-16. Stress-Rupture of Haynes 188 at 951 and 1144°K (1250 and 1600°F) at 3.45 MN/m² (500 psig) Pressure; Heat YGDM — DF 96666

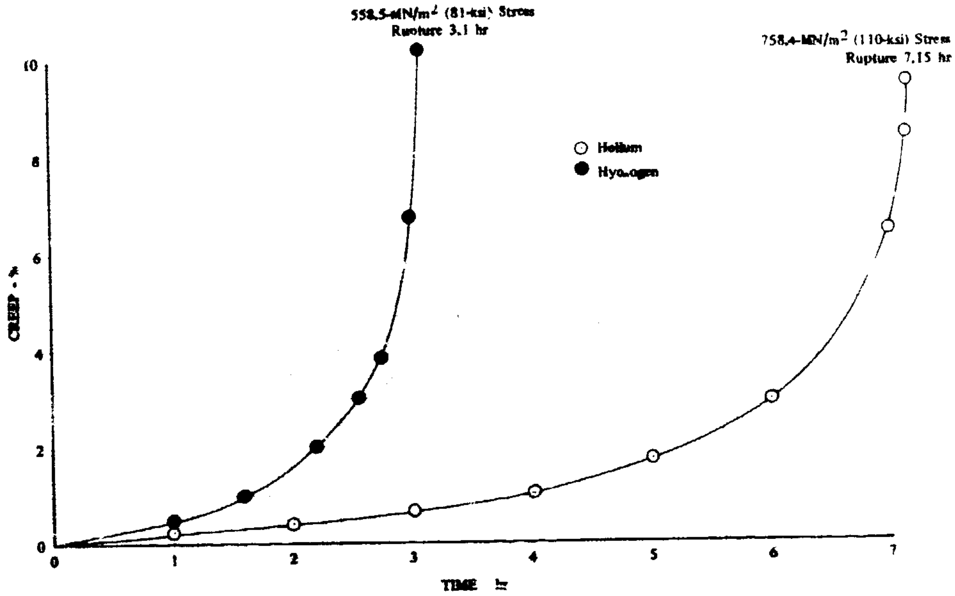


Figure VII-17. Creep/Stress-Rupture of Inconel 718, 1227°K (1750°F) Solution Plus Age Heat Treat at 951°K (1250°F), and 34.5 MN/m^2 (5000 psig) Pressure — DF 96667

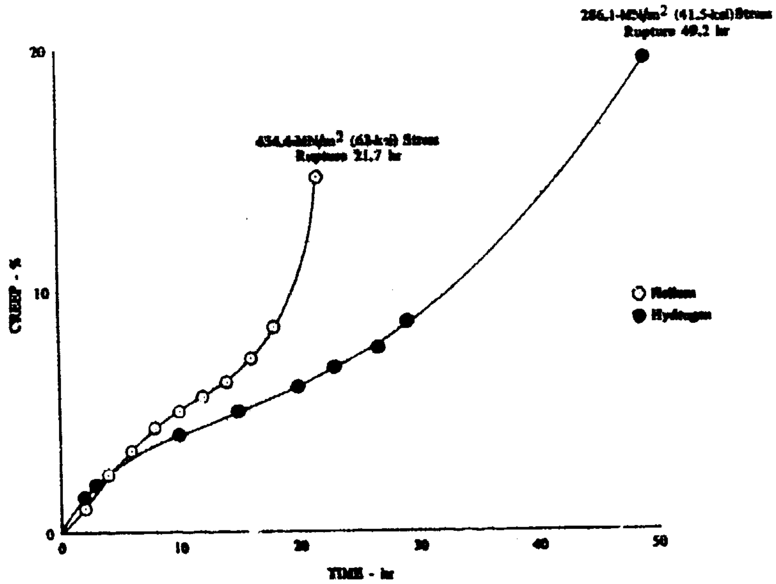


Figure VII-18. Creep/Stress-Rupture of Inconel 625 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96668

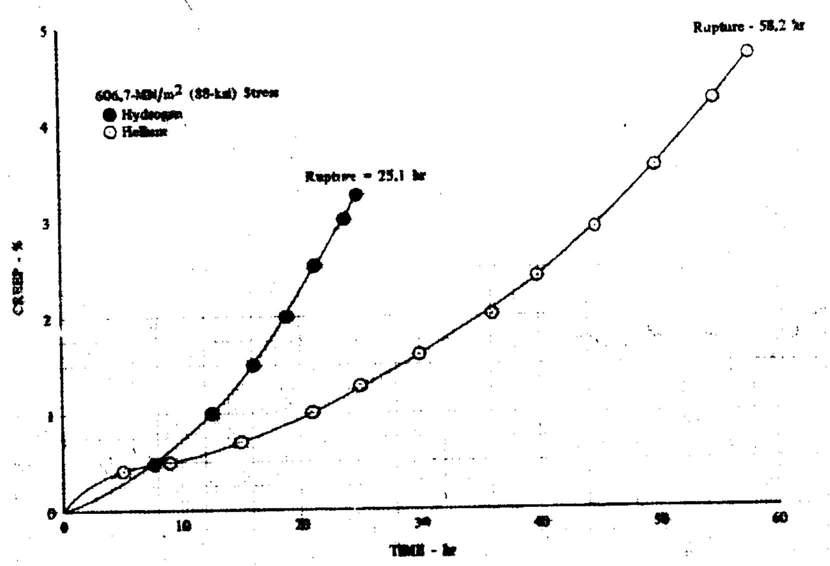


Figure VII-19. Creep/Stress-Rupture of WASPALOY® DF 96669 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure

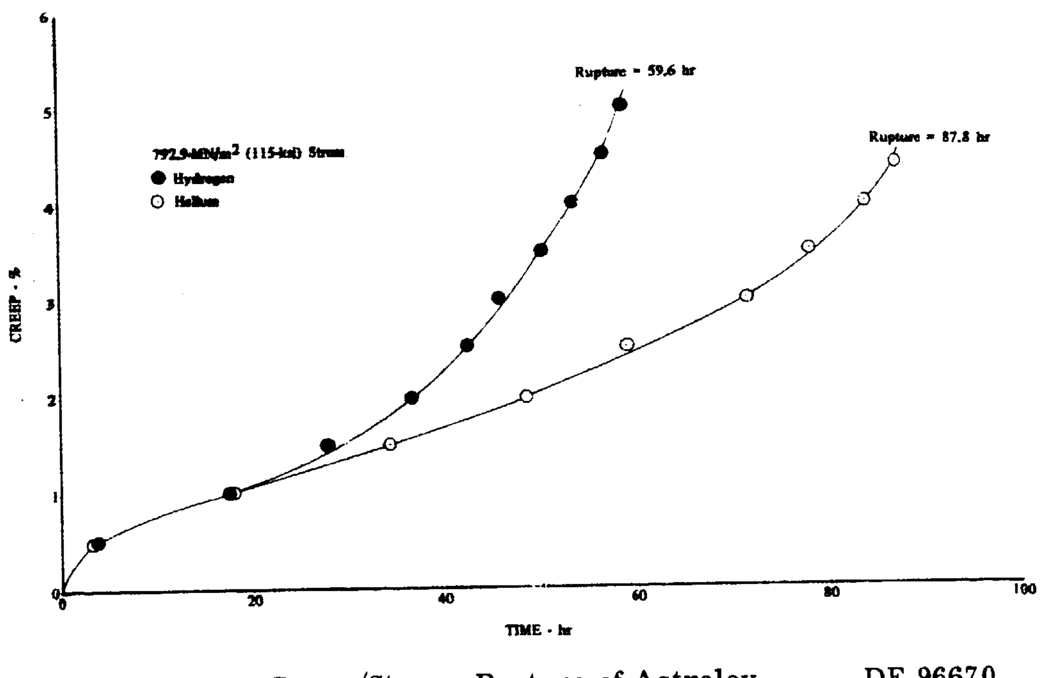


Figure VII-20. Creep/Stress-Rupture of Astroloy DF 96670 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat IKKC

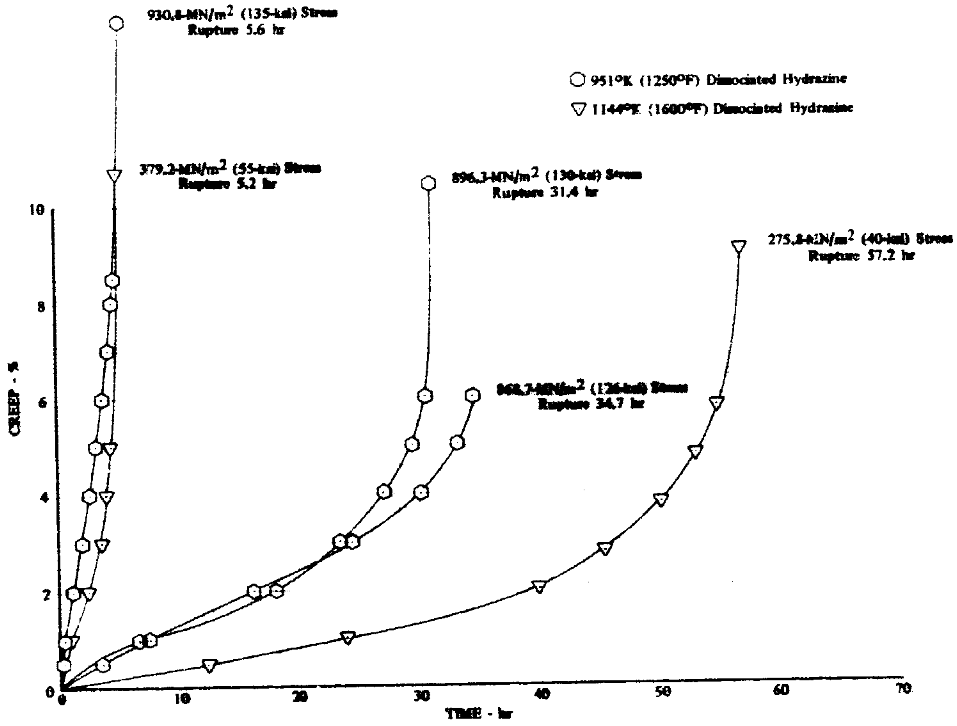


Figure VII-21. Creep/Stress-Rupture of Astroloy at 951 and 1144°K (1250 and 1600°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat BYQO DF 96671

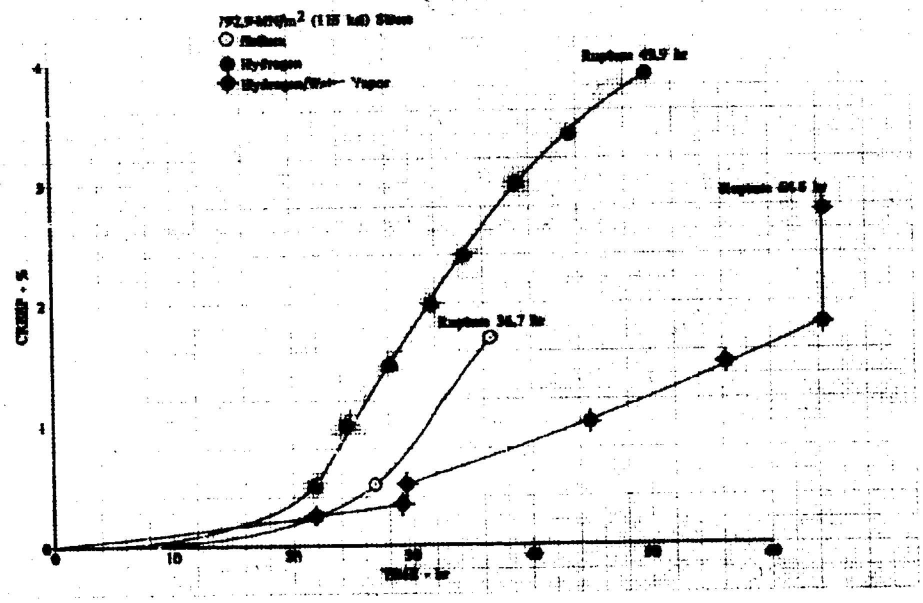


Figure VII-22. Creep/Stress-Rupture of MAR M-200 DS at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat P-9108 DF 96672

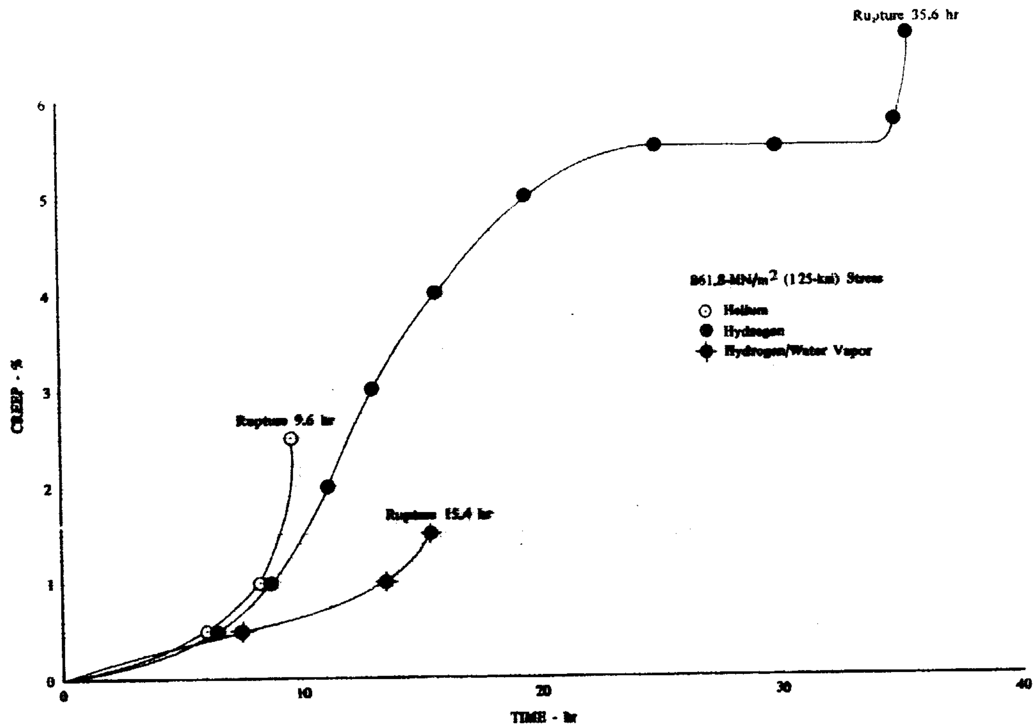


Figure VII-23. Creep/Stress-Rupture of MAR M-200 DS at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat P-9108 DF 96673

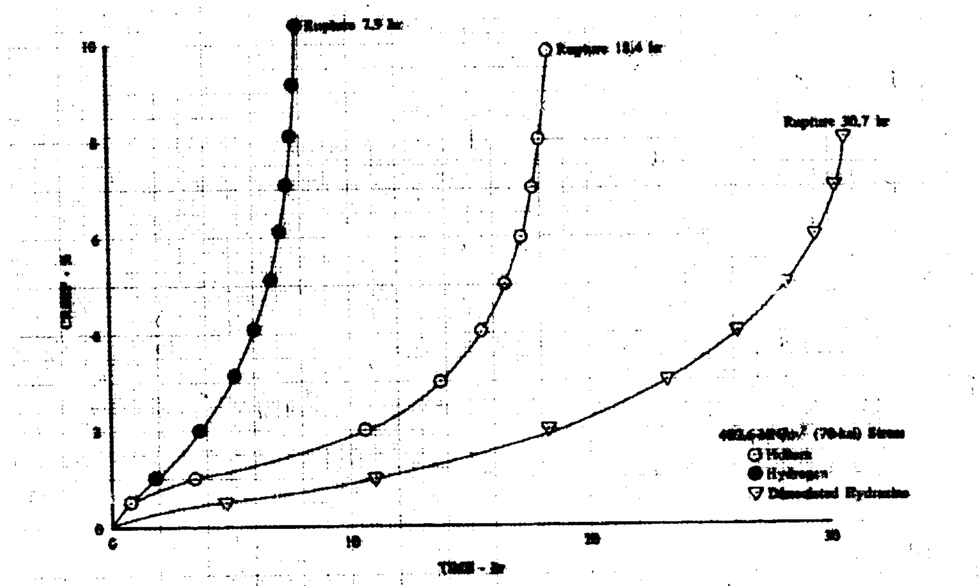


Figure VII-24. Creep/Stress-Rupture of MAR M-200 DS at 1144°K (1600°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat P-9199 DF 96674

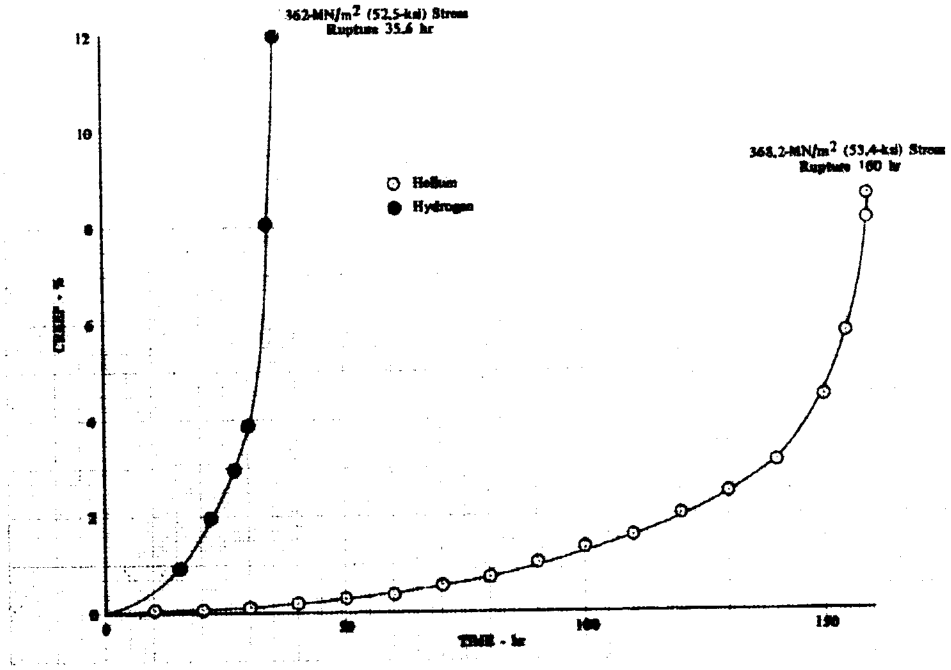


Figure VII-25. Creep/Stress-Rupture of A-286 at 951°K (1250°F) and 34.5 MN/m² (5000 psig) Pressure; Heat BZCU — DF 96675

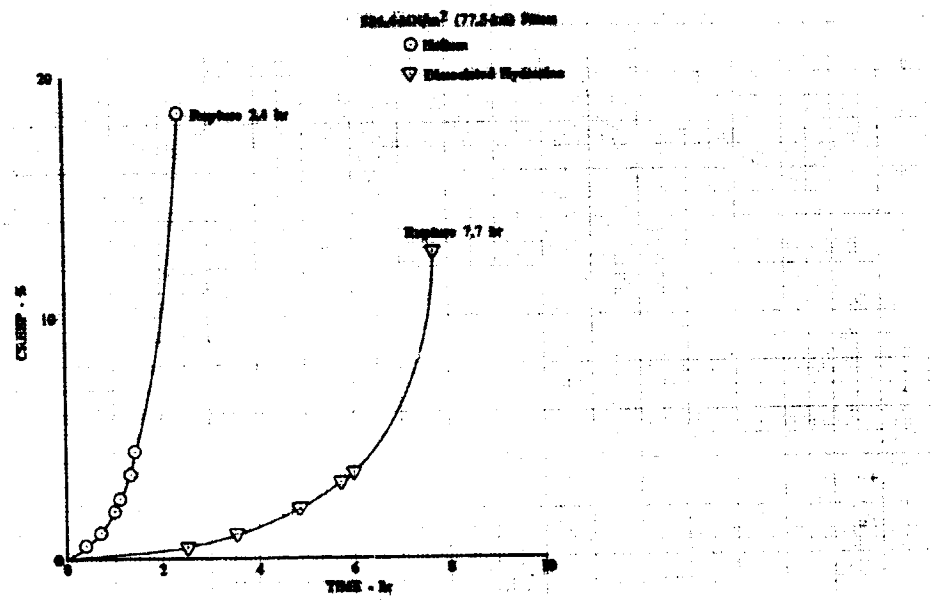


Figure VII-26. Creep/Stress-Rupture of A-286 at 951°K (1250°F) and 3.45 MN/m² (500 psig) Pressure; Heat BXOY — DF 96676

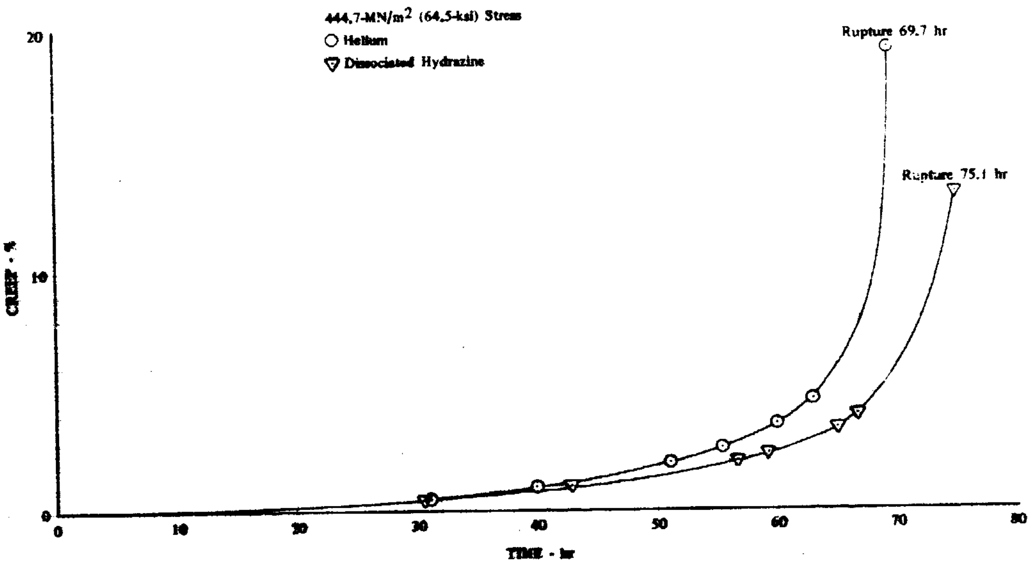


Figure VII-27. Creep/Stress-Rupture of A-286 at 951°K (1250°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat BXOY DF 96677

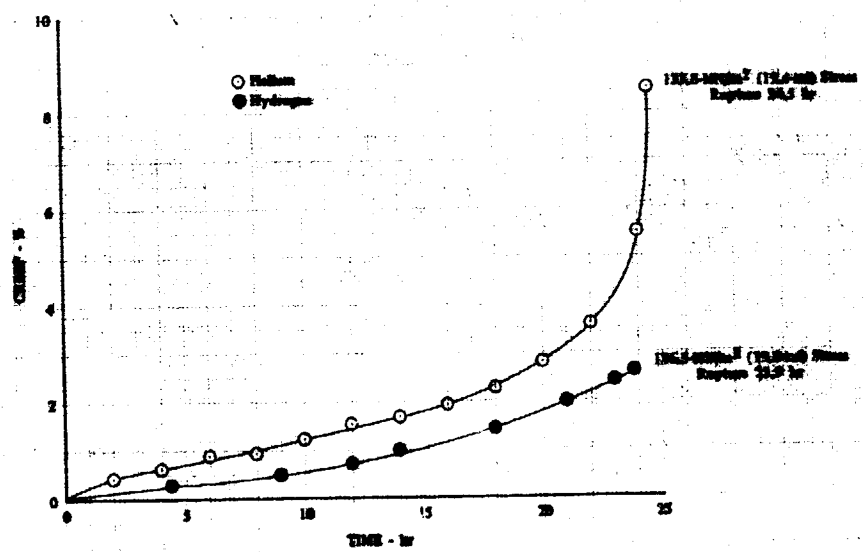


Figure VII-28. Creep/Stress-Rupture of AISI 347 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure DF 96678

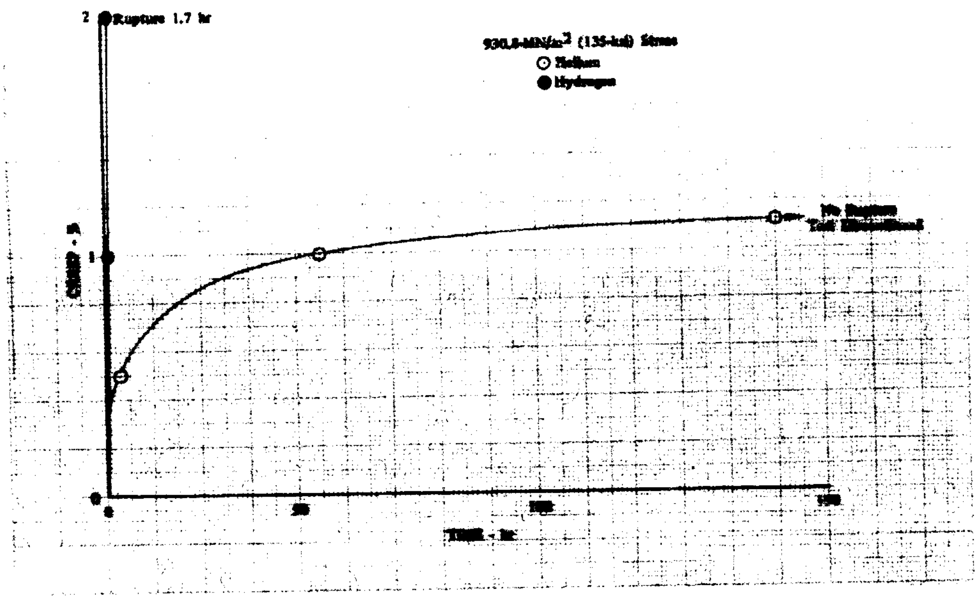


Figure VII-29. Creep/Stress-Rupture of Titanium 6-4 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96679

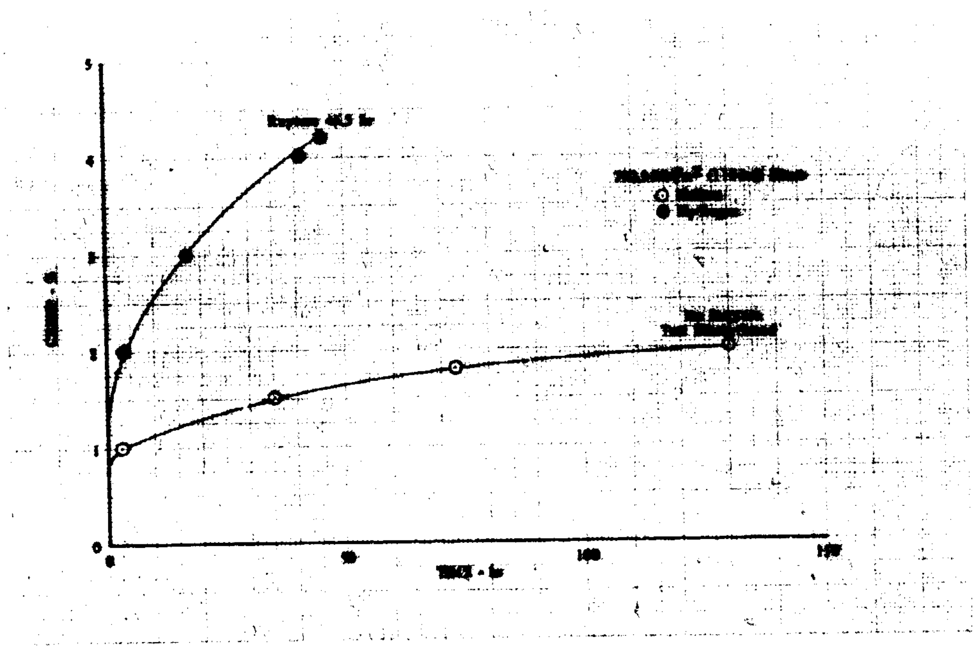


Figure VII-30. Creep/Stress-Rupture of Titanium A-110 at 366°K (200°F) and 34.5 MN/m^2 (5000 psig) Pressure — DF 96680

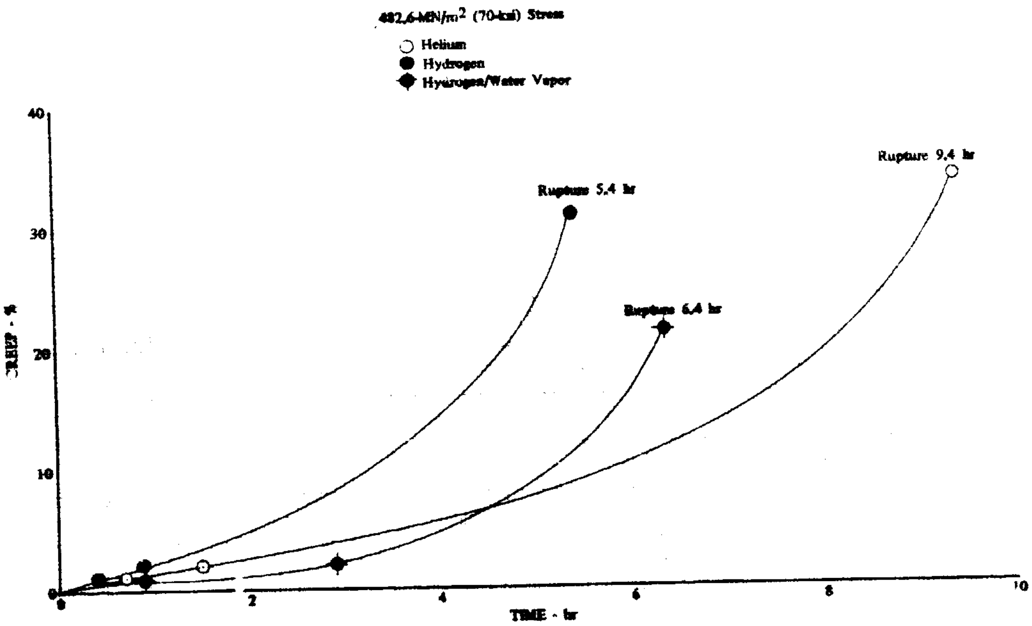


Figure VII-31. Creep/Stress-Rupture of Haynes 188 at 951°K (1250°F) and 34.5 MN/m^2 (5000 psig) Pressure; Heat YFYR DF 96681

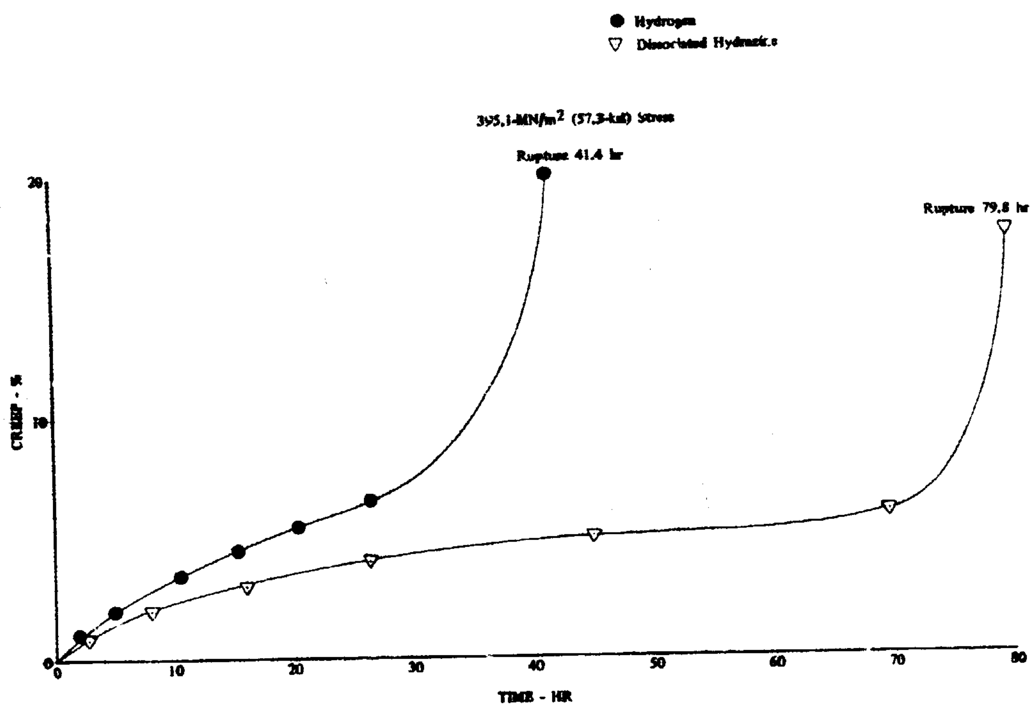


Figure VII-32. Creep/Stress-Rupture of Haynes 188 at 951°K (1250°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat YGDM DF 96682

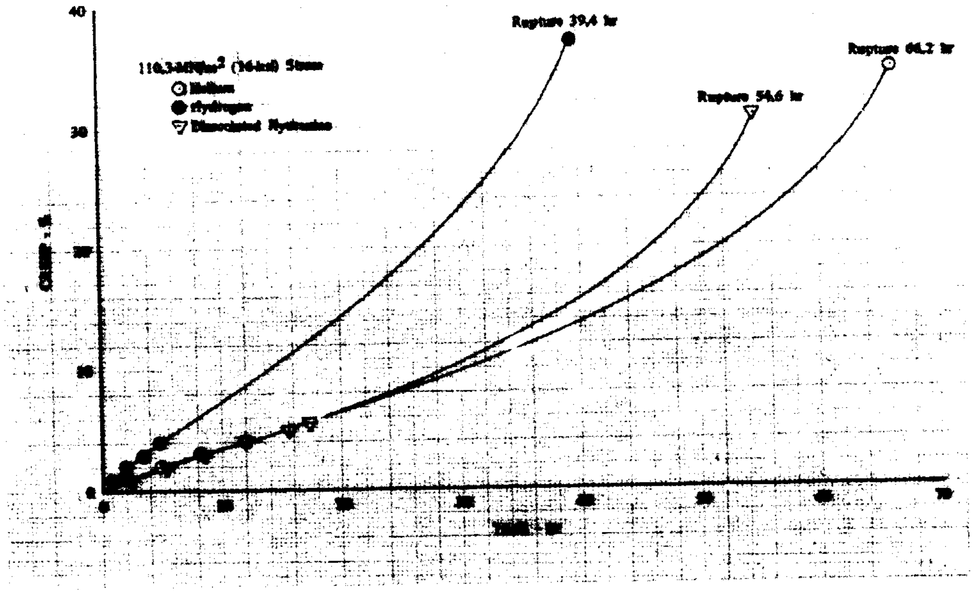


Figure VII-33. Creep/Stress-Rupture of Haynes 188 at 1144°K (1600°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat YGDM DF 96683

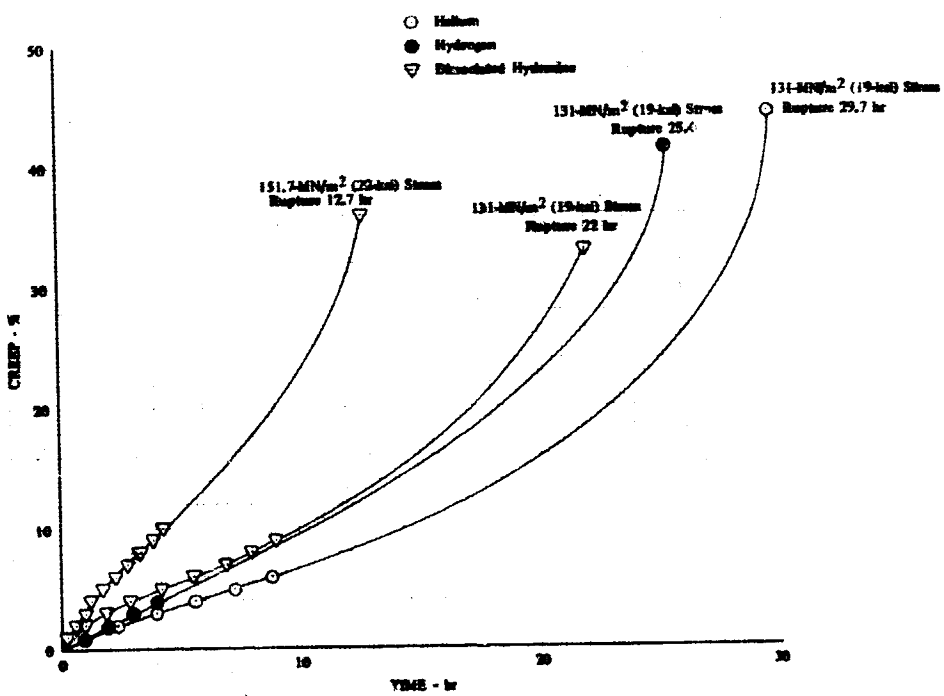


Figure VII-34. Creep/Stress-Rupture of Haynes 188 at 1144°K (1600°F) and 3.45 MN/m^2 (500 psig) Pressure; Heat YGDM DF 96684

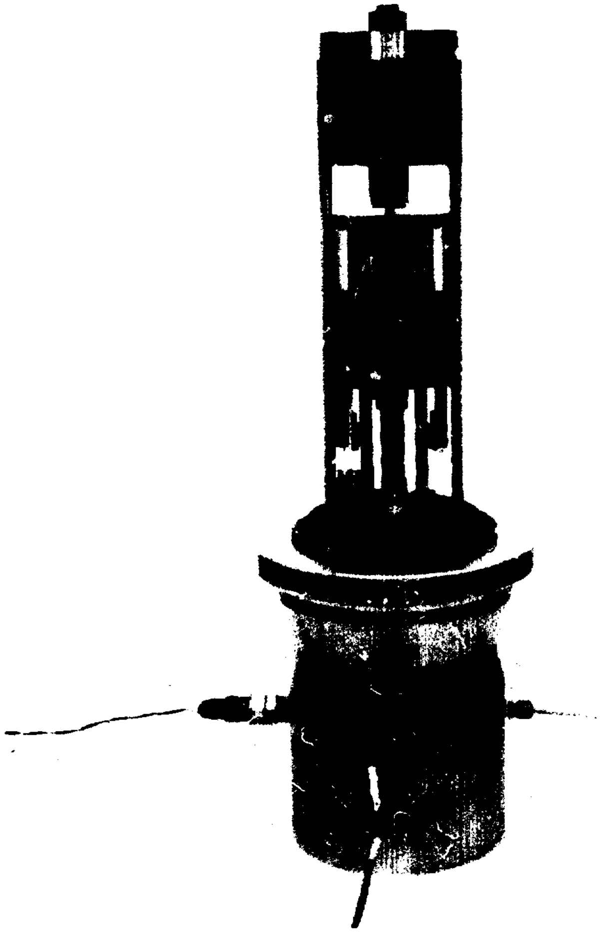


Figure VII-35. Creep-Rupture Pressure Vessel With Chamber Wall Removed. Specimen, Extensometer, Universal Pin Joints, and Half of Oven in Place — FC 21869

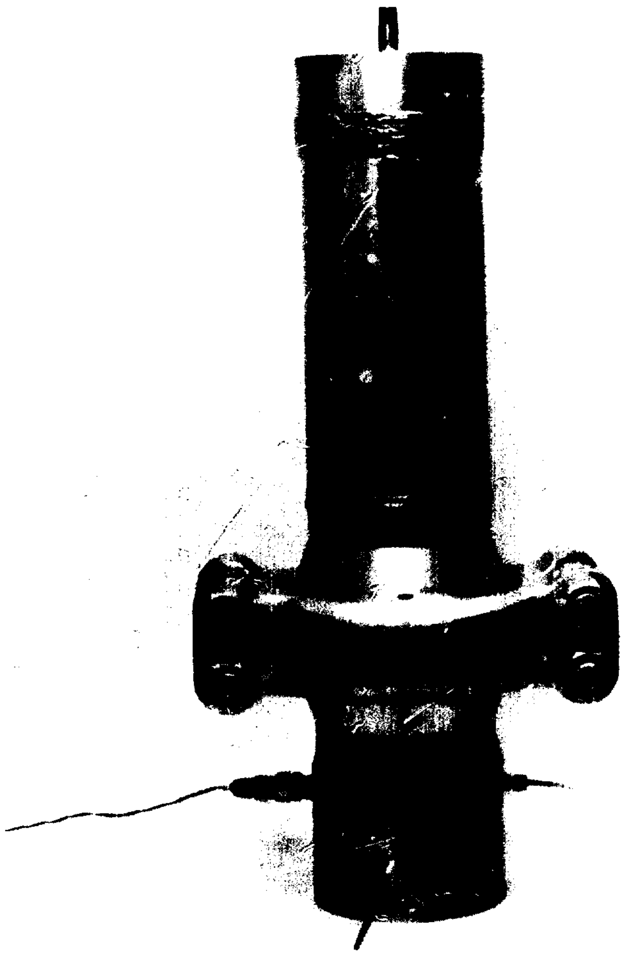


Figure VII-36. Creep-Rupture Pressure Vessel Complete Assembly — FC 21867

SECTION VIII
TENSILE PROPERTIES

SECTION VIII
TENSILE PROPERTIES

A. INTRODUCTION

Tensile properties of 12 alloys were investigated at pressures of 3.45 or 34.5 MN/m^2 (500 or 5000 psig) and at temperatures ranging from 111 to 1144°K (-260 to 1600°F). Nickel-, iron-, titanium-, and cobalt-base alloys were tested in the parent condition, and two nickel-base alloys (Inconel 718 and Inconel 625) were tested in the welded condition. Smooth tensile tests established the 0.2% yield and ultimate strengths, elongation, and reduction of area. The notched (K_T = 8.0) tensile tests established ultimate strength. Results of tests in hydrogen and/or hydrogen and water vapor environments were compared to those in helium to determine property degradation.

B. RESULTS AND CONCLUSIONS

The tensile test data were subjected to a statistical analysis to determine if the measured mechanical properties of the various alloys reflected true environmental degradation or simply data scatter. The results of these analyses were used as the basis for the conclusions herein. In some cases, the experimental test matrix did not have sufficient tests to enable determination of degradation on a statistical basis. This does not mean that there was no degradation; only that it could not be established from a statistical approach.

The individual tensile properties (yield, ultimate, elongation, reduction of area, and notch tensile ultimate) of the alloys tested did not reflect the influence of the hydrogen environments to the same degree. The relative degree of environmental degradation is summarized in table VIII-1. None of the alloys tested exhibited degradation in the 0.2% yield strength, with the possible exception of MAR M-200 DS at 300°K (80°F). Based upon the limited tests conducted, a degradation of approximately 11% in 0.2% yield strength in the hydrogen environment was indicated. The statistical analysis could not attach significance to this degradation due to the small sample size.

The ultimate strength, either smooth or notched, was degraded at one of the test conditions for seven of the alloys. Only the two cast nickel-base materials, IN-100 at 300°K (80°F) and MAR M-200 DS at both 300 and 951°K (80 and 1250°F), exhibited smooth tensile strength degradation. The titanium alloys, Titanium 6-4 and Titanium A-110, and the nickel alloys, Inconel 718 (1227°K solution), Inconel 718 welded (1313°K solution), and Hastelloy X, were degraded in notch ultimate strength for at least one of the temperature/pressure combinations tested. Astroloy and MAR M-200 DS notch tensile strengths were degraded by the hydrogen and water vapor environment at 1144°K (1600°F). The yield and ultimate strengths of the iron-base alloys A-286 and AISI 347 (figure VIII-1) and the ultimate strength of the cobalt-base alloy Haynes 188 (figure VIII-2) were not degraded.

Table VIII-1. Degradation of Tensile Properties of Materials in Gaseous Hydrogen and Hydrogen-Water Vapor Environments

Material	Stress Concentration Factor	Temperature, °K	Temperature, °F	Pressure, MN/m^2	Pressure, psig	Degradation (Change From Helium, %), in. Ultimate Strength	Degradation El	Degradation RA	Ratio of Ultimate Strength $\frac{Hydrogen}{Helium}$	Ratio of Ultimate Strength $\frac{Hydrogen - H_2O}{Helium}$	Ratio of Notch Smooth Ultimate Strength Helium	Ratio of Notch Smooth Ultimate Strength Hydrogen
Inconel 718	Smooth	300	80	34.5	5000	ND[1]	76	67	0.91		1.26	0.74
1227°K (1750°F)	8.0	300	80	34.5	5000	47			0.53			
Solution - Age	Smooth	951	1250	34.5	5000	ND	ND	ND	0.99		1.33	1.27
	8.0	951	1250	34.5	5000	ND			0.94			
Inconel 718	Smooth	300	80	34.5	5000	ND	13	24	0.99		1.61	1.54
1313°K (1900°F)	8.0	300	80	34.5	5000	ND			0.94			
Solution - Age	Smooth	951	1250	34.5	5000	ND	ND	ND	1.0		1.29	1.17
	8.0	951	1250	34.5	5000	ND			0.92			
Inconel 718 Welds 1313°K (1900°F) Solution - Age	Smooth	300	80	34.5	5000	ND	38	11	0.97		1.03	0.75
	8.0	300	80	34.5	5000	29			0.71			
Inconel 625	Smooth	300	80	34.5	5000	ND	51	52	0.97		1.25	1.26
	8.0	300	80	34.5	5000	ND			0.98			
	Smooth	951	1250	34.5	5000	ND	ND	12	1.0		1.20	1.19
	8.0	951	1250	34.5	5000	ND			1.0			
Inconel 625 Welds	Smooth	300	80	34.5	5000	ND	ND	ND	1.0		1.33	1.15
	8.0	300	80	34.5	5000	ND			0.90			
Hastelloy X	Smooth	300	80	34.5	5000	ND	ND	ND	1.0		1.39	1.20
	8.0	300	80	34.5	5000	13			0.87			
	Smooth	951	1250	34.5	5000	ND	ND	ND	1.0		1.14	1.12
	8.0	951	1250	34.5	5000	ND			0.99			
WASPALOY*	Smooth	951	1250	34.5	5000	ND	18	ND	1.0			
Astroloy	Smooth	951	1250	3.45	500	ND	ND	ND	1.0		1.25	1.22
	Smooth	1144	1600	3.45	500	ND	ND	ND	1.1		1.72	1.60
	8.0	300	80	3.45	500	ND			0.95			
	8.0	951	1250	3.45	500	ND			0.97			
	8.0	1144	1600	3.45	500	ND			1.0			
	8.0	300	80	34.5	5000	ND			0.99			
	8.0	951	1250	34.5	5000	ND			0.91			
	8.0	1144	1600	34.5	5000	ND			1.1			
(2)	8.0	951	1250	3.45	500	ND				0.94		
(2)	8.0	1144	1600	3.45	500	17				0.83		

Table VIII-1. Degradation of Tensile Properties of Materials in Gaseous Hydrogen and Hydrogen-Water Vapor Environments (Continued)

Material	Stress Concentration Factor	Temperature, °K	Temperature, °F	Pressure, MN/m²	Pressure, psig	Degradation (Change From Helium, %), in: Ultimate Strength	Degradation: El	Degradation: RA	Ratio of Ultimate Strength: Hydrogen/Helium	Ratio of Ultimate Strength: Hydrogen + H_2O/Helium	Ratio of Notch Smooth Ultimate Strength: Helium	Ratio of Notch Smooth Ultimate Strength: Hydrogen
IN-100	Smooth	300	80	34.5	5000	18	71	70	0.82			
	Smooth	951	1250	34.5	5000	ND	13	32	1.0			
MAR M-200DS	Smooth	300	80	34.5	5000	24	75	54	0.75		0.95	1.03
	Smooth	951	1250	34.5	5000	14	47	47	0.86			
	8.0	951	1250	34.5	5000	ND			0.93			
	8.0	1144	1600	3.45	500	ND			1.0			
(2)	8.0	951	1250	34.5	5000	22				0.78		
(2)	8.0	1144	1600	3.45	500	18				0.82		
A-286	Smooth	300	80	34.5	5000	ND	ND	ND	1.0		1.48	1.54
	8.0	300	80	34.5	5000	ND			1.0			
	Smooth	951	1250	34.5	5000	ND	14	15	0.94		1.44	1.59
	8.0	951	1250	34.5	5000	ND			1.0			
AISI 347	Smooth	111	-260	34.5	5000	ND	ND	ND	0.92		1.15	1.22
	8.0	111	-260	34.5	5000	ND			0.98			
	Smooth	300	80	34.5	5000	ND	ND	ND	1.0		1.70	1.43
	8.0	300	80	34.5	5000	ND			0.91			
	Smooth	951	1250	34.5	5000	ND	ND	ND	1.0		1.63	1.72
	8.0	951	1250	34.5	5000	ND			0.97			
Titanium 6-4	Smooth	300	80	34.5	5000	ND	10	20	1.0		1.37	1.23
	8.0	300	80	34.5	5000	11			0.89			
	Smooth	366	200	34.5	5000	ND	27	42	0.97		1.38	1.32
	8.0	366	200	34.5	5000	ND			0.93			
A-110	Smooth	300	80	34.5	5000	ND	20	25	1.0		1.51	1.21
	8.0	300	80	34.5	5000	17			0.83			
	Smooth	366	200	34.5	5000	ND	18	43	0.98		1.44	1.31
	8.0	366	200	34.5	5000	11			0.90			
Haynes 188	8.0	300	80	3.45	500	ND			0.98			
	8.0	951	1250	3.45	500	ND			1.0			
	8.0	1144	1600	3.45	500	ND			1.0			
(2)	8.0	951	1250	3.45	500	ND				1.0		
(2)	8.0	1144	1600	3.45	500	ND				1.0		

(1) ND - Negligible Degradation, Less Than 10%.

(2) Hydrogen-Water Vapor Environment.

0.2% Yield Strength Did Not Reflect Distinct Property Reduction.

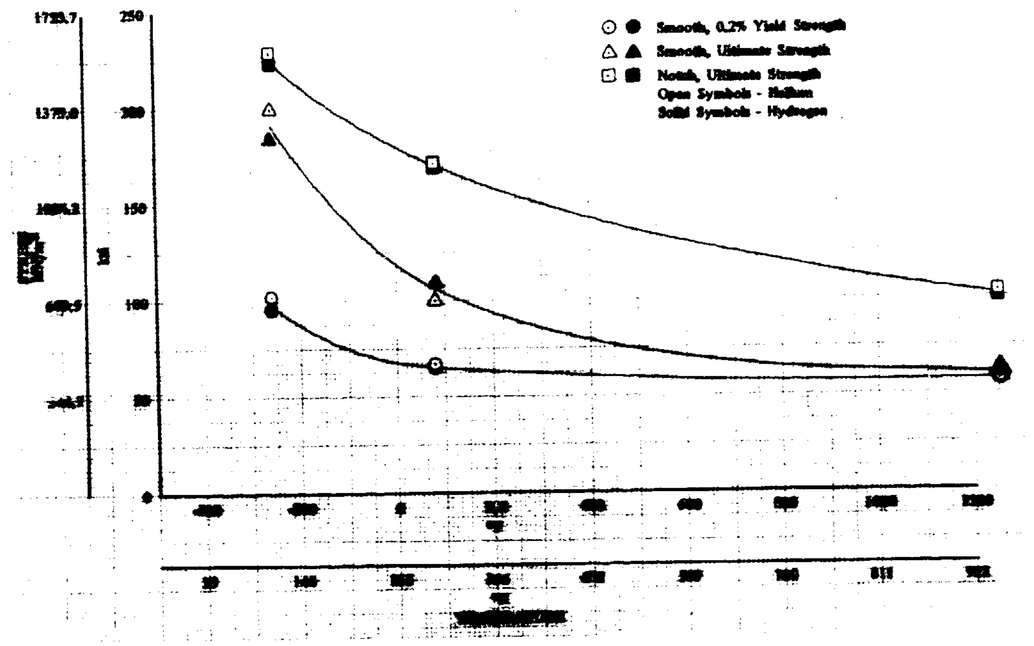


Figure VIII-1. Effect of Temperature and Environment Upon Tensile Strength of AISI 347 at 34.5 MN/m^2 (5000 psig) Pressure DF 96691

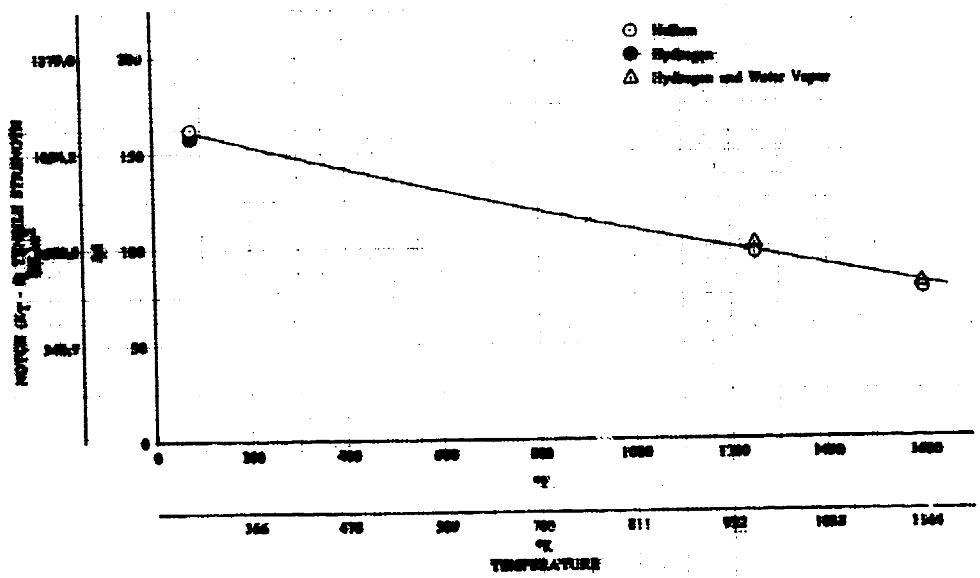


Figure VIII-2. Effect of Temperature and Environment Upon Notch (K_T = 8.0) Strength of Haynes 188 at 3.45 MN/m^2 (500 psig) Pressure DF 96692

The tensile properties of Astroloy were the most thoroughly investigated, with smooth and notch strengths determined at three temperatures and notch strength at two pressures. The effect of temperature upon smooth tensile strength of Astroloy at 3.45 MN/m^2 (500 psig) is shown in figure VIII-3, with no evidence of hydrogen degradation. The notched tensile strength was degraded at room temperature and 951°K (1250°F), but not at 1144°K (1600°F), in both 3.45- and 34.5-MN/m^2 (500- and 5000-psig) pressure hydrogen (figures VIII-4 and VIII-5).[1]

The hydrogen and water vapor environment at 3.45 MN/m^2 (500 psig) degraded the notch strength at both 951 and 1144°K (1250 and 1600°F). The effects of temperature, pressure, and environment upon degree of degradation of Astroloy are shown in figure VIII-6. While the degradation at both pressures is approximately 10% or less and is classified as ND (negligible degradation), there is a definite effect of pressure upon degradation at temperatures below 977°K (1300°F). The strength is degraded approximately twice as much at 35.4 MN/m^2 (5000 psig) as at 3.45 MN/m^2 (500 psig). The reversal in properties at 1144°K (1600°F) (that is, higher strengths in hydrogen than in helium) was verified, but no specific reason for this reversal could be established (Reference FR-5129).

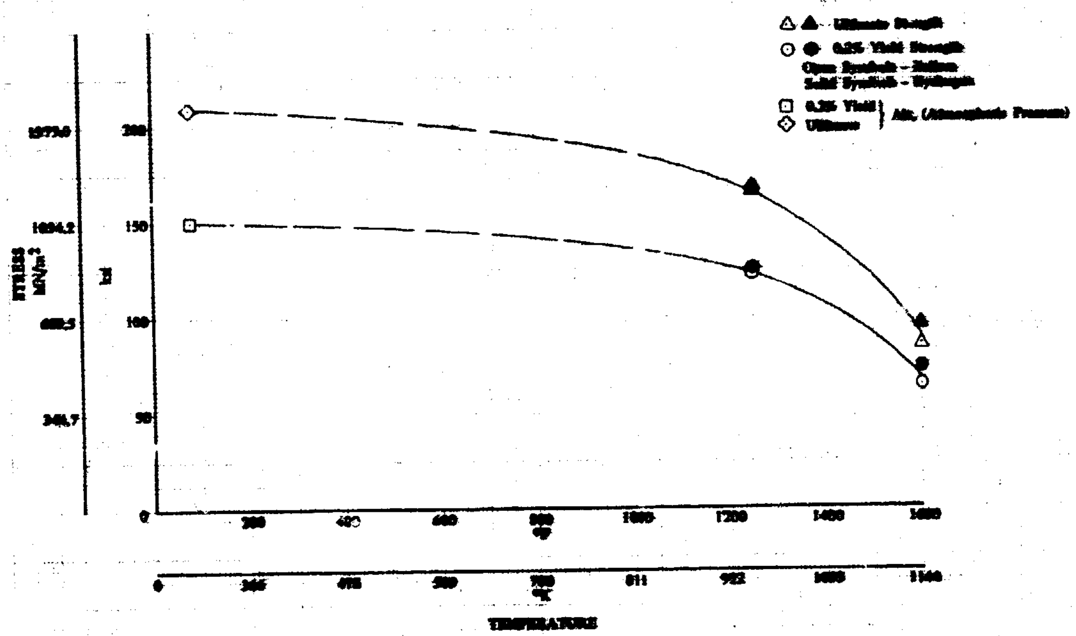


Figure VIII-3. Effect of Temperature and Environment Upon Smooth Tensile Strength of Astroloy at 3.45 MN/m^2 (500 psig) Pressure DF 96693

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(1) Astroloy tests at 1144°K and 34.5 MN/m^2 (1600°F and 5000 psig) were attempted on a best effort basis. The maximum hydrogen pressure at which a temperature of 1144°K (1600°F) could be maintained with the tensile test pressure vessel and furnace was 30.3 MN/m^2 (4400 psig).

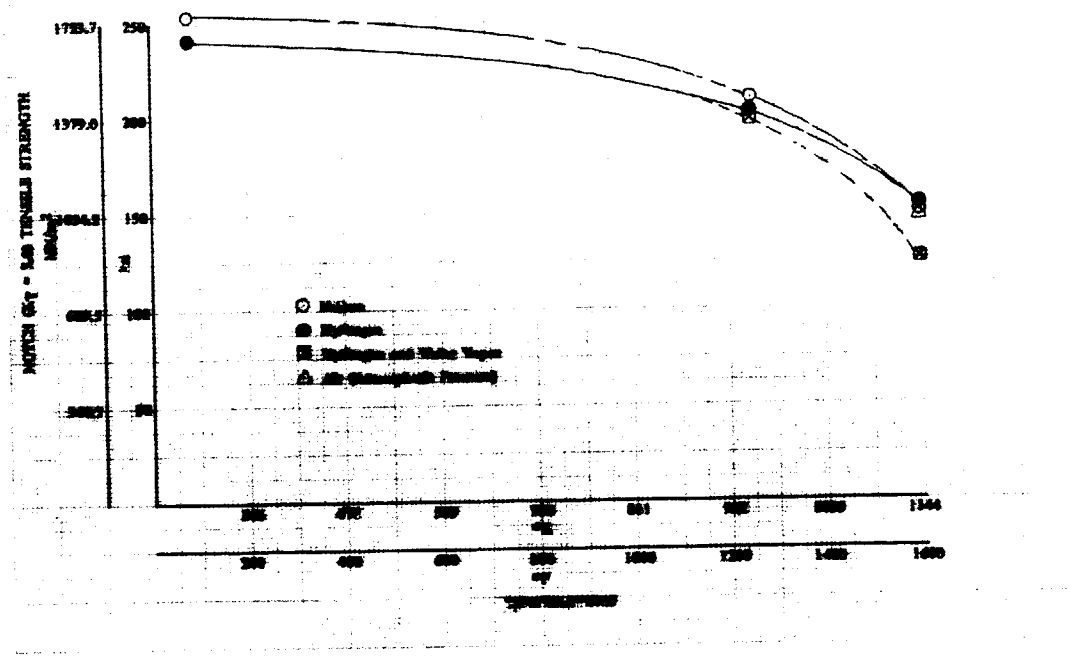


Figure VIII-4. Effect of Temperature and Environment Upon Notch (K_T = 8.0) Strength of Astroloy at 3.45 MN/m^2 (500 psig) Pressure DF 96694

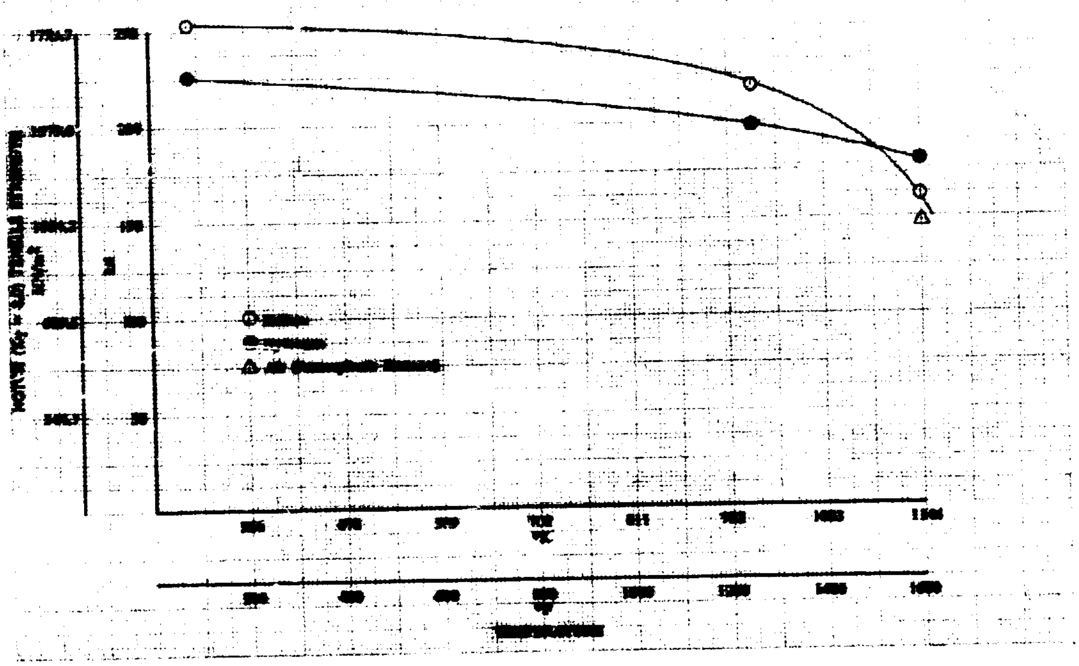


Figure VIII-5. Effect of Temperature and Environment Upon Notch (K_T = 8.0) Strength of Astroloy at 34.5 MN/m^2 (5000 psig) Pressure DF 96655

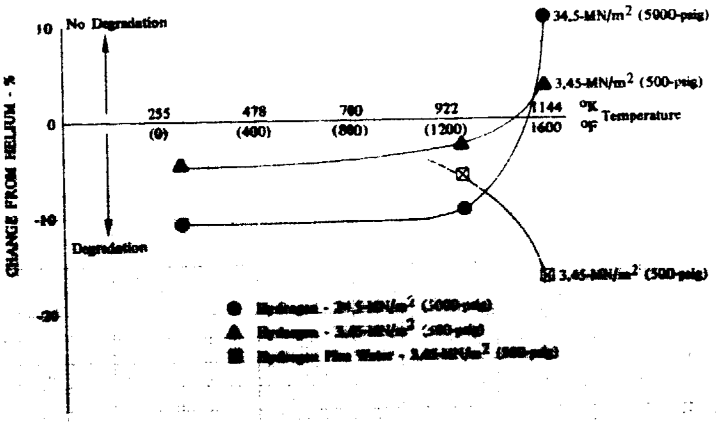


Figure VIII-6. Effect of Temperature and Pressure Upon Environmental Degradation of Astroloy Notch (K_T = 8.0) Tensile Strength

DF 96656

The increase in degradation in the hydrogen and water vapor environment at 1144°K (1600°F) is attributed primarily to the occurrence of oxides on the metal. At 1144°K (1600°F), the water vapor dissociates at the specimen surface into hydrogen and oxygen. The oxygen immediately reacts with the metal to form oxides, and the hydrogen remains in the environment. The occurrence of oxides on the specimen surfaces was also noted for the MAR M-200 DS and Haynes 188 tests in the 1144°K (1600°F) hydrogen and water vapor environment. In the case of Haynes 188, however, these oxides did not contribute to a property degradation.

The most prominent and consistent indicator of hydrogen degradation in the tensile tests was the loss of ductility for smooth specimens. In the case of some alloys, ductility was extremely degraded, while the ultimate strength showed negligible degradation. The elongation and reduction of area were degraded for most of the nickel-base alloys, with IN-100 and MAR M-200 DS (both cast alloys) and Inconel 718 (1227°K [1750°F] solution plus age) the most severely degraded. Generally, ductility was affected at 300°K (80°F) more than at the elevated temperatures. Of all the materials evaluated, only the iron-base alloy, AISI 347, appeared completely immune to degradation in any smooth or notch tensile property for the conditions tested. The cobalt-base alloy, Haynes 188, was not tested for smooth tensile properties, but was not degraded in notch strength at any of the conditions tested.

Complete test results are listed in table VIII-2.

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment

Material	Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	Test Conditions MN/m²	Pressure, psig	Yield Strength MN/m²	Yield, ksi	Ultimate Strength MN/m²	Ultimate, ksi	Ductility El,(1) %	Ductility RA,(2) %
Inconel 718	300	80	Smooth	Helium	34.5	5000	1094.2	158.7	1352.8	196.2	23.5	35.8
1227°K (1750°F)	300	80	Smooth	Helium	34.5	5000	1111.4	161.2	1387.2	201.2	16.0	17.5
Solution + Age	300	80	Smooth	Helium	34.5	5000	1112.1	161.3	1389.3	201.5	23.5	35.3
	300	80	8.0	Helium	34.5	5000			1752.7	254.2		
	300	80	8.0	Helium	34.5	5000			1707.8	247.7		
	300	80	Smooth	Hydrogen	34.5	5000	1041.8	151.1	1241.7	180.1	5.5	9.4
	300	80	Smooth	Hydrogen	34.5	5000	1083.9	157.2	1254.9	182.0	4.5	10.9
	300	80	8.0	Hydrogen	34.5	5000			786.7	114.1		
	300	80	8.0	Hydrogen	34.5	5000			1063.2	154.2		
	300	80	3.0	Hydrogen	34.5	5000			921.1	133.6		
	951	1250	Smooth	Helium	34.5	5000	942.5	136.7	1074.9	155.9	20.0	19.7
	951	1250	Smooth	Helium	34.5	5000	905.3	131.3	1063.9	154.3	25.0	47.5
	951	1250	8.0	Helium	34.5	5000			1396.2	202.5		
	951	1250	8.0	Helium	34.5	5000			1451.4	210.5		
	951	1250	Smooth	Hydrogen	34.5	5000	948.0	137.5	1036.3	150.3	22.0	46.8
	951	1250	Smooth	Hydrogen	34.5	5000	937.7	136.0	1072.1	155.5	21.5	34.9
	951	1250	8.0	Hydrogen	34.5	5000			1292.8	187.5		
	951	1250	8.0	Hydrogen	34.5	500			1354.8	196.5		
	951	1250	8.0	Hydrogen	34.5	5000			1372.8	199.1		
Inconel 718	300	80	Smooth	Helium	34.5	5000	1083.9	157.2	1306.6	189.5	26.0	51.3
1313°K (1900°F)	300	80	Smooth	Helium	34.5	5000	1072.8	155.6	1283.8	186.2	25.5	49.9
Solution + Age	300	80	8.0	Helium	34.5	5000			2087.7	302.8		
	300	80	8.0	Helium	34.5	5000			2083.6	302.2		
	300	80	Smooth	Hydrogen	34.5	5000	1091.4	158.3	1283.8	186.2	21.5	40.3
	300	80	Smooth	Hydrogen	34.5	5000	1078.3	156.4	1279.7	185.6	23.5	36.6
	300	80	8.0	Hydrogen	34.5	5000			1911.2	277.2		
	300	80	8.0	Hydrogen	34.5	5000			2009.1	291.4		
	300	30	8.0	Hydrogen	34.5	5000			1931.2	288.1		
	951	1250	Smooth	Helium	34.5	5000	930.1	134.9	1036.3	150.3	17.5	31.8
	951	1250	Smooth	Helium	34.5	5000	974.2	141.3	1037.0	150.4	11.5	18.0
	951	1250	8.0	Helium	34.5	5000			1310.0	190.0		
	951	1250	8.0	Helium	34.5	5000			1355.5	196.6		
	951	1250	Smooth	Hydrogen	34.5	5000	919.1	133.3	1036.3	150.3	17.0	27.0
	951	1250	Smooth	Hydrogen	34.5	5000	927.4	134.5	1046.0	151.8	15.5	19.6
	951	1250	6.0	Hydrogen	34.5	5000			1214.9	176.2		
	951	1250	8.0	Hydrogen	34.5	5000			1204.5	174.7		
	951	1250	8.0	Hydrogen	34.5	5000			1246.6	180.8		
Inconel 718	300	80	Smooth	Helium	34.5	5000	1108.0	160.7	1212.8	175.9	2.5	7.8
Welds 1313°K	300	80	Smooth	Helium	34.5	5000	1152.1	167.1	1308.6	189.8	12.0	18.8
(1900°F) Solution	300	80	8.0	Helium	34.5	5000			1099.0	159.4		
+ Age	300	80	8.0	Helium	34.5	5000			1494.1	216.7		
	300	80	Smooth	Hydrogen	34.5	5000	1113.5	161.5	1264.5	183.4	6.0	15.2
	300	80	Smooth	Hydrogen	34.5	5000	1123.2	162.9	1186.6	172.1	3.0	8.6
	300	80	8.0	Hydrogen	34.5	5000			954.2	138.4		
	300	80	8.0	Hydrogen	34.5	5000			1176.9	170.7		
	300	80	8.0	Hydrogen	34.5	5000			624.0	90.5		
Inconel 625	300	80	Smooth	Helium	34.5	5000	635.0	92.1	1018.4	147.7	46.5	62.5
	300	80	Smooth	Helium	34.5	5000	522.6	75.8	985.3	142.9	48.0	61.5
	300	80	8.0	Helium	34.5	5000			1192.8	173.0		
	300	80	8.0	Helium	34.5	5000			1311.4	190.2		
	300	80	Smooth	Hydrogen	34.5	5000	704.6	102.2	968.7	140.5	23.0	30.1
	300	80	Smooth	Hydrogen	34.5	5000	684.0	99.2	981.1	142.3	23.0	30.1
	300	80	8.0	Hydrogen	34.5	5000			1208.0	175.2		
	300	80	8.0	Hydrogen	34.5	5000			1219.0	176.8		
	300	80	8.0	Hydrogen	34.5	5000			1253.5	181.8		
	951	1250	Smooth	Helium	34.5	5000	519.2	75.3	806.0	116.9	62.5	70.4
	951	1250	Smooth	Helium	34.5	5000	494.4	71.7	832.9	120.8	59.0	66.2

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	Test Conditions MN/m^2	Pressure, psig	Yield Strength MN/m^2	Yield, ksi	Ultimate Strength MN/m^2	Ultimate, ksi	Ductility El,(1) %	Ductility RA,(2) %
	951	1250	8.0	Helium	34.5	5000			1034.2	150.0		
	951	1250	8.0	Helium	34.5	5000			936.3	135.8		
	951	1250	Smooth	Hydrogen	34.5	5000	501.3	72.7	821.2	119.1	57.5	64.2
	951	1250	Smooth	Hydrogen	34.5	5000	533.6	77.4	878.4	127.4	52.5	55.9
	951	1250	8.0	Hydrogen	34.5	5000			1006.6	146.0		
	951	1250	8.0	Hydrogen	34.5	5000			1016.3	147.4		
	951	1250	8.0	Hydrogen	34.5	5000			1020.4	148.0		
Inconel 625 Welds	300	80	Smooth	Helium	34.5	5000	375.8	54.5	761.2	110.4	26.0	42.5
	300	80	Smooth	Helium	34.5	5000	363.4	52.7	710.2	103.0	22.5	33.9
	300	80	8.0	Helium	34.5	5000			1017.7	147.6		
	300	80	8.0	Helium	34.5	5000			942.5	136.7		
	300	80	Smooth	Hydrogen	34.5	5000	363.4	52.7	786.0	114.0	27.5	40.1
	300	80	Smooth	Hydrogen	34.5	5000	363.4	52.7	746.0	108.2	23.0	35.1
	300	80	8.0	Hydrogen	34.5	5000			884.6	128.3		
	300	80	8.0	Hydrogen	34.5	5000			908.0	131.7		
	300	80	8.0	Hydrogen	34.5	5000			884.6	122.5		
Hastelloy X	300	80	Smooth	Helium	34.5	5000	335.1	48.6	735.7	106.7	54.0	63.0
	300	80	Smooth	Helium	34.5	5000	307.5	44.6	710.2	103.0	53.5	62.8
	300	80	8.0	Helium	34.5	5000			880.5	127.7		
	300	80	8.0	Helium	34.5	5000			1129.4	163.8		
	300	80	Smooth	Hydrogen	34.5	5000	335.1	48.6	727.4	105.5	51.5	60.5
	300	80	Smooth	Hydrogen	34.5	5000	345.4	50.1	726.0	105.3	54.5	66.4
	300	80	8.0	Hydrogen	34.5	5000			855.0	124.0		
	300	80	8.0	Hydrogen	34.5	5000			865.3	125.5		
	300	80	8.0	Hydrogen	34.5	5000			901.8	130.8		
	951	1250	Smooth	Helium	34.5	5000	242.7	35.2	566.1	82.1	53.0	57.5
	951	1250	Smooth	Helium	34.5	5000	228.2	33.1	534.3	77.5	53.0	57.6
	951	1250	8.0	Helium	34.5	5000			621.9	90.2		
	951	1250	8.0	Helium	34.5	5000			628.8	91.2		
	951	1250	Smooth	Hydrogen	34.5	5000	233.7	33.9	557.1	80.8	51.5	58.2
	951	1250	Smooth	Hydrogen	34.5	5000	235.1	34.1	553.6	80.3	51.0	48.9
	951	1250	8.0	Hydrogen	34.5	5000			610.9	88.6		
	951	1250	8.0	Hydrogen	34.5	5000			626.0	90.8		
	951	1250	8.0	Hydrogen	34.5	5000			627.4	91.0		
WASPALOY®	951	1250	Smooth	Helium	34.5	5000	910.1	132.0	[illegible]	174.0	22.5	36.7
	951	1250	Smooth	Hydrogen	34.5	5000	930.8	135.0	1189.4	172.5	18.0	29.3
	951	1250	Smooth	Hydrogen	34.5	5000	942.5	136.7	1244.5	180.5	19.0	43.4
Astroloy Heat Code LKKC	300	80	Smooth	Air	One Atmosphere		1014.9	147.2	1434.8	208.1	20	25
	300	80	Smooth	Air	One Atmosphere		1051.5	152.5	1452.7	210.7	20	23
	300	80	8.0	Helium	3.45	500			1744.4	253.0		
	300	80	8.0	Hydrogen	3.45	500			1654.4	240.0		
	300	80	8.0	Hydrogen	3.45	500			1675.4	243.0		
	951	1250	Smooth	Helium	3.45	500	834.3	121.0	1154.9	167.5	21.0	28.7
	951	1250	8.0	Helium	3.45	500			1441.0	209.0		
	951	1250	Smooth	Hydrogen	3.45	500	844.6	122.5	1148.0	[illegible]	22.0	32.1
	951	1250	8.0	Hydrogen	3.45	500			1379.0	200.0		
	951	1250	8.0	Hydrogen	3.45	500			1427.2	207.		
	1144	1600	Smooth	Helium	3.45	500	398.5	57.8	575.7	83.5	14.0	40.1
	1144	1600	Smooth	Helium	3.45	500	465.4	67.5	610.2	88.5	21.0	49.1
	1144	1600	8.0	Helium	3.45	500			972.2	141.0		
	1144	1600	8.0	Helium	3.45	500			1027.3	149.0		
	1144	1600	6.65	Helium	3.45	500			1068.7	155.0		
	1144	1600	Smooth	Hydrogen	3.45	500	569.5	82.6	755.0	109.5	20.0	49.8
	1144	1600	Smooth	Hydrogen	3.45	500	470.9	68.3	635.0	92.1	20.0	46.3
	1144	1600	Smooth	Hydrogen	3.45	500	487.5	70.7	587.4	85.2	18.0	38.9

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	Pressure, MN/m²	Pressure, psig	Yield, MN/m²	Yield, ksi	Ultimate Strength, MN/m²	Ultimate Strength, ksi	Ductility El,(1) %	Ductility RA,(2) %
	1144	1600	8.0	Hydrogen	3.45	500			1079.0	156.5		
	1144	1600	8.0	Hydrogen	3.45	500			1106.6	160.5		
	1144	1600	8.0	Hydrogen	3.45	500			986.0	143.0		
	300	80	8.0	Helium	34.5	5000			1744.4	253.0		
	300	80	8.0	Hydrogen	34.5	5000			1554.8	225.5		
	300	80	8.0	Hydrogen	34.5	5000			1565.1	227.0		
	951	1250	8.0	Helium	34.5	5000			1516.9	220.0		
	951	1250	6.6	Helium	34.5	5000			1503.1	218.0		
	951	1250	8.0	Hydrogen	34.5	5000			1365.2	198.0		
	951	1250	8.0	Hydrogen	34.5	5000			1372.1	199.0		
	1144	1600	5.8	Air	One Atmosphere				1021.8	148.2		
	1144	1600	8.0	Air	One Atmosphere				1020.4	148.0		
	1144	1600	8.0	Helium	34.5	5000			1130.7	164.0		
	1144	1600	8.0	Helium	30.3	4400(3)			1085.9	157.5		
	1144	1600	8.0	Hydrogen	30.3	4400(3)			1185.9	172.0		
	1144	1600	8.0	Hydrogen	30.3	4400(3)			1289.3	187.[illegible]		
Astroloy Heat Code BYQO	951	1250	8.0	Hydrogen and Water Vapor	3.45	500			1323.8	192.0		
	951	1250	8.0	Hydrogen and Water Vapor	3.45	500			1372.1	199.0		
	951	1250	8.0	Hydrogen and Water Vapor	3.45	500			1379.0	200.0		
	1144	1600	8.0	Hydrogen and Water Vapor	3.45	500			799.8	116.0		
	1144	1600	8.0	Hydrogen and Water Vapor	3.45	500			827.4	120.0		
	1144	1600	8.0	Hydrogen and Water Vapor	3.45	500			934.2	135.5		
IN-100	300	80	Smooth	Helium	34.5	5000	688.1	99.8	817.0	118.5	9.5	14.7
	300	80	Smooth	Hydrogen	34.5	5000	734.3	106.5	741.2	107.5	3.0	3.3
	300	80	Smooth	Hydrogen	34.5	5000	603.3	87.5	603.3	87.5	2.5	5.5
	951	1250	Smooth	Helium	34.5	5000	701.9	101.8	711.5	103.2	2.0	6.7
	951	1250	Smooth	Hydrogen	34.5	5000	682.6	99.0	682.6	99.0	1.5	2.0
	951	1250	Smooth	Hydrogen	34.5	5000	731.5	106.1	786.0	114.0	2.0	7.1
MAR M-200 DS Heat Code P-9108	300	80	Smooth	Helium	34.5	5000	924.6	134.1	1090.8	158.2	8.0	9.7
	300	80	Smooth	Hydrogen	34.5	5000	787.4	114.2	787.4	114.2	2.0	1.6
	300	80	Smooth	Hydrogen	34.5	5000	851.5	123.5	851.5	123.5	2.0	2.9
	951	1250	Smooth	Helium	34.5	5000	941.1	136.5	1182.5	171.5	7.5	11.[illegible]
	951	1250	Smooth	Hydrogen	34.5	5000	896.3	130.0	961.8	139.5	4.0	5.5
	951	1250	Smooth	Hydrogen	34.5	5000	958.4	139.0	1065.2	154.5	4.0	6.7
MAR M-200 DS Heat Code P-9199	951	1250	8.0	Helium	34.5	5000			1123.8	163.0		
	951	1250	8.0	Hydrogen	34.5	5000			1057.7	153.4		
	951	1250	8.0	Hydrogen	34.5	5000			954.9	138.5		
	951	1250	8.0	Hydrogen	34.5	5000			1134.2	164.5		
	951	1250	8.0	Hydrogen H_2O	34.5	5000			751.5	109.0		
	951	1250	8.0	Hydrogen H_2O	34.5	5000			1010.1	146.5		
	951	1250	8.0	Hydrogen H_2O	34.5	5000			879.1	127.5		
	1144	1600	8.0	Helium	3.45	500			909.4	131.9		
	1144	1600	8.0	Hydrogen	3.45	500			903.2	131.0		
	1144	1600	8.0	Hydrogen	3.45	500			918.4	133.2		
	1144	1600	8.0	Hydrogen	3.45	500			879.1	127.5		
	1144	1600	8.0	Hydrogen H_2O	3.45	500			765.3	111.0		
	1144	1600	8.0	Hydrogen H_2O	3.45	500			724.0	105.0		
	1144	1600	8.0	Hydrogen H_2O	3.45	500			751.5	109.0		

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	MN m²	Pressure, psig	Yield Strength MN m²	Yield, ksi	Ultimate Strength MN m²	Ultimate, ksi	Ductility El, (1)	Ductility RA, (2)
A-286	300	80	Smooth	Helium	34.5	5000	719.8	104.4	1044.6	151.5	28.5	45.5
	300	80	Smooth	Helium	34.5	5000	734.3	109.4	1044.6	151.5	27.0	47.5
	300	80	8.0	Helium	34.5	5000			1536.2	222.8		
	300	80	8.0	Helium	34.5	5000			1554.8	225.5		
	300	80	Smooth	Hydrogen	34.5	5000	739.8	110.2	1074.2	155.8	28.5	46.4
	300	80	Smooth	Hydrogen	34.5	5000	766.7	111.2	1072.8	155.6	27.0	45.8
	300	80	8.0	Hydrogen	34.5	5000			1554.1	225.4		
	300	80	8.0	Hydrogen	34.5	5000			1546.5	224.3		
	300	80	8.0	Hydrogen	34.5	5000			1554.1	225.4		
	951	1250	Smooth	Helium	34.5	5000	721.2	104.6	820.5	119.0	25.5	52.1
	951	1250	Smooth	Helium	34.5	5000	717.1	104.0	843.9	122.4	29.0	54.5
	951	1250	8.0	Helium	34.5	5000			1148.0	166.5		
	951	1250	8.0	Helium	34.5	5000			1254.9	182.0		
	951	1250	Smooth	Hydrogen	34.5	5000	735.7	106.7	819.8	118.9	22.0	44.6
	951	1250	Smooth	Hydrogen	34.5	5000	843.2	122.3	753.6	109.3	25.0	46.6
	951	1250	8.0	Hydrogen	34.5	5000			1196.2	173.5		
	951	1250	8.0	Hydrogen	34.5	5000			1272.1	184.5		
	951	1250	8.0	Hydrogen	34.5	5000			1283.8	186.2		
AISI 347	111	-260	Smooth	Helium	34.5	5000	688.1	99.8	1357.6	196.9	43.5	64.0
	111	-260	Smooth	Helium	34.5	5000	751.5	109.0	1395.5	202.4	42.5	64.6
	111	-260	8.0	Helium	34.5	5000			1588.6	230.4		
	111	-260	8.0	Helium	34.5	5000			1567.9	227.4		
	111	-260	Smooth	Hydrogen	34.5	5000	665.7	96.5	1310.0	190.0	42.5	64.6
	111	-260	Smooth	Hydrogen	34.5	5000	661.9	96.0	1221.8	177.2	43.5	62.8
	111	-260	8.0	Hydrogen	34.5	5000			1525.8	221.3		
	111	-260	8.0	Hydrogen	34.5	5000			1557.5	225.9		
	111	-260	8.0	Hydrogen	34.5	5000			1534.8	222.6		
	300	80	Smooth	Helium	34.5	5000	480.6	69.7	703.3	102.0	38.0	70.1
	300	80	Smooth	Helium	34.5	5000	440.6	63.9	685.3	99.4	36.5	70.5
	300	80	8.0	Helium	34.5	5000			1172.1	170.0		
	300	80	8.0	Helium	34.5	5000			1194.9	173.3		
	300	80	Smooth	Hydrogen	34.5	5090	445.4	64.6	756.4	109.7	41.5	71.1
	300	80	Smooth	Hydrogen	34.5	5000	464.7	67.4	746.7	108.3	39.0	70.4
	300	80	8.0	Hydrogen	34.5	5000			1150.1	166.8		
	300	80	8.0	Hydrogen	34.5	5000			1186.6	172.1		
	300	80	8.0	Hydrogen	34.5	5000			881.2	127.8		
	951	1250	Smooth	Helium	34.5	5000	398.5	57.8	416.4	60.4	24.5	66.3
	951	1250	Smooth	Helium	34.5	5000	395.8	57.4	410.9	59.6	25.0	68.1
	951	1250	8.0	Helium	34.5	5000			717.1	104.0		
	951	1250	8.0	Helium	34.5	5000			708.1	102.7		
	951	1250	Smooth	Hydrogen	34.5	5000	377.1	54.7	405.4	58.8	25.5	68.3
	951	1250	Smooth	Hydrogen	34.5	5000	415.8	60.3	442.6	64.2	25.0	66.7
	951	1250	8.0	Hydrogen	34.5	5000			699.1	101.4		
	951	1250	8.0	Hydrogen	34.5	5000			689.5	100.0		
	951	1250	8.0	Hydrogen	34.5	5000			688.1	99.8		

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	Pressure, MN/m²	Pressure, Psig	Yield Strength, MN/m²	Yield Strength, ksi	Ultimate Strength, MN/m²	Ultimate Strength, ksi	Ductility El,(1) %	Ductility RA,(2) %
Titanium 6-4	300	80	Smooth	Helium	34.5	5000	1002.5	145.4	1041.8	151.1	15.0	45.4
	300	80	Smooth	Helium	34.5	5000	1011.5	146.7	1041.1	151.0	15.0	44.0
	300	80	8.0	Helium	34.5	5000			1415.5	205.3		
	300	80	8.0	Helium	34.5	5000			1444.5	209.5		
	300	80	Smooth	Hydrogen	34.5	5000	993.5	144.1	1019.1	147.8	13.5	34.0
	300	80	Smooth	Hydrogen	34.5	5000	1004.6	145.7	1052.8	152.7	13.5	37.8
	300	80	8.0	Hydrogen	34.5	5000			1307.9	189.7		
	300	80	8.0	Hydrogen	34.5	5000			1265.2	183.5		
	300	80	8.0	Hydrogen	34.5	5000			1232.8	178.8		
	366	200	Smooth	Helium	34.5	5000	953.6	138.3	1051.5	152.5	15.0	46.6
	366	200	Smooth	Helium	34.5	5000	959.8	139.2	1017.7	147.6	16.0	47.0
	366	200	8.0	Helium	34.5	5000			1430.7	207.5		
	366	200	8.0	Helium	34.5	5000			1430.7	207.5		
	366	200	8.0	Helium	34.5	5000			1423.8	206.5		
	366	200	Smooth	Hydrogen	34.5	5000	972.2	141.0	1008.7	146.3	10.5	22.6
	366	200	Smooth	Hydrogen	34.5	5000	954.9	138.5	997.7	144.7	12.0	31.4
	366	200	8.0	Hydrogen	34.5	5000			1441.0	209.0		
	366	200	8.0	Hydrogen	34.5	5000			1271.4	184.4		
	366	200	8.0	Hydrogen	34.5	5000			1263.8	183.3		
A-110	300	80	Smooth	Helium	34.5	5000	797.0	115.6	913.6	132.5	20.0	44.6
	300	80	Smooth	Helium	34.5	5000	898.4	130.3	917.7	133.1	18.5	43.9
	300	80	8.0	Helium	34.5	5000			1412.7	204.9		
	300	80	8.0	Helium	34.5	5000			1359.6	197.2		
	300	80	Smooth	Hydrogen	34.5	5000	834.3	121.0	929.4	134.8	14.5	35.4
	300	80	Smooth	Hydrogen	34.5	5000	921.1	133.6	957.7	138.9	16.5	31.4
	300	80	8.0	Hydrogen	34.5	5000			1090.8	158.2		
	300	80	8.0	Hydrogen	34.5	5000			1194.9	173.3		
	300	80	8.0	Hydrogen	34.5	5000			1152.1	167.1		
	366	200	Smooth	Helium	34.5	5000	770.8	111.8	879.8	127.6	20.5	45.4
	366	200	Smooth	Helium	34.5	5000	783.2	113.6	914.9	132.7	17.5	46.6
	366	200	8.0	Helium	34.5	5000			1313.5	190.5		
	366	200	8.0	Helium	34.5	5000			1277.6	185.3		
	366	200	8.0	Helium	34.5	5000			1289.3	187.0		
	366	200	Smooth	Hydrogen	34.5	5000	788.8	114.4	868.7	126.0	14.0	20.8
	366	200	Smooth	Hydrogen	34.5	5000	775.7	112.5	896.3	130.0	17.0	31.9
	366	200	8.0	Hydrogen	34.5	5000			1104.5	160.2		
	366	200	8.0	Hydrogen	34.5	5000			1158.3	168.0		
	366	200	8.0	Hydrogen	34.5	5000			1210.0	175.5		

Table VIII-2. Tensile Properties of Materials in High Pressure Gaseous Environment (Continued)

Material	Test Conditions: Test Temperature, °K	Test Temperature, °F	Stress Concentration Factor	Environment	Pressure, MN/m²	Pressure, Psig	Test Results: Strength: Yield, MN/m²	Yield, ksi	Ultimate, MN/m²	Ultimate, ksi	Ductility: El,(1) %	RA,(2) %
Haynes 188	300	80	8.0	Helium	3.45	500			1120.4	162.5		
	300	80	8.0	Hydrogen	3.45	500			1092.8	158.5		
	300	80	8.0	Hydrogen	3.45	500			1106.6	160.5		
	300	80	8.0	Hydrogen	3.45	500			1081.1	156.8		
	951	1250	8.0	Helium	3.45	500			677.8	98.3		
	951	1250	8.0	Hydrogen	3.45	500			688.1	99.8		
	951	1250	8.0	Hydrogen	3.45	500			675.0	97.9		
	951	1250	8.0	Hydrogen	3.45	500			690.9	100.2		
	951	1250	8.0	Hydrogen + H_2O	3.45	500			712.9	103.4		
	951	1250	8.0	Hydrogen + H_2O	3.45	500			696.4	101.0		
	951	1250	8.0	Hydrogen + H_2O	3.45	500			708.8	102.8		
	1144	1600	8.0	Helium	3.45	500			534.4	77.5		
	1144	1600	8.0	Hydrogen	3.45	500			570.9	82.8		
	1144	1600	8.0	Hydrogen	3.45	500			551.6	80.0		
	1144	1600	8.0	Hydrogen	3.45	500			488.8	70.9		
	1144	1600	8.0	Hydrogen + H_2O	3.45	500			565.4	82.0		
	1144	1600	8.0	Hydrogen + H_2O	3.45	500			561.9	81.5		
	1144	1600	8.0	Hydrogen + H_2O	3.45	500			555.0	80.5		

Notes:

(1) Elongation Measured Over 25.4-mm (1-in.) Original Gage Length.

(2) Reduction of Area.

(3) Maximum Pressure Obtainable at 1144°K (1600°F).

Strain Rate for Smooth Tensile Specimens was 0.005 mm/mm/min (in./in./min) Through Yield and 0.010 mm/mm/min (in./in./min) From Yield Through Ultimate. Crosshead Speed for Notched Tensile Tests was 0.127 mm/min (0.050 in./min).

C. TEST PROCEDURE

Two types of tensile specimens, smooth and notched, were used for this testing. Smooth tensile specimens had a 6.40-mm (0.252-in.) gage diameter and a gage length of 25.4-mm (1.00-in.). Notched specimens (K_T of 8.0) had a larger diameter of 12.70-mm (0.500-in.) and a notch diameter of 8.00-mm (0.315-in.) machined in the center of the specimen gage at a 60-deg angle, with a 0.051-mm (0.002-in.) radius at the apex of the notch. The specimens are described in Section III and detailed by figures III-8 and III-9.

Tensile testing was done with a Tinius Olsen 133.4-KN (30,000-lb) capacity tensile machine, equipped with a P&WA designed pressure vessel. All controls and instrumentation readout equipment were located inside an adjacent blockhouse. This equipment is shown in figures VIII-7 and VIII-8a. The pressure vessel is shown open with a notched specimen in place in figure VIII-8b and in figure VIII-8c, with a smooth specimen and room temperature extensometer in place. The vessel is made of AISI 347 stainless steel and incorporates a high pressure GrayLoc connector. A compensating device built into the base of the vessel eliminates the effect of load resulting from differential specimen and adapter cross-sectional areas.

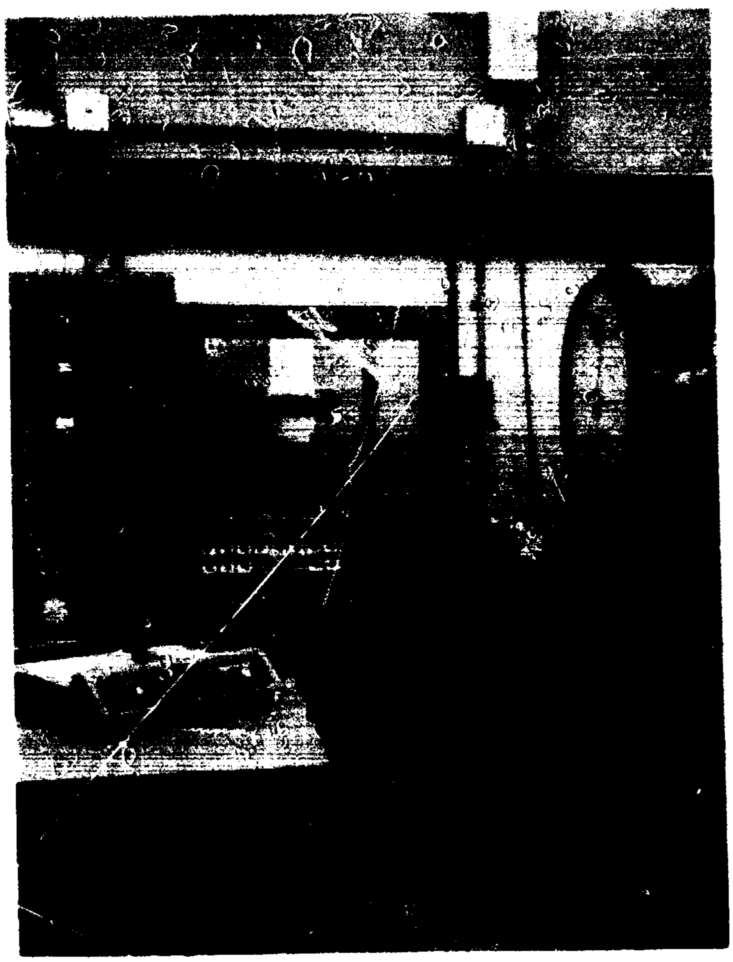
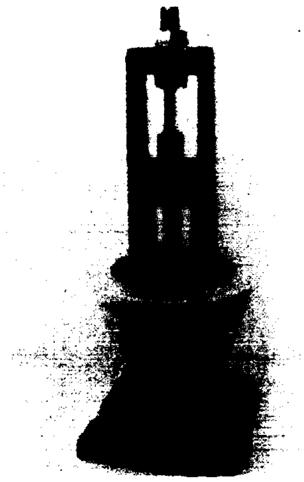


Figure VIII-7. Tensile Machine and Test Environment Controls and Data Acquisition Equipment FC 21272

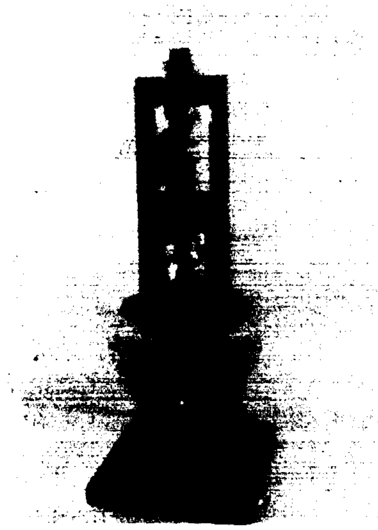
FC 21268

a) Test Vessel Installed on Tensile Machine in Remote Test Cell



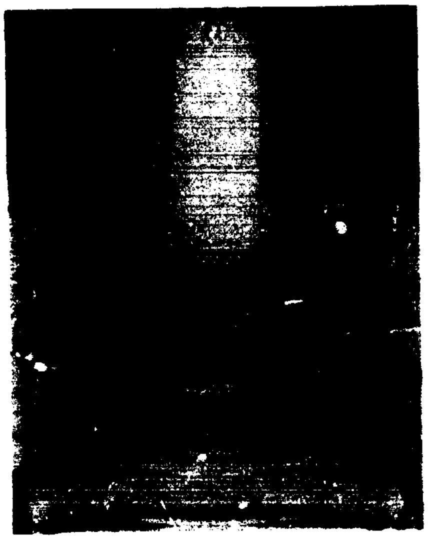
FC 21572

b) Test Vessel Open With Notch Tensile Specimen in Place



FC 21573

c) Test Vessel Open With Smooth Specimen in Place and Room Temperature Extensometer Attached



FE 107940

d) Test Vessel With Cryogenic Jacket Installed

Figure VIII-8. Various Views of Test Vessel FD 53125

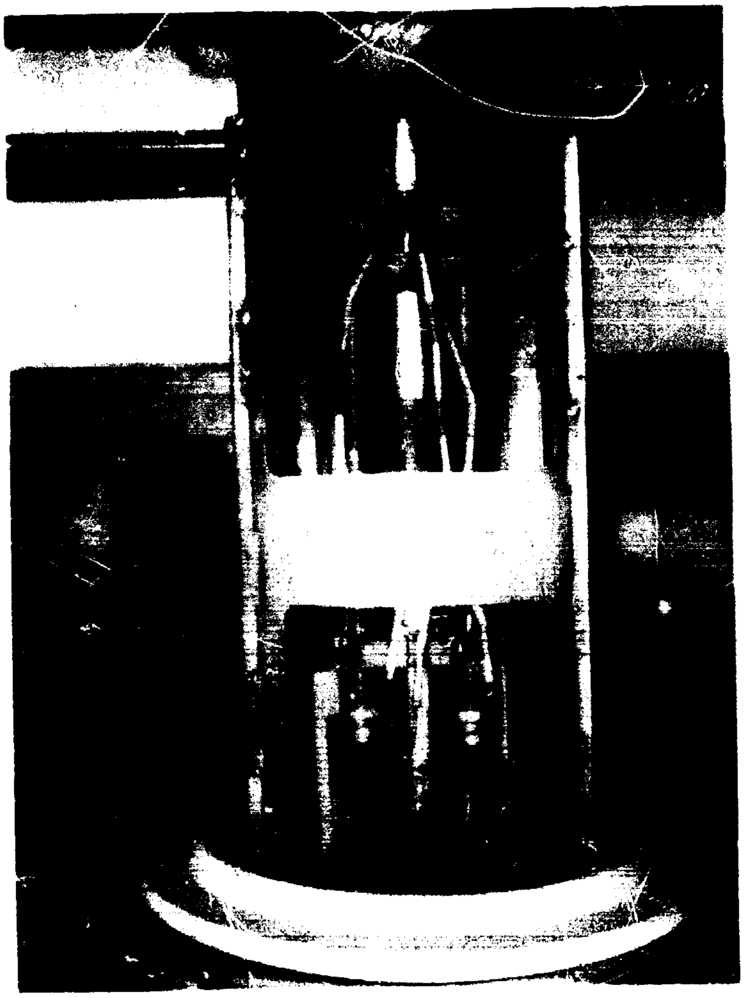


Figure VIII-9. Test Vessel With Outer Chamber Removed, Showing Specimen, Extensometer, and One-Half of Furnace in Place FE 107943

After the initial series of 300°K (80°F) tests were completed, the tensile machine was upgraded from a 133.4 (30,000) to a 266.8-KN (60,000-lb) capacity, and the extensometer system was converted to a proximity probe type similar to those systems in use for the creep-rupture and low-cycle fatigue tests. Specimen load was determined by both the tensile machine load measuring system and an internal strain gage-type load cell; thus, absolute specimen load was known and friction at the pressure vessel seals was of no consequence. Electrical connections to the internal load cell, extensometer, thermocouples, and furnace were made through the bottom of the pressure vessel via high pressure bulkhead connectors.

To conduct cryogenic tests, the pressure vessel was modified by the addition of an insulated jacket placed over the upper portion. (See figure VIII-8d.) This jacket has provisions for filling with and flowing LN_2, thus providing cryogenic temperature inside the pressure vessel. To enhance cooling, the test gas passed through a heat exchanger coil, located inside the LN_2 jacket, before passing into the pressure vessel.

To conduct elevated temperature tests, a two-zone furnace with separate control systems for each zone was used that minimized any heat gradient due to the high thermal conductivity of the gases. This furnace fit within the pressure vessel load frame. Thermocouples looped around the specimen gage section (or notch) were used to control and monitor specimen temperature during each test. Temperature variation over the 24.5-mm (1-in.) gage length of the smooth specimen was less than 2% during testing. The high temperature extensometer system is shown, with thermocouples installed on a smooth specimen and half of the furnace in place in the test vessel, in figure VIII-9. For the hydrogen and water vapor tests, a thin wall retort was constructed to fit inside the existing furnace. This was possible because of the relatively large space available and the absence of extensometry for notched tensile tests. Pressure inside the retort and the heavy wall pressure vessel was equalized; therefore, the retort contained the hydrogen-water vapor environment only and did not withstand the pressure loading. Distilled water was injected into the base of the retort, and the percent of water vapor content in the environment regulated by controlling the temperature at the bottom of the retort. This temperature was adjusted to obtain approximately 50% by weight water vapor, which simulated the hydrogen-water vapor environment present in the preburner area of a staged combustion rocket engine.

The following procedure was used during testing:

1. Chamber is opened and prepared specimen installed. In the case of a smooth specimen, the extensometer is attached.

2. Instrumentation continuity checks are conducted.

3. Chamber is closed, sealed, and vessel/plumbing evacuated.

4. Chamber is backfilled with nitrogen and purged.

5. Chamber is filled and "pop" purged six times in succession with the test gas to 3.45 MN/m^2 (500 psig). A prerun test gas sample is taken.

6. Chamber is pressurized with test gas to 3.45 (500) or 34.5 MN/m^2 (5000 psig).

7. In the case of either cryogenic or elevated tests, the temperature is attained and the specimen soaked at test temperature for 10 min.

8. Test is conducted. During the test, specimen load is obtained from both internal and external load cells. Specimen strain, for smooth specimens, is obtained from an extensometer attached directly to the specimen.

9. After specimen failure, a post-run gas sample is taken, and the chamber is then vented (and purged with nitrogen in the case of a hydrogen test), opened, and failed specimen removed.

END

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