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LOW CYCLE FATIGUE PROPERTIES OF MAR-M-246 Hf IN HYDROGEN

FINAL REPORT

Contract NAS8-33106

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

PRATT & WHITNEY AIRCRAFT GROUP
Government Products Division
P. O. Box 2691
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UNITED TECHNOLOGIES®

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FOREWORD

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

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

J. R. Warren	— Principal Investigator
D. T. DiGenova	— Data Analysis and Report Preparation
D. P. Shoemaker	— Low-Cycle Fatigue Mechanical Tests
J. W. Lee	— TLP Bonding and Metallurgical Investigations

SECTION I

INTRODUCTION

The objective of this program is to determine the transverse, low-cycle fatigue (LCF) properties of MAR-M-246 Hf in a gaseous hydrogen environment at 760°C (1400°F). This alloy is proposed for use in space propulsion systems in pure or partial high-pressure hydrogen environments at elevated temperatures.

The MAR-M-246 Hf was supplied in two conditions: directionally solidified (DS) and single crystal (SC). The test environment was hydrogen gas at a pressure of 34.5 MPa (5000 psig).

All testing was conducted using solid specimens exposed to external gaseous pressure. Low-cycle fatigue life was established by strain controlled testing using smooth specimens and a servohydraulic closed-loop test machine modified with a high pressure environmental chamber.

This report is arranged in sections that cover the material tested and results and conclusions of the LCF property tests.

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

SECTION II

MATERIALS AND SPECIMENS

A. TEST MATERIAL

The purpose of this program was to determine the low cycle fatigue properties of MAR-M-246 Hf, a cast nickel-base alloy, in the transverse direction in a high-pressure hydrogen environment. Testing evaluated the material in two forms: directionally solidified (DS), and single crystal (SC).

All test materials were supplied in the fully heat-treated condition by the Marshall Space Flight Center (MSFC). The DS material was supplied in the form of rectangular castings, 1.3 cm × 3.8 cm × 14.0 cm, with solidification in the 14.0 cm direction. The SC material was also supplied as rectangular bars, 1.3 cm × 3.8 cm × 10.2 cm, with the primary crystal axis [001] in the 10.2 cm direction.

The materials tested, their form, heat treatments, and chemical compositions are listed in Table I.

B. TEST GASES

Hydrogen gas was used during the testing of specimens, and nitrogen was used as a preliminary purge gas. Propellant grade hydrogen was provided under Military Specification P-27201, which requires the gas to have an oxygen content of less than 1 part per million.

The gas handling system supplying the test vessel was equipped to enable sampling before and after specimen tests. Samples were analyzed with a gas chromatograph with accuracy in the parts per million range. Analysis verified that the gas was of the required purity (<1 ppm O_2). Hydrogen gas pressure was maintained at 34.5 MPa (5000 psig) during testing.

C. TEST SPECIMENS

The requirement for obtaining LCF properties in the transverse direction made it necessary to section the rectangular bar raw material in preparation for bonding. This bonding of additional material allowed for sufficient length such that test specimens could be obtained in the transverse direction (see Figures 1, 2, and 3).

The specimen blank material was bonded by the transient-liquid-phase (TLP) diffusion bonding process. In this process, a bonding alloy foil is placed between the bond faying surfaces. Pressure is utilized to cause intimate contact between the mating surfaces. The bonding alloy foil melts and solidifies isothermally at the bonding temperature. Bond joint mechanical properties approaching those of the base metal are obtained by proper selection of the bonding alloy composition, time, temperature and pressure.

The bonding alloy used was PWA 1182 which has a composition similar to MAR-M-200 Hf. The standard heat treatment for MAR-M-246 was incorporated into the TLP bond cycle. The TLP bond process was as follows:

A. 1196°C (2185°F) ±8°C (15°F)/22 hr in vacuum (TLP Bond), Heat to 1221°C (2230°F).

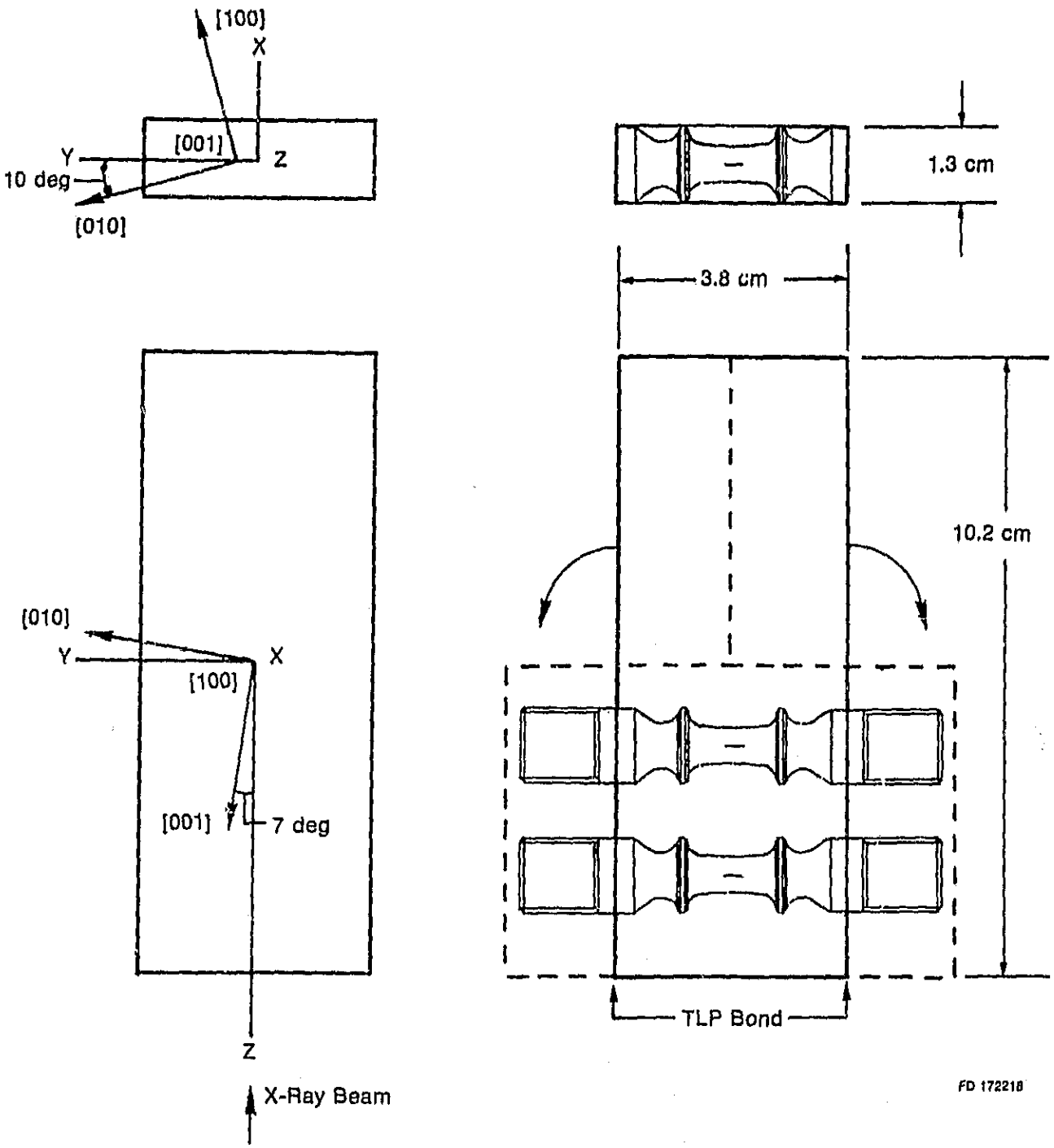


Figure 1. Crystallographic Analysis and Specimen Orientation for Single Crystal MAR-M-246 Hf (Specimens A-1 and A-2)

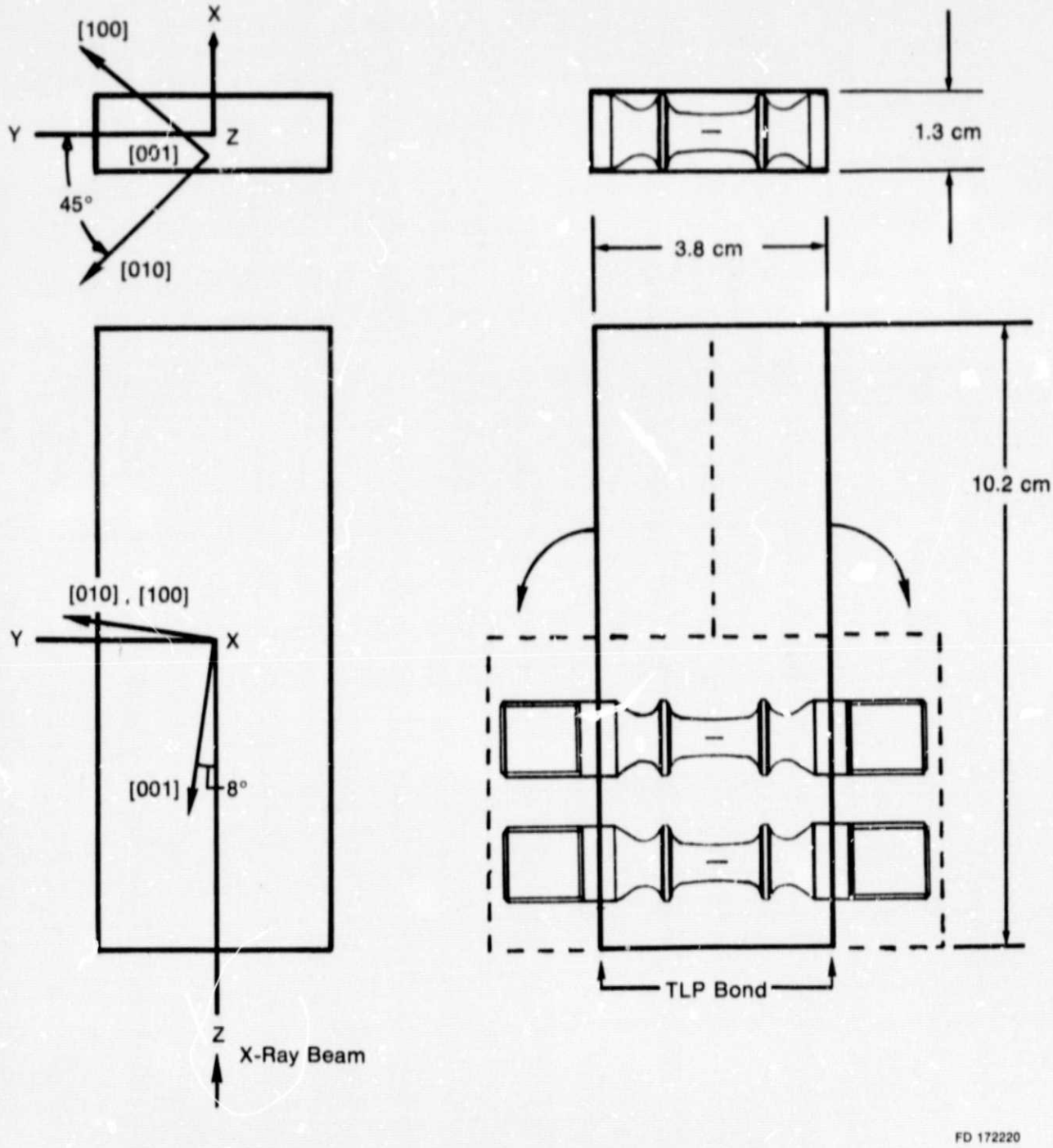


Figure 2. Crystallographic Analysis and Specimen Orientation for Single Crystal MAR-M-246 Hf (Specimens B-1 and B-2)

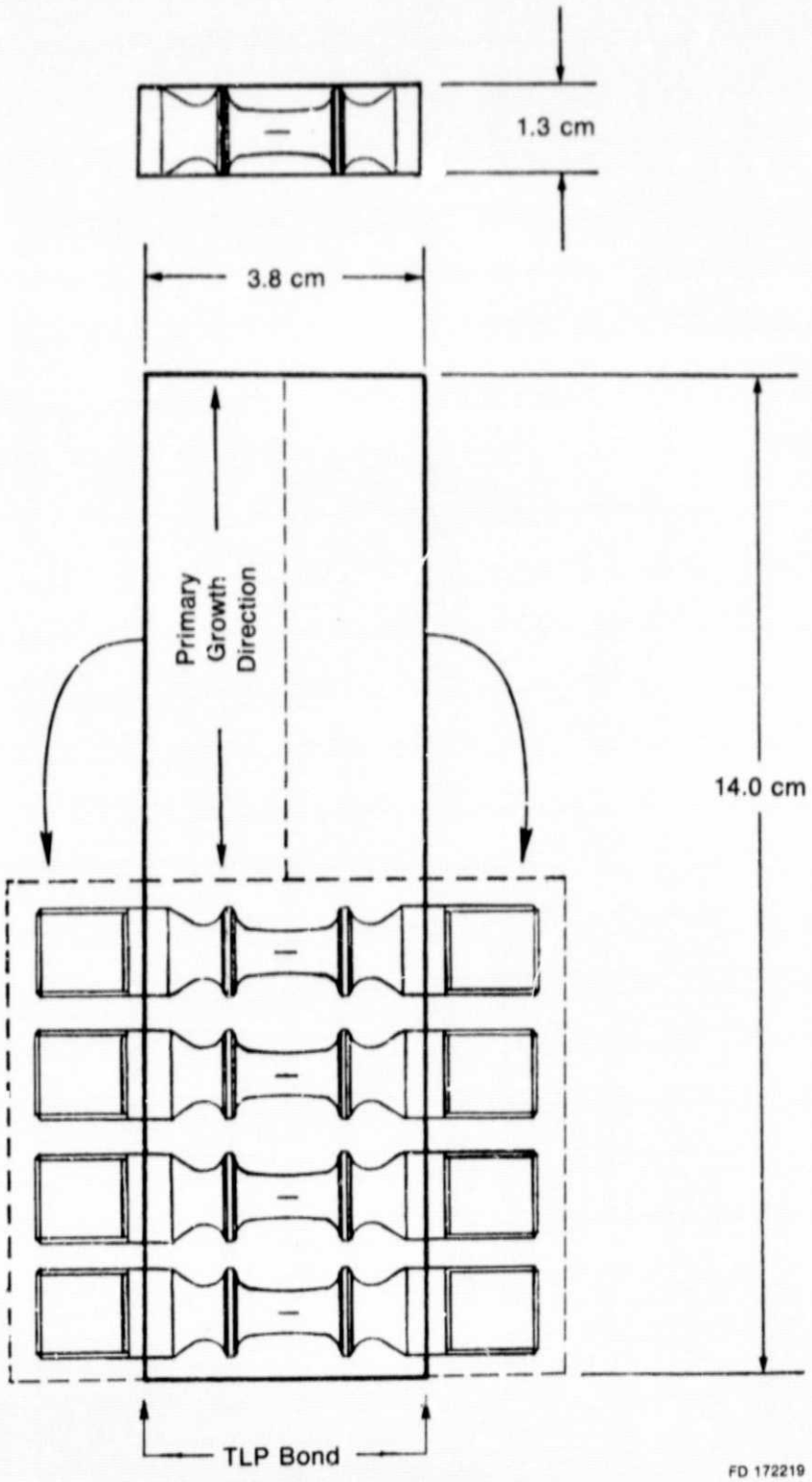


Figure 3. Specimen Orientation for Directionally Solidified MAR-M-246 Hf (Specimens E-1 Through E-4)

B. 1221°C (2230°F) ±8°C (15°F)/2 hr in vacuum. Cool to room temperature (Solution H.T.).

C. 871°C (1600°F) ±8°C (15°F)/24 hr in vacuum. Cool to room temperature (Precipitation H.T.).

The microstructure was examined after the TLP bond and heat treatment cycle (see Figure 4). Both single crystal and directionally solidified materials exhibited similar microstructure. They were characterized by eutectic gamma prime and needle-like carbide particles in a matrix of gamma prime.

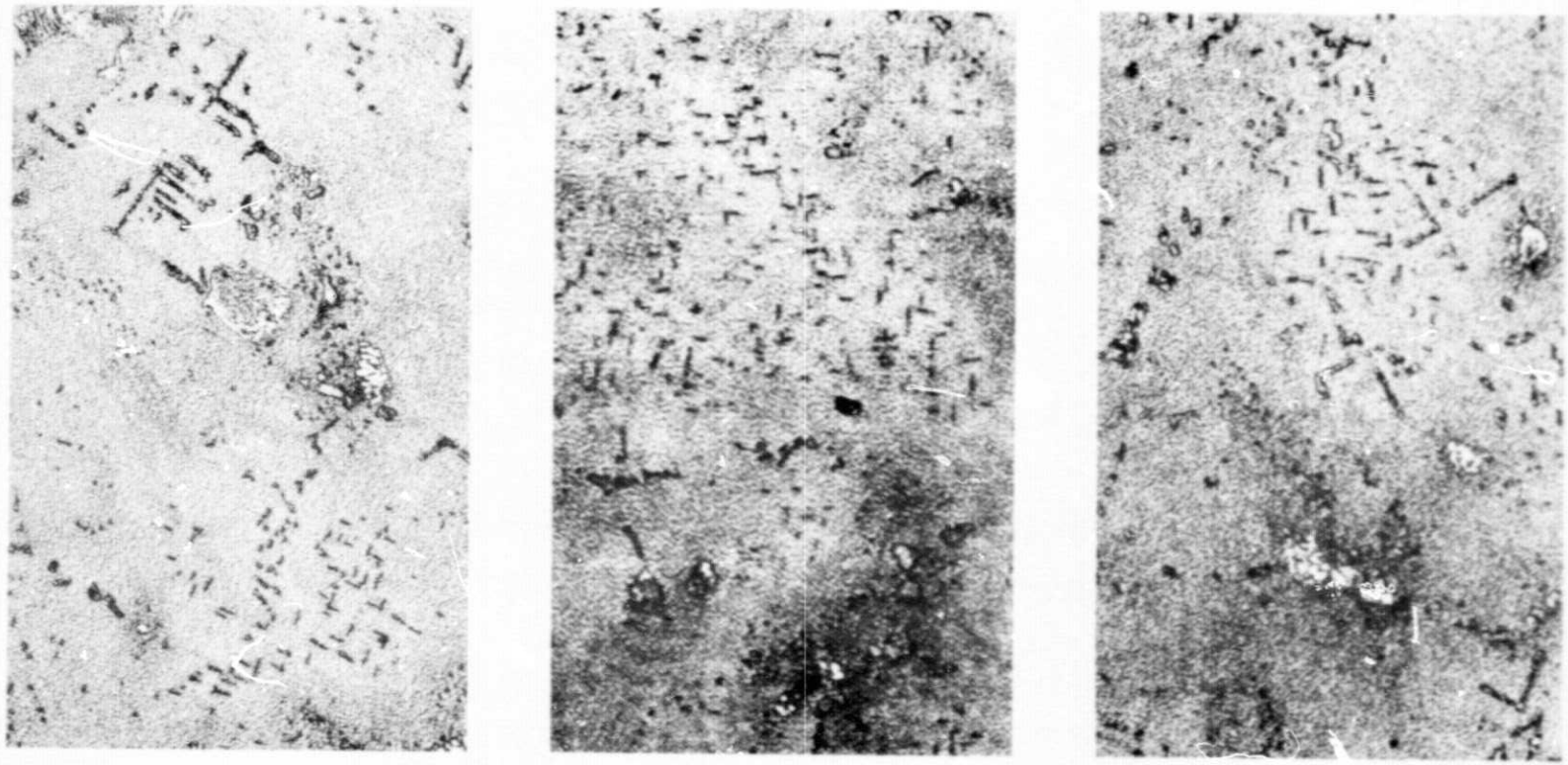
The crystallographic orientation of the single crystal material was determined by the Laue back reflection technique. Results are presented in Figures 1 and 2. The primary axis [001] orientations in relation to the cast bar longitudinal axes were 7 deg and 8 deg for raw material blanks A and B, respectively. These are both within general manufacturing requirements (i.e., within 10 deg of the longitudinal axis of the manufactured part). The transverse [010] axes however, varied in orientation from the specimen longitudinal axes, by 10 deg for bar A, to 45 deg for bar B. The crystallographic axes orientations are characterized in a unit stereographic triangle in Figure 5.

TABLE I. MATERIAL FORM, HEAT TREATMENTS, AND CHEMICAL COMPOSITION OF MAR-M-246 Hf RAW MATERIAL

Form:	Single Crystal	Directionally Solidified
Heat No.:	DE 006	DE 006
Identification No.:	C11(A), M12(B)	D6, M7
Supplier:	Howmet Turbine Components Corporation/Dover Division	
Standard Heat Treatment:	1221°C (2230°F)/2 hr in vacuum/inert Cool to room temperature (Sol. H.T.) +871°C (1600°F)/24 hr in vacuum/inert Cool to room temperature (Precip. H.T.)	
TLP Bond Process:	1196°C (2185°F)/22 hr in vacuum, (TLP Bond) Heat to 1221°C (2230°F) 1221°C (2230°F)/2 hr in vacuum Cool to room temperature (Re-Sol. H.T.) 871°C (1600°F)/24 hr in vacuum Cool to room temperature (Re-Precip. H.T.)	

Chemical Composition:

Element	*Percent by Weight*
C	0.15
Mn	0.01
Si	0.05
S	0.003
Cr	8.26
Ni	Balance
W	9.16
Fe	0.03
Co	10.37
Mo	2.56
Al	5.66
Ti	1.69
Ta	1.69
Zr	0.04
B	0.016
Cu	0.01
Mg	0.0004
Hf	1.99



Blank A

Blank B

Blank E

Figure 4. Representative Microstructures of Single Crystal (Blanks A and B) and Directionally Solidified (Blank E) MAR-M-246 Hf in Postbond, Fully Heat Treated Condition (Mag 250X)

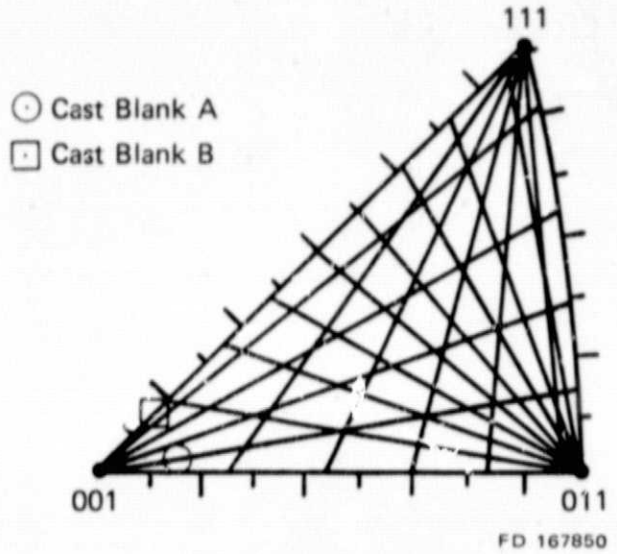


Figure 5. Unit Stereographic Triangle (Inverse Pole Figure) for Crystallographic Axes Orientations of Cast Blank Single Crystal MAR-M-246 Hf Test Material

Test specimens were machined from the bonded blanks by the P&WA Group, Materials Control Laboratory Machine Shop and finished to an average roughness of 8 μ-in. rms, or less. Specimens were oriented to obtain fatigue properties in the transverse direction and machined to print per Figure 6.

The specimen configuration, which incorporates integral machined extensometer collars, was determined experimentally using photoelastic and elastic-plastic strain analyses, and has been used extensively by P&WA for low cycle fatigue evaluations. A calibration procedure was established to relate the maximum strain to collar deflection during both the elastic and the plastic portions of the strain cycle. Subsequently, the specimen design and calibration procedures were analytically verified using finite element and mathematical model analyses.

All test specimens were visually examined prior to testing in normal light and with fluorescent penetrant to screen for machining anomalies or surface discontinuities. Additional specimens are randomly selected for thorough dimensional inspection to ensure conformance to print requirements.

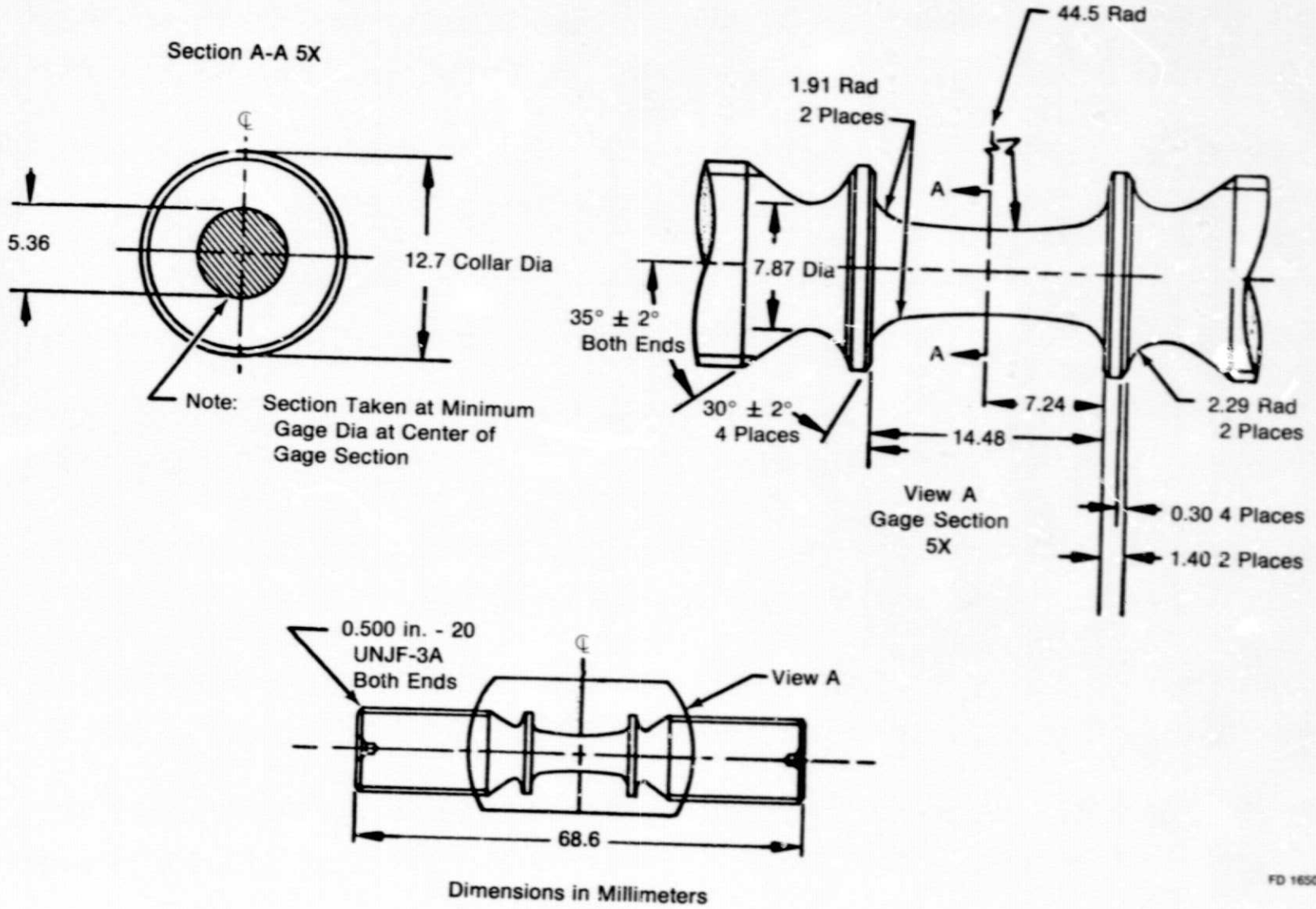


Figure 6. Strain Control Low Cycle Fatigue Specimen

SECTION III

LOW-CYCLE FATIGUE

A. INTRODUCTION

Strain controlled low-cycle fatigue (LCF) tests were conducted to establish the cyclic life of MAR-M-246 Hf in the transverse direction from two conditions, directionally solidified (DS) and single crystal (SC), in a high-pressure gaseous hydrogen environment.

The LCF tests were run in a strain-controlled mode using a triangular strain-time waveform at a frequency of 0.067 Hz (4 cycles per minute). Tests were performed with a fully reversed strain cycle (mean strain = 0), having a constant total strain range (elastic plus inelastic) and a compressive strain initial test start-up. A typical test stress-strain hysteresis loop and strain-time waveform are illustrated in Figure 7.

Strain ranges were selected based upon previous experience and current test experience to define total strain range vs cycles to failure from 50 to approximately 1,000 cycles. Tests were run to complete specimen fracture.

All tests were conducted at 760°C (1400°F) in a gaseous hydrogen environment at a pressure of 34.5 MPa (5000 psig).

B. RESULTS AND CONCLUSIONS

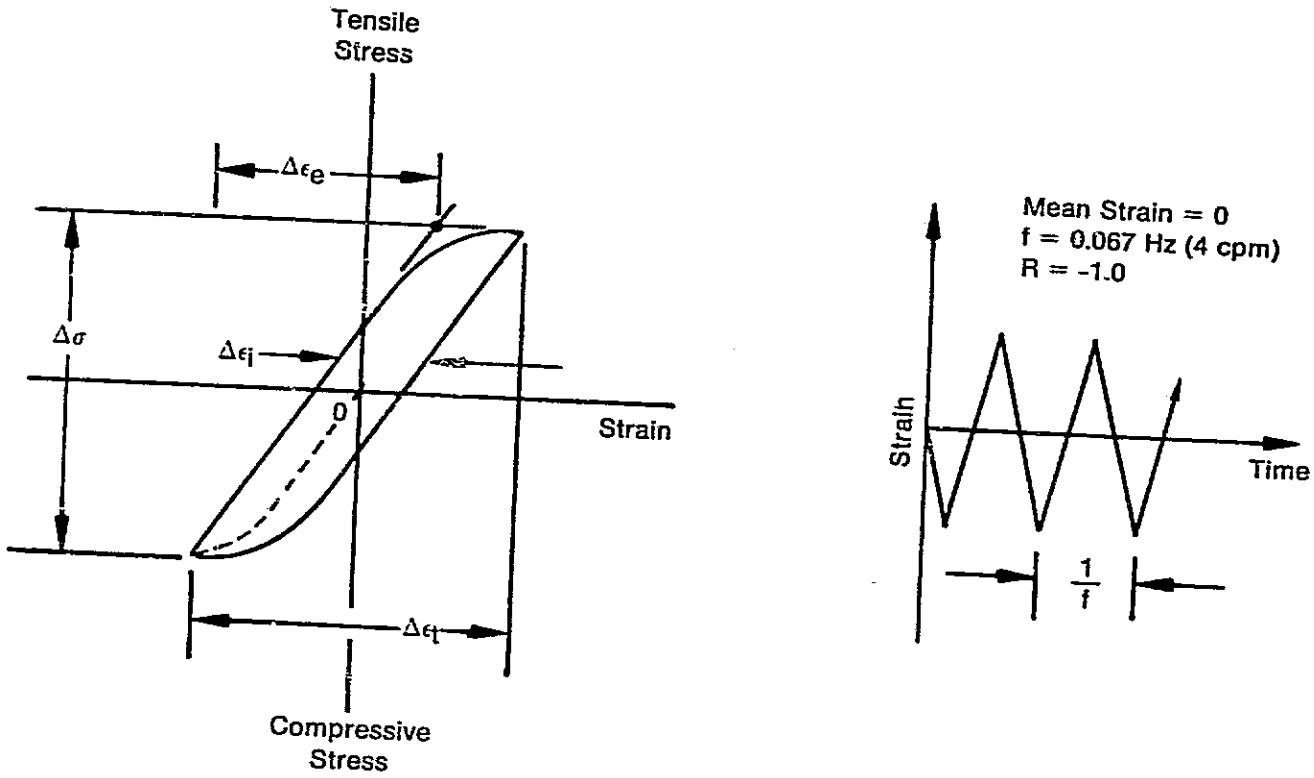
Eight strain-controlled tests were conducted to establish the transverse LCF material capability of MAR-M-246 Hf.

All test data are listed in Table II. Directionally solidified MAR-M-246 Hf test results are plotted in Figure 8, and single crystal test results are plotted in Figure 9. Both DS and SC test results are compared in Figure 10.

TABLE II. STRAIN CONTROL LCF TEST RESULTS FOR MAR-M-246 Hf (IN TRANSVERSE DIRECTION) IN HYDROGEN ATMOSPHERE AT 34.5 MPa (5000 psig). MEAN STRAIN =0, TEMPERATURE = 760°C (1400°F), FREQUENCY = 0.067 Hz (4 cpm)

				Strain (%)					
		Elastic Modulus				Inelastic			Cycles to
Condition	S/N	MPa × 10³	psi × 10⁶	Total	Elastic	Average	Min	Max	Failure
Directionally Solidified	E-1	144.8	21.0	1.39	1.10	0.29	0.17	0.41	19
	E-2	139.3	20.2	1.08	1.02	0.06	0.03	0.08	380
	E-3	151.7	22.0	0.91	0.88	0.03	0.02	0.04	1,480
	E-4	148.2	21.5	1.23	1.14	0.09	0.06	0.12	59
Single Crystal (A)*	A-1	106.2	15.4	1.25	1.23	0.02	0.01	0.03	17,162
	A-2	104.1	15.1	1.92	1.76	0.16	0.07	0.25	335
Single Crystal (B)*	B-1	164.1	23.8	1.79	1.18	0.61	0.40	0.82	78
	B-2	176.5	25.6	1.12	1.03	0.09	0.05	0.13	493

* Condition: Crystallographic analysis showed that the [010] transverse axis was inclined 10 deg from longitudinal axis of specimens machined from bar (A), and 45 deg from longitudinal axis of specimens machined from bar (B). See Figures 1, 2 and 5.



$\Delta\sigma$ =Total Stress Range
$\Delta\epsilon_t$ =Total Strain Range = $\Delta\epsilon_e + \Delta\epsilon_i$
$\Delta\epsilon_i$ =Inelastic Strain Range
$\Delta\epsilon_e$ =Elastic Strain Range = $\Delta\epsilon_t - \Delta\epsilon_i$
R =Minimum Strain/Maximum Strain
f =Cyclic Frequency

FD 165048

Figure 7. Typical Nondwell LCF Test With Mean Strain of Zero

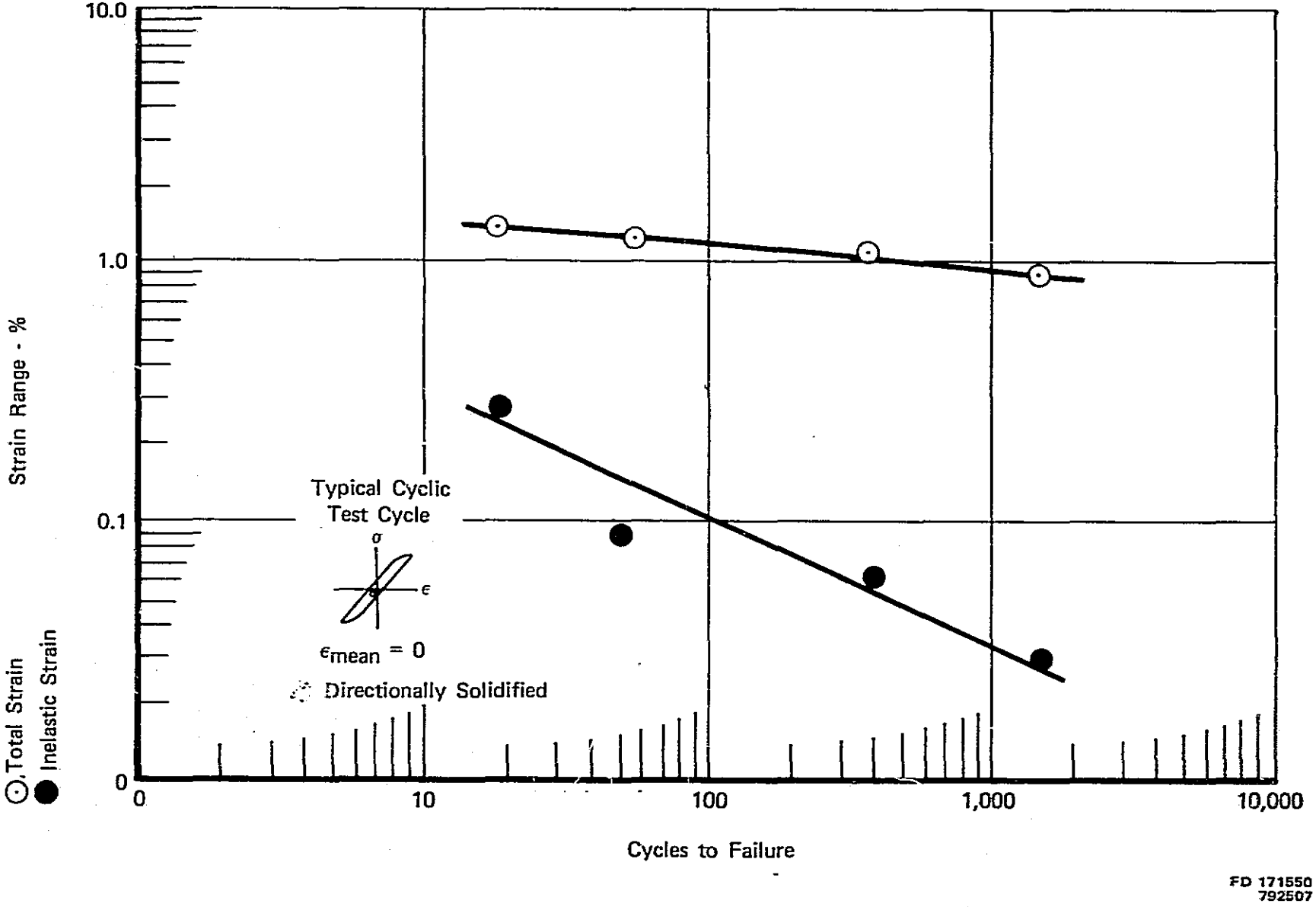


Figure 8. Strain Controlled LCF of Directionally Solidified MAR-M-246 Hf Tested in Transverse Direction in Hydrogen Atmosphere at 34.5 MPa (5000 psig). Temperature = 760°C (1400°F), Mean Strain = 0, Frequency - 0.067 Hz (4cpm)

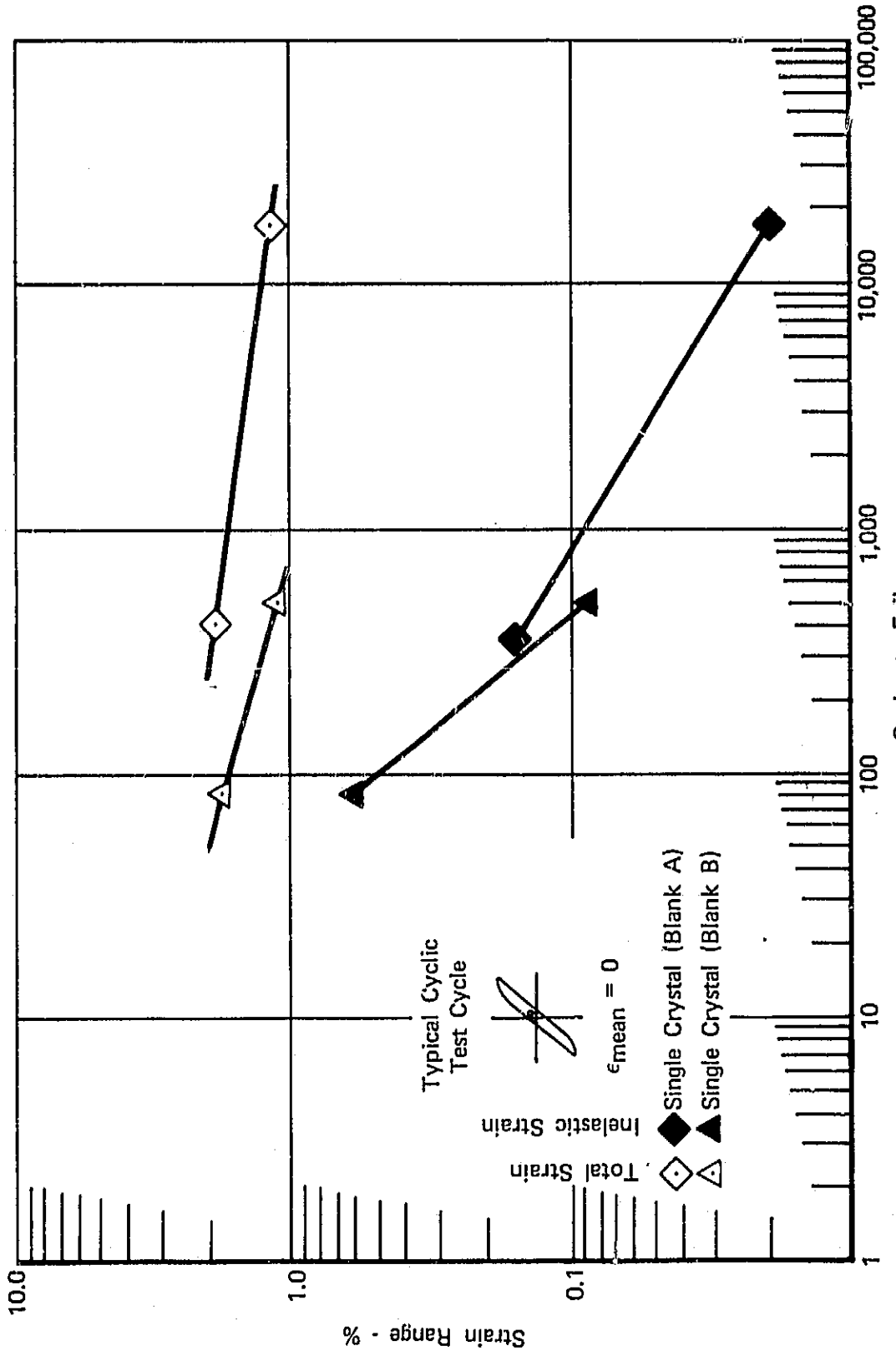


Figure 9. Comparison of Strain Controlled LCF Results of Single Crystal MAR-M-246 Hf Tested in Transverse Direction in Hydrogen Atmosphere at 34.5 MPa (5000 psig). Temperature = 760°C (1400°F), Mean Strain = 0, Frequency = 0.067 Hz (4 cpm.)

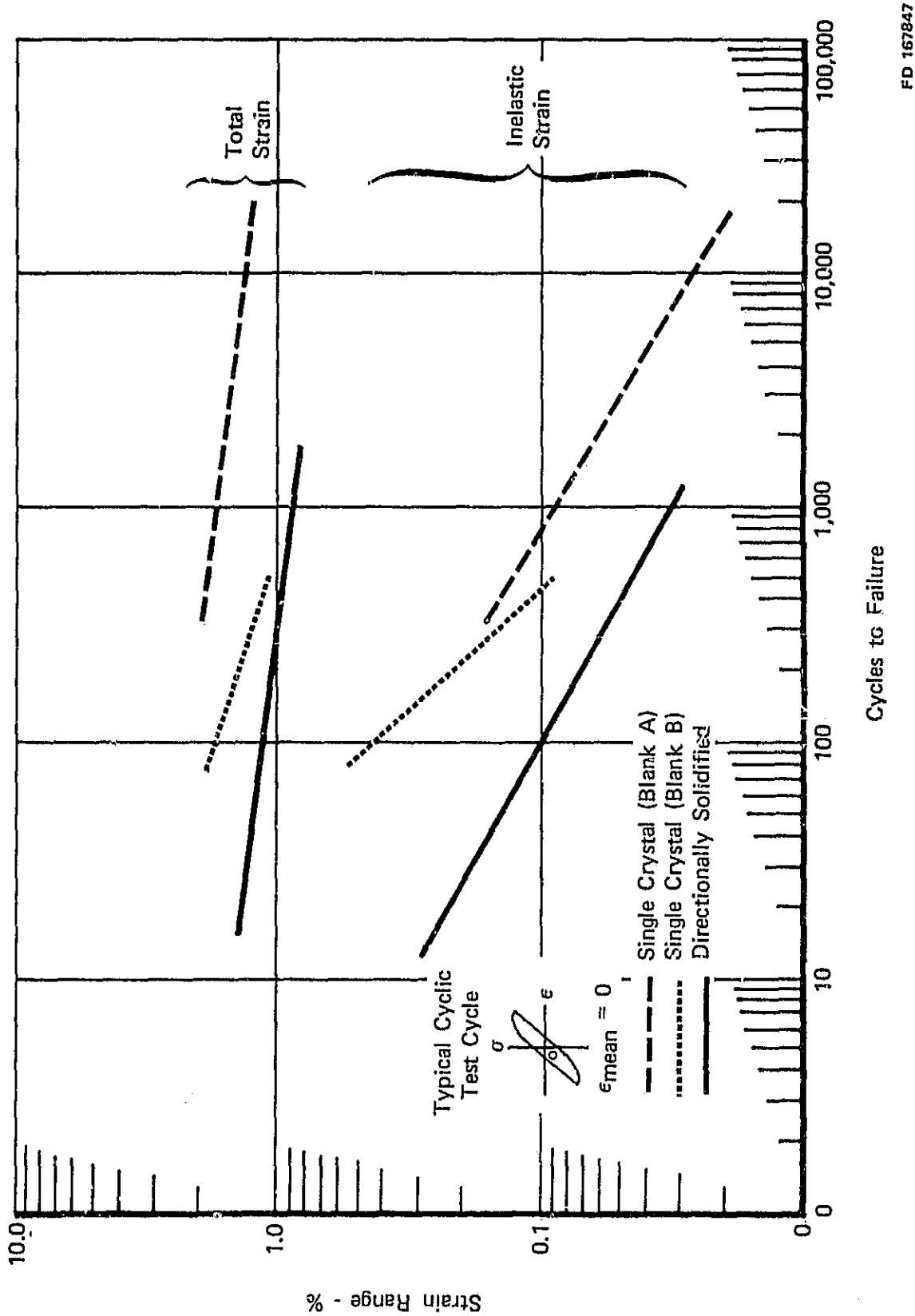


Figure 10. Comparison of Strain Controlled LCF Results of Single Crystal and Directionally Solidified MAR-M-246 Hf Tested in Transverse Direction In Hydrogen Atmosphere at 34.5 MPa (5000 psig). Temperature = 760°C (1400°F), Mean Strain = 0, Frequency = 0.067 Hz (4 cpm)

The single crystal material exhibited considerable scatter (Figure 9), however this could be attributed to the transverse [010] axis orientation. Specimens machined from raw material blank A (with a 10 deg inclination of the [010] transverse axis from the specimen longitudinal axis) had lives approximately an order of magnitude greater than specimens from raw material blank B (having a 45 deg inclination of the [010] transverse axis). Also, the elastic modulus differed significantly between specimens from blank A (E ≈ 105,000 MPa) and specimens from blank B (E ≈ 170,000 MPa) as shown in Table II.

The LCF capability of MAR-M-246 Hf in the single crystal condition was significantly greater than the directionally solidified material (Figure 10) at the conditions investigated. The degree of improvement in LCF properties varied from a factor of two to approximately a factor of one-hundred depending upon the total strain range and the single crystal transverse axis orientation.

The anisotropic nature of the single crystal material is illustrated in Figure 11. It can be seen here that the fracture face from specimen B-1 has an ovalized shape. It is interesting to note that the fracture location shown here and on all test specimens was within the specimen gage section, and that the TLP bond process was successful. Apparently TLP bond strength was not degraded at the temperature and environmental conditions studied.

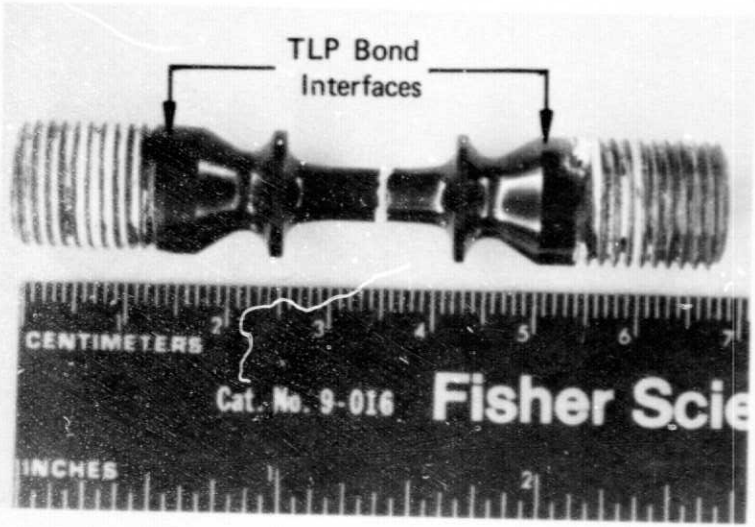
C. TEST PROCEDURE

All testing machines are controlled under a system of calibration and preventative maintenance schedules. System accuracies are within 2%. Approved calibration procedures, records, and NBS traceability are required for all test equipment from which data are obtained.

Isothermal strain-controlled LCF characteristics were determined using a servohydraulic, closed-loop-on-axial strain, LCF testing machine, designed and built at the Pratt & Whitney Aircraft, Government Products Division in Florida. The test machine is located in an isolated test cell with all controls and instrumentation located in an adjacent blockhouse (Figure 12). A P&WA designed pressure vessel was mounted on the upper platen of the test machine. The vessel incorporates a Grayloc type high-pressure flange for sealing and ease of assembly. The test machine compensates through the servosystem for the load in the specimen due to pressure acting over differential specimen/adapter areas. A pressure transducer provided a feedback signal, proportional to chamber pressure, to the servocontroller. This signal was used in controlling a mean load applied to the linkage so zero strain in the specimen gage was maintained when the vessel assembly was pressurized. This same load was then superimposed on the cyclic load during testing. Figure 13 illustrates the environmental test chamber.

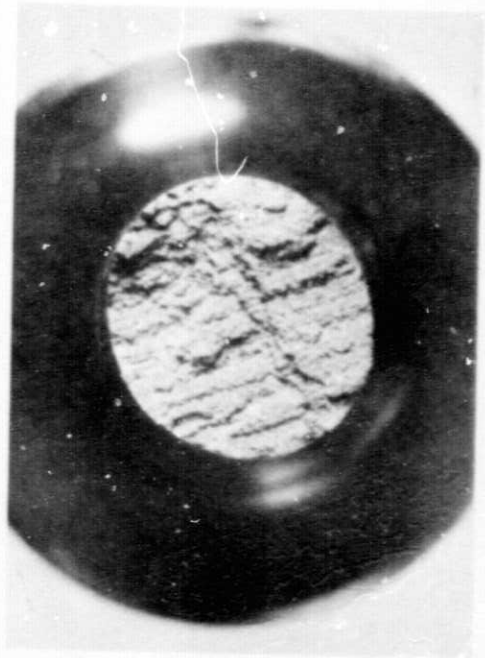
An external load cell was used to obtain cyclic load data, and the effect of friction at the load rod seals was determined during rig shakedown and calibration, and was accounted for. Electrical connections to the extensometer system, furnace, and thermocouples were made through the vessel wall via high-pressure bulkhead connectors.

Specimen axial strain was measured and controlled by means of a proximity probe extensometer. Split extensometer heads are attached to the specimen by mating the grooves in the heads with the integral collars on the specimen and bolting the assembly together. Collar deflection is measured and controlled via proximity probes attached to the open ends of the extensometer tubes so that the extensometer rod ends move relative to the probes as the specimen collars deflect (see Figure 14).



Mag: 1.40X FAL 53097

Final Machined Specimen
Showing TLP Bond Locations
and Fracture Location



Mag: 7X FAL 53096

Ovalized Fracture Face

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Figure 11. Final Machined and TLP Bonded LCF Specimen, and Ovalized Fracture Face for Single Crystal MAR-M-246 Hf (Specimen S/N B-1)

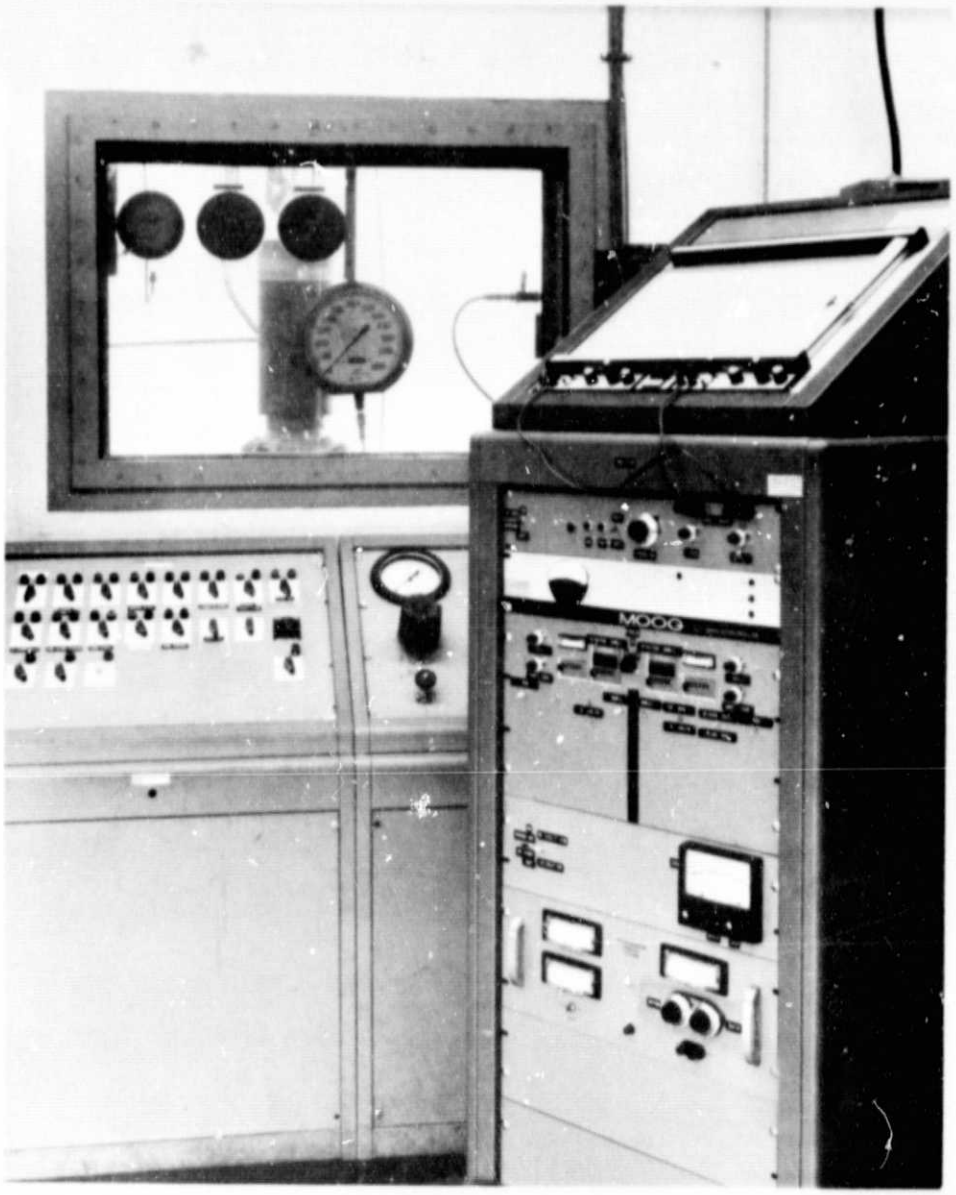

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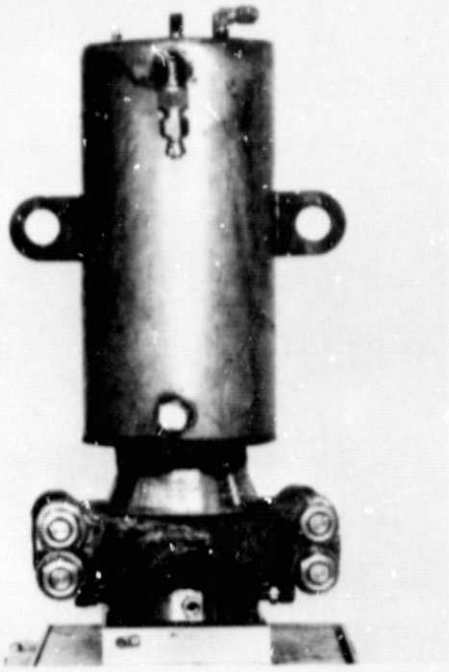
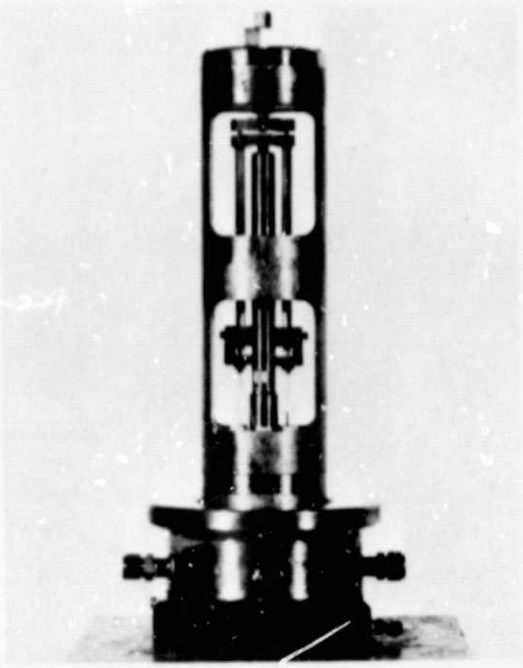
Figure 12. High-Pressure Environment Low-Cycle Fatigue Testing Machine, Environmental Controls, and Data Acquisition Equipment



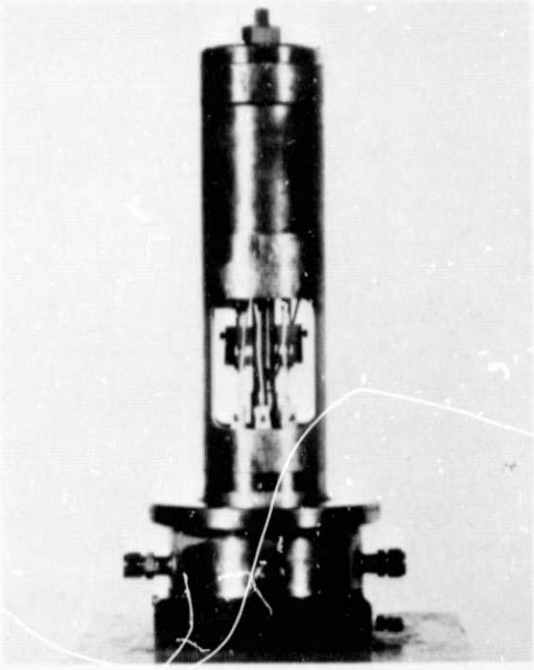
FAE 146129

a) Test Vessel Closed



FAE 146121

b) Test Vessel Open Showing Extensometer System



FAE 146122

c) Test Vessel Open Showing Furnace In Place

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Figure 13. Low-Cycle Fatigue High-Pressure Environmental Test Vessel

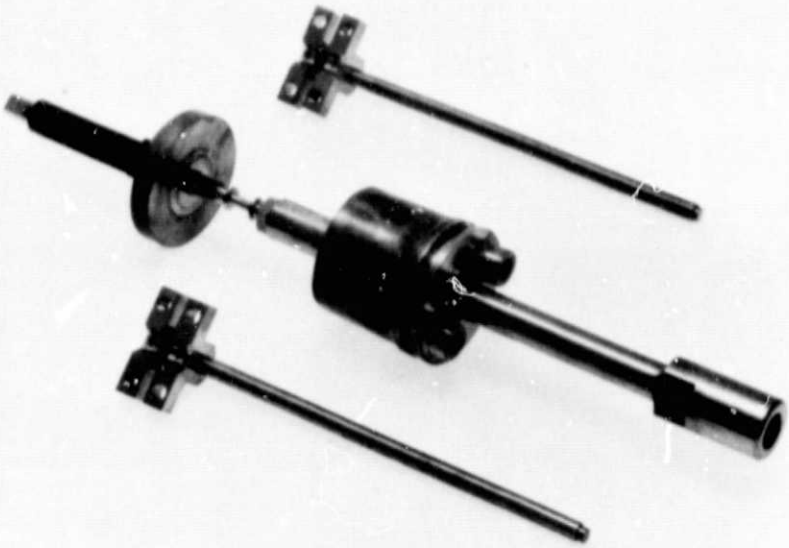

Figure 14. Open Extensometer Head Assembly, Low-Cycle Fatigue Specimen, and Retort Pistons

An x-y recorder was used for recording load vs strain plots at predetermined cyclic intervals during testing. The recorder was calibrated with the extensometer so that the ratio of specimen collar deflection to x-y recorder pen movements in the "x" direction was known. The "y" axis of the x-y recorder was calibrated with the load so that the ratio of specimen load to x-y recorder pen movement was known.

The command signal for the strain cycle was produced by a triangular wave signal generator with feedback from the extensometer output to complete the closed-loop-on-strain circuit necessary for the triangular strain wave form. The frequency and ramp of the triangular wave, and, therefore, the strain rate could be adjusted from 11×10^{-6} to 6×10^{-2} cm/cm/sec. Tests were conducted using ramp rates corresponding to a 0.067 Hz (4 cpm) cyclic frequency.

For elevated temperature testing, a two-zone resistance furnace with separate control systems for each zone was used. The furnace surrounds the specimen and fits within the frame of the pressure vessel. Thermocouples attached to the specimen gage section were used to monitor and control temperature during test. Temperature was controlled uniformly over the specimen gage section at 760°C (1400°F) for all tests using calibrated thermocouple, temperature readout, and control instrumentation.

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

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

1. Secure pressure vessel

2. Pressurize to 0.345 MPa (50 psig) with nitrogen gas and leak check

3. Vent system to 0.0345 MPa (5 psig)

4. Repressurize and vent with nitrogen gas (steps 2 and 3) two additional times

5. Pressure purge to 3.45 MPa (500 psig) with hydrogen gas

6. Evacuate pressure vessel, gas supply, and sampling system to an indicated absolute pressure of 0.00007 MPa (0.0096 psia, 500 microns)

7. Repressurize and evacuate system (steps 5 and 6) two additional times

8. Pressurize system to 3.45 MPa (500 psig) with hydrogen gas and obtain gas sample

9. Pressurize to 34.5 MPa (5,000 psig) with hydrogen gas and conduct test

10 Vent to atmospheric pressure, flow and pressure purge with nitrogen gas, open pressure vessel and remove failed specimen.

Tests were allowed to continue until complete specimen fracture. Total strain range, and the individual components of strain, elastic and inelastic, were measured and calculated from the x-y recorder hysteresis loops. The inelastic strain was measured at hysteresis loops exhibiting the minimum and maximum loop width. An average inelastic strain component was determined and this value, along with the total strain range and cyclic life were plotted in the data Figures 8, 9, and 10.

The data were statistically regressed for the directionally solidified material. This was the only case where sufficient data was available to make a regression. The form of the best-fit regression was $N = A \cdot \Delta\epsilon_t^B$, where N = cycles to failure, $\Delta\epsilon_t$ = total strain range, and A and B are constants. This is a straight line on a log-log coordinate system. The single crystal data could not be statistically regressed and a simple straight line connecting the pairs of data was used so that approximate comparisons could be made between the materials.

Elastic moduli were calculated based upon the elastic strain components and the measured cyclic load from the hysteresis loops.