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TUNGSTEN WIRE-REINFORCED SUPERALLOYS FOR 1093°C (2000°F) TURBINE BLADE APPLICATIONS

FINAL REPORT

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D.W. PETRASEK, Project Manager

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16. Abstract

Various combinations of fiber and matrix materials were fabricated and evaluated for the purpose of selecting a specific combination that exhibited the best overall properties for a turbine blade application. A total of seven matrix alloys, including Hastelloy X, Nimonic 80A, Inconel 600, Inconel 625, IN-102, FeCrAlY and FeNiCrAlY, were investigated reinforced with either 218CS tungsten, or W-Hf-C fibers. Based on preliminary screening studies, FeCrAlY, Inconel 600 and Inconel 625 matrix composites systems were selected for extended thermal cycle tests and for property evaluations which included stress-rupture, impact and oxidation resistance. Of those investigated, the FeCrAlY matrix composite system exhibited the best overall properties required for a turbine blade application. The W-Hf-C/FeCrAlY system was selected for further property evaluation. Tensile strength values of up to 724 MPa (105,000 psi) were obtained for this material at 982°C (1800°F) and 607 MPa (88,000 psi) at 1093°C (2000°F).

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October, 1979

Prepared Under Contract NAS 3-20084

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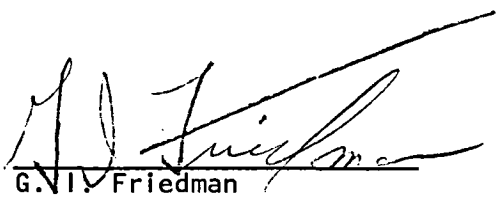
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

D. W. Petrasek, Project Manager

FOREWORD

This program was conducted by the TRW Materials Technology Laboratories under Contract No. NAS 3-20084 for the Lewis Research Center of the National Aeronautics and Space Administration. Mr. J. N. Fleck, Manager of the Metal Composites and Powder Technology Section, was the Program Manager. At the beginning of the program, Mr. W. D. Brentnall was the Project Engineer. Upon his departure, this responsibility was assumed by Mr. G. I. Friedman. Technical support was provided by Messrs. L. Chojnowski, Mr. Cooney, J. Sweeney, C. Tyndall, C. Harris and D. Engeman. This work was conducted within the TRW Materials Development Department, Dr. I. J. Toth, Manager, and was assigned the TRW Internal Project Number 512-002095-88.

The NASA Technical Manager was Mr. D. W. Petrasek.

Prepared by 
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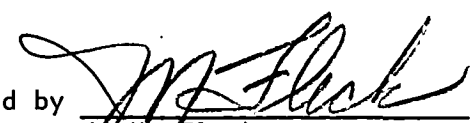
Approved by 
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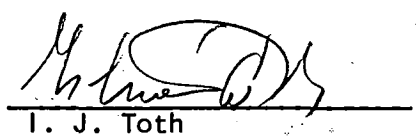
and 
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Department Manager

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF TABLES

1.0 SUMMARY

Various combinations of fiber and matrix materials were fabricated and evaluated for the purpose of selecting a specific combination that exhibited the best overall properties for a turbine blade application. A total of seven matrix alloys, including Hastelloy X, Nimonic 80A, Inconel 600, Inconel 625, IN-102, FeCrAlY and FeNiCrAlY, were investigated reinforced with either 218CS tungsten or W-Hf-C fibers. Based on preliminary screening studies, FeCrAlY, Inconel 600 and Inconel 625 matrix composites systems were selected for extended thermal cycle tests and for property evaluations which included stress-rupture, impact and oxidation resistance. Of those investigated, the FeCrAlY matrix composite system exhibited the best overall properties required for a turbine blade application. The W-Hf-C/FeCrAlY system was selected for further property evaluation. Tensile strength values of up to 724 MPa (105,000 psi) were obtained for this material at 982°C (1800°F) and 607 MPa (88,000 psi) at 1093°C (2000°F).

2.0 INTRODUCTION

The users of aircraft gas turbines exert a constant pressure for more efficient engines for both military and commercial airplanes. Engine efficiency can be improved by increasing the compressor pressure ratio and the turbine inlet temperature, but engines can achieve these advanced characteristics only if their materials of construction can withstand the increased mechanical loads and oxidation/corrosion burdens caused by the more severe operating conditions.

The superalloys, so-named because of their unique retention of high strength at temperatures close to their melting point, are capable of use at metal temperatures up to 975°C (1787°F). The more efficient, advanced engines referred to above require alloys that can operate at temperatures above 1100°C (2012°F) which is beyond the superalloys' stress-rupture capabilities. Refractory metals have more than sufficient high temperature strength but lack oxidation resistance. The concept of refractory metal-fiber-reinforced superalloys makes use of the best characteristics of these two classes of metals, wherein the superalloy is used as a ductile, oxidation-resistant, load-distributing matrix for the load-bearing, high-strength refractory-metal fibers.

Combinations of refractory-metal fibers and superalloys in the form of composites have been made by NASA and others and showed promise from stress-rupture and impact data that have been generated. For example, programs have been conducted at NASA-Lewis Research Center(1-4) which have demonstrated that composites could be produced having superior stress-rupture properties compared to conventional superalloys at temperatures of 1093° and 1204°C (2000° and 2200°F). Additionally, it was shown that, by careful fabrication techniques and selection of compatible matrix alloys, W/Ni alloy composites could be produced having adequate impact properties for turbine blade or vane applications. This work identified the relationships of matrix composition, compatibility (diffusional stability), elevated temperature strength and impact toughness in these systems.

A turbine blade material needs a combination of properties in addition to strength and impact resistance. Among these are oxidation resistance, thermal fatigue resistance, mechanical fatigue resistance and fabricability.

Thermally induced stresses are generated in turbine blades because of temperature gradients. These gradients change with time, leading to cyclic stresses and, hence, potential fatigue failures, particularly at stress concentrations such as cooling holes. Superimposed on these stresses, in the case of the composite, are internal stresses caused by the difference in expansion coefficient between the fibers and matrix. This is potentially the most serious limitation on composite usefulness. Consequently, much attention is being paid to improving the resistance of W/superalloy composites to damage due to thermal cycling.

An investigation concerned with the response of tungsten/superalloy composites to thermal cycling was conducted (ref. 5). The results obtained in this investigation indicated that thermal cycling can cause a number of effects in reinforced tungsten/superalloy composites which include: dimensional instability,

warpage, delamination, matrix debonding and matrix microcracking. The results also indicated that these effects may be controlled by proper selection of matrix strength, matrix ductility and the amount and distribution of reinforcement.

The purpose of this program was to continue development of tungsten/superalloy composites which have high potential for advanced aircraft engine applications such as turbine blades. Of major concern in this program was the composite's response to thermal cycling and much of the effort was devoted towards developing a composite having good thermal cycling properties. Based on preliminary screening studies, three tungsten/superalloy composite systems were selected for extended thermal cycle tests and for property evaluations which included stress rupture, impact and oxidation resistance. A specific fiber-matrix combination was then selected for further property evaluation.

3.0 PROGRAM PLAN

The objective of this program was to continue the development of a tungsten-wire/superalloy matrix composite for eventual application as an advanced turbine blade material. The matrix alloy composition and processing parameters were to be optimized to the point where the ability to withstand a 1000-cycle thermal fatigue test was demonstrated. The work was to result in development and characterization of a composite reinforced with the advanced, high strength W-Hf-C fiber. The experimental program was to be conducted in three tasks as specified in the following paragraphs.

3.1 Task I - Selection of Fiber/Matrix Combination

The work of this task was concerned with the investigation of various combinations of fiber and matrix materials for the purpose of selecting a specific combination that affords the best compromise in overall properties. Task I was divided into three phases: Selection of Material; Optimization of Fabrication Process; and Evaluation of Composite Properties.

3.1.1 Selection of Materials

a. Fiber

General Electric type 218CS tungsten lamp filament wire, 0.038 cm (0.015 in.) diameter was to be used for the majority of the composite specimens fabricated in Task I. W-Hf-C wire of similar diameter was to be used for the remaining composite specimens as specified below.

b. Matrix Materials

Nickel-base alloy matrix materials were to be selected based on their expected compatibility with tungsten wire, oxidation resistance, thermal fatigue resistance, impact resistance and elevated temperature strength properties. Five different nickel-base alloy compositions will be selected for the fabrication optimization phase.

3.1.2 Optimization of Fabrication Process

Composite specimens were to be fabricated by the diffusion bonding processes as described in NASA CR-134664[6]. The fabrication process was to be optimized by changing the time and pressure parameters to further inhibit fiber-matrix reaction and to obtain fully densified specimens. Specimens containing 35 volume percent 218CS tungsten wire and all of the selected matrix compositions were to be fabricated. It was planned to investigate three combinations of time, temperature and pressure for each composite system subject to the approval of the NASA Project Manager. The fabricated specimens were to be examined metallographically to determine the extent of fiber-matrix reaction and specimen densification. An optimized fabrication process would be selected for each matrix/fiber combination.

3.1.3 Thermal Fatigue Tests

Composite thermal fatigue specimens were to be fabricated utilizing the optimized fabrication process(es) and the five nickel base alloys and a fiber content of 50 volume percent of 218CS tungsten wire. The specimens were to be tested in thermal fatigue following the test procedure described in NASC Report TRW ER-7722-1[7] in which the specimen is heated by direct resistance heating and subjected to a programmed heating and cooling cycle (Gleeble Tests). The specimens would be cycled from 427 to 1093°C (800 to 2000°F) for 1000 cycles. Evaluation of the specimens after testing was to include visual inspections, measurement of geometric distortion and dimensional changes, optical microscopy, dye penetrant inspection for matrix cracking and delamination effects and metallographic investigations.

After the thermal fatigue tests had been evaluated, three matrix alloys would be selected from the compositions evaluated, but not necessarily restricted to this group, for evaluation of composite properties. In the event that three matrix alloys not included in the original five alloys were selected, fabrication optimization experiments would be conducted.

3.1.4 Evaluation of Composite Properties

Composite specimens were to be fabricated utilizing the optimized fabrication process and the selected nickel-base alloys and fiber contents of 35 and 50 volume percent of 218 CS tungsten wire. Specimens of the matrix materials without fibers would also be fabricated. The composite specimens were to be tested and characterized by the evaluations listed below. In addition, a limited number of composite specimens using the same matrix materials and fabrication procedure were to be made using W-Hf-C wire and having a fiber content of 35 and 50 volume percent. These composites would also be evaluated by the tests listed below:

a. Stress-Rupture Tests

1) Test Range: Stress-to-rupture in 100 hours was to be determined at 1093°C (2000°F).

2) Evaluation: Constant load stress-rupture tests were to be used. Fracture and cross sections of specimens would be examined after failure to determine the mode of fracture. The actual volume percent fiber content of the tested specimens would also be determined. At least two specimens were to be tested at each of three stresses used to determine the stress-to-rupture in 100 hours for specimens containing 218CS tungsten wire. At least one specimen was to be tested at each of three stresses used to determine the stress-to-rupture in 100 hours for specimens containing W-Hf-C wire.

b. Impact Tests

1) Test Range: Impact tests were to be conducted at room temperature, 149°C (300°F), 260°C (500°F) and 371°C (700°F).

2) Evaluation: Izod impact tests would be used. At least two notched specimens of each matrix material and of each 218CS tungsten fiber-matrix combination were to be tested at each temperature. At least one notched specimen of each W-Hf-C fiber-matrix combination would be tested at each temperature. Fracture surfaces and cross-sections of specimens were to be examined after failure to determine probable failure mode. The actual fiber content of tested specimens would be determined.

c. Elevated Temperature Compatibility

1) Test Temperature: 1093°C (2000°F).

2) Test Environment: Air or Inert Atmosphere.

3) Evaluation: At least three specimens of each fiber-matrix combination were to be held at test temperature for various times up to 1000 hours to determine the rate of reaction between the fiber and the matrix. Compatibility would be determined by metallographic examination. Specimens tested in air would have completely encased fibers to protect them from oxidation.

d. Oxidation Tests

1) Test Temperature: 1093°C (2000°F).

2) Evaluation: Static air oxidation tests were to be made at 1093°C (2000°F). Continuous weight-gain/weight-loss measurements would be made on at least two specimens of each matrix material and of each wire content and matrix composition for times up to 100 hours for specimens containing 218CS tungsten wire only. Evaluations would be made by metallographic examinations in the longitudinal and transverse directions of the specimens.

e. Thermal Fatigue Tests

The effect of a number of variables on the resistance of the composites to rapid thermal cycling was to be investigated. The test procedure would be as described in NASC Report TRW ER-7722-1[7] by which the specimen is heated by direct resistance heating and subjected to a programmed heating and cooling cycle (Gleeble Tests). The preliminary tests would be restricted to 100-cycle exposures from room temperature to 1093°C (2000°F). Evaluation was to include visual inspections, measurement of geometric distortion and dimensional changes, optical microscopy, dye penetrant inspection for matrix cracking and delamination effects, and metallographic and SEM investigations. The thermal fatigue tests to be conducted are as listed below:

1) Effect of Wire Distribution

The effect of wire distribution on thermal fatigue performance was to be determined using 218CS tungsten wire composite specimens. A minimum of three wire distribution patterns would be investigated. One matrix composition and fiber content was to be selected for this study subject to the approval of the NASA Project Manager. The distribution patterns were to be recommended by TRW for approval by the NASA Project Manager.

2) External Load Effects

All combinations of matrix and fiber were to be tested without any external load. One matrix composition and fiber composition would be selected subject to approval of the NASA Project Manager for determination of the effect of an external load. Three load conditions and one reinforcement level were to be selected subject to the approval of the NASA Project Manager.

Based on the preceding investigations, composite systems comprising two matrices and two selected fiber reinforcement levels would be further evaluated in thermal fatigue as detailed below. System selection was to be made with prior approval from the NASA Project Manager.

3) Advanced Thermal Fatigue Characterization Studies

The composite systems were to be subjected to 1000-cycle Gleeble tests from 427°C (800°F) to 1093°C (2000°F). Evaluations would be as detailed in Paragraph 3.1.4.e., Thermal Fatigue Tests.

4) Effect of Thermal Cycle on Stress-Rupture Strength

The effect of prior exposure of a composite to 1000 cycles from 427°C (800°F) to 1093°C (2000°F) on its 100-hour stress-rupture strength was to be determined. One matrix composition, one reinforcement level and one fiber composition would be selected subject to the approval of the NASA Project Manager. A minimum of three specimens were to be subjected to 1000-cycle Gleeble tests after which they would be tested in stress-rupture at 1093°C (2000°F).

3.2 Task II - Detailed Characterization of Selected Composite

3.2.1 Selection of Materials

a. Fiber

W-Hf-C wire, 0.038 cm (0.015 in.) diameter was to be used for the composites fabricated in Task II.

b. Matrix Material

One nickel-base alloy was to be selected as a matrix material subject to the NASA Project Manager's approval. Matrix material selection would be based

upon that matrix in Task I which imparted the best overall properties to the composite.

3.2.2 Characterization Studies

The following properties were to be determined for composites containing low fiber contents (about 25 to 35 volume percent) and high fiber contents (about 50 to 70 volume percent) and for matrix material without fibers, using a minimum of two specimens for each fiber-matrix combination and for each property determination:

a. Ultimate tensile and yield strength in the longitudinal and transverse direction of composite specimens at 982°C, 1093°C and 1204°C (1800°F, 2000°F and 2200°F).

b. Transverse shear strength at 982°C, 1093°C and 1204°C (1800°F, 2000°F and 2200°F).

c. Transverse shear modulus and longitudinal and transverse elastic modulus at 982°C, 1093°C and 1204°C (1800°F, 2000°F and 2200°F).

d. Coefficient of expansion in both the transverse and longitudinal direction from 20° to 1093°C (68° to 2000°F).

e. Thermal conductivity at 871°, 982° and 1093°C (1600°, 1800° and 2000°F).

f. Poisson's ratio at 871°, 982° and 1093°C (1600°, 1800° and 2000°F).

g. Fatigue endurance limit for 1×10^6 cycles at 1093°C (2000°F).

h. Creep strength to cause one percent total strain at 982° and 1093°C (1800° and 2000°F) for 100 hours.

3.3 Task III - Fabrication and Test of Blade Airfoil-Like Shape

The work of this task would be concerned with the demonstration of the fabrication of turbine blade airfoil-like shapes using the materials selected in Task II. Both unidirectional and cross-ply oriented fiber blade shapes would be fabricated and evaluated. Task III was to be divided into two phases: Blade Shape Fabrication Development and Property Evaluation Study. The matrix composition and fiber composition selected for Task II would be used in this task. The fiber content for the fabricated blade shapes was to be recommended by TRW based on the results obtained in Tasks I and II and would be subject to the NASA Project Manager's approval. The initial blade shape fabrication studies were to use 218CS tungsten wire as the reinforcing fiber. After a fabrication process had been developed, blade shapes were to be fabricated containing W-Hf-C wire as the reinforcing fiber.

3.3.1 Blade Shape Fabrication Development

A fabrication process was to be developed to fabricate composite material turbine blade airfoil-like shapes. The fabricationprocess would allow for cross-ply orientation of the fibers and was to utilize the diffusion bonding process developed and optimized in Task I. Airfoil-like blade shapes (without twist) were to be fabricated having a width of 0.4 to 0.5 cm (1 to 1.2 inches) and a length of 1.6 to 2 cm (4 to 5 inches). The cross section of the fabricated blades was to be 0.1 to 0.16 cm (0.25 to 0.4 inch) at the leading edge and 0.04 to 0.1 cm (0.10 to 0.30 inch) at the trailing edge.

3.3.2 Property Evaluation Study

A minimum of two blade airfoil-like shapes of each of three different fiber orientations would be fabricated. These were to have the dimensions stated in Task III, Para. 1 and have a volume percent fiber content of W-Hf-C wire recommended by TRW and subject to the NASA Project Manager's approval. These airfoils would be sectioned longitudinally into test size specimens and the following properties determined for each of the three different airfoil fiber configurations:

a. Stress-Rupture Tests

1) Test Range: Stress-to-rupture in 100 hours was to be determined at 1093°C (2000°F) in an inert atmosphere.

2) Evaluation: Constant load stress-rupture tests were to be used. Fracture and cross sections of specimens would be examined metallographically after failure. The actual volume percent fiber content of the tested specimen was to be determined. At lease two specimens from each airfoil fiber configuration would be tested at each of three stresses to determine the stress-to-cause rupture in 100 hours.

b. Impact Tests

1) Test Range: Impact tests were to be conducted at room temperature, 149°C (300°F), 260°C (500°F) and 371°C (700°F).

2) Evaluation: Izod impact tests would be used. At least two notched specimens from each airfoil fiber configuration were to be tested at each temperature and for each type airfoil-like blade described in Task III, Para. 2. Fracture surfaces and cross-sections of specimens were to be examined metallographically after failure. The actual fiber content of tested specimens would be determined.

c. Thermal Fatigue Tests

A minimum of one specimen from each airfoil configuration was to be tested. Thermal fatigue tests using the procedure described in NASC Report TRW

ER-7722-1[7] and Task I of this program in which the specimen is heated by direct resistance heating and subjected to a prolonged heating and cooling cycle (Gleeble Tests) were to be conducted. A 100-cycle exposure would be used for this evaluation. Evaluation was to include visual inspection, measurement of geometric distortion and dimensional changes, optical microscopy and dye penetrant inspection methods for matrix cracking and delamination effects and metallographic and SEM investigations.

4.0 PROCEDURES AND RESULTS

4.1 Task I

4.1.1 Material Selection and Procurement

As a result of discussions at NASA-Lewis, five matrix alloys were selected for Task I thermal fatigue screening. The matrix selection criteria were those properties expected to significantly affect the composite mechanical properties and thermal fatigue resistance; practical considerations were also included. Selection criteria were:

- High Temperature Strength and Ductility
- Thermal Expansion and Conductivity
- Modulus
- 1093°C (2000°F) Shear Rupture
- Shear Creep
- Oxidation/Sulfidation Resistance
- Compatibility with Reinforcing Fiber
- Workability
- Availability

In addition to one experimental alloy which was under development by NASA,* the following four commercial alloys were selected for initial consideration:

Commercial Designation	Composition, w/o
Inconel 600	Ni-15.5Cr-8Fe-0.5Mn-0.2Si-0.08C
Hastelloy X	Ni-22Cr-18.5Fe-9Mo-1.5Co-0.6W-0.5Mn, -0.5Si-0.1C
Nimonic 80A	Ni-19.5Cr-2.4Ti-1.4Al-0.3Mn-0.3Si-0.06C, -0.6Zr-0.003B
IN-102	Ni-15Cr-7Fe-3Mo, 3W, 3Cb-0.6Ti-0.4Al-0.06C, 0.03Zr, 0.005B-0.02Mg

As is noted in Tables I, II and III, these four commercial alloys represent a range of key properties.

The first four (nickel) alloys were obtained in wrought form, while the iron alloy would be obtained as powder, its exact composition being determined by NASA in-house work proceeding at the time. The source and form of these matrix alloys were:

---

* Fe-24.5Cr-20.5Ni-4.5Al-1.5W-1Y

TABLE I

Coefficient of Thermal Expansion, μm/m/°C (μin/in/°F)
for Candidate Matrix Alloys

	93°C	(200°F)	760°C	(1400°F)	871°C	(1600°F)
Nimonic 80A	12.6	(7.0)	14.8	(8.2)	15.5	(8.6)
Hastelloy X	13.9	(7.7)	15.9	(8.81)	16.2	(9.02)
Inconel 600	13.3	(7.4)	16.0	(8.9)	16.4	(9.1)
IN-102	13.2	(7.32)	15.8	(8.75)	--	--

TABLE II

Tensile Yield Strength (0.2% Offset) in MPa (ksi) for Candidate Matrix Alloys

	RT	538°C	(1000°F)	650°C	(1200°F)	760°C	(1400°F)	870°C	(1600°F)	981°C	(1800°F)
Inconel 600	285 (41)	220	(32)	205	(30)	180	(26)	75	(11)	41	(6)
Hastelloy X	360 (52)	290	(42)	275	(4)	260	(38)	180	(26)	110	(16)
IN-102	505 (73)	400	(58)	400	(58)	385	(56)	200	(29)	-	-
Nimonic 80A	620 (90)	530	(77)	550	(80)	505	(73)	260	(38	62	(9)

TABLE III

Tensile Ductility (% Elongation) for Candidate Matrix Alloys

	RT	538°C	(1000°F)	650°C	(1200°F)	760°C	(1400°F)	870°C	(1600°F)	981°C	(1800°F)
Inconel 600	45		41		49		70		80		115
Hastelloy X	43		45		37		37		50		45
IN-102	47		48		64		110		110		--
Nimonic 80A	39		37		21		17		30		--

Inconel 600	0.25 mm (0.010")	Sheet - Ulbrich and Special Metals
Hastelloy X	0.25 mm (0.010")	Sheet - Atek Metals Center
Nimonic 80A	30.5 mm (1.2")	Dia. Bar - INCO
IN 102	19 mm (3/4")	Dia. Bar - INCO
Fe-Cr-Ni Alloy		Remelt Ingot - TRW Powder - HMI

The refractory-metal fiber selected for Task I was 0.38 mm (0.015 inch) Grade 218 (lamp-filament grade CS (cleaned and straightened) tungsten.

TZM molybdenum hot pressing die materials were procured from AMAX Specialty Metals in the form of arc melted, extruded and forged bar. The die bases, inserts and punches were machined using conventional machining methods.

4.1.2 Preliminary Process Optimization Studies

4.1.2.1 General Fabrication Approach

Fibers were collimated by drum winding with a fugitive polystyrene binder to form a mat. These mats were cut to size and sandwiched between either foils or powder cloth (Fe-based alloy only) of matrix alloy of appropriate thickness. These assemblies were then bonded in a TZM channel die like that shown in Figure 1. Prior to insertion of the filament-foil assemblies, the die was coated with boron nitride to prevent bonding to the workpieces.

The channel die was positioned inside an induction coil within the vacuum chamber shown installed in the 75 ton hydraulic press in Figure 2. The chamber was evacuated, purged for 3 minutes with argon, and then maintained under a flowing 14 l/sec (30 ft^3/min) hydrogen atmosphere. Normal heating time to bonding temperature was one hour. During that period, a clamp load was maintained. When the desired bonding temperature was reached, the load was increased to the full value. Upon completion of the bonding cycle, the system was cooled under argon.

4.1.2.2 Optimization Studies

Initial variables in the optimization cycle included time-temperature-pressure cycles, surface preparation methods, and surface bonding aids for the four wrought alloys. The latter were considered necessary because of the stable, tenacious oxide films which are characteristic of such alloys. Actual parameters are listed in Table IV.

Typical microstructures of test panels are shown in Figures 3 through 7.

Major observations and conclusions are noted below.

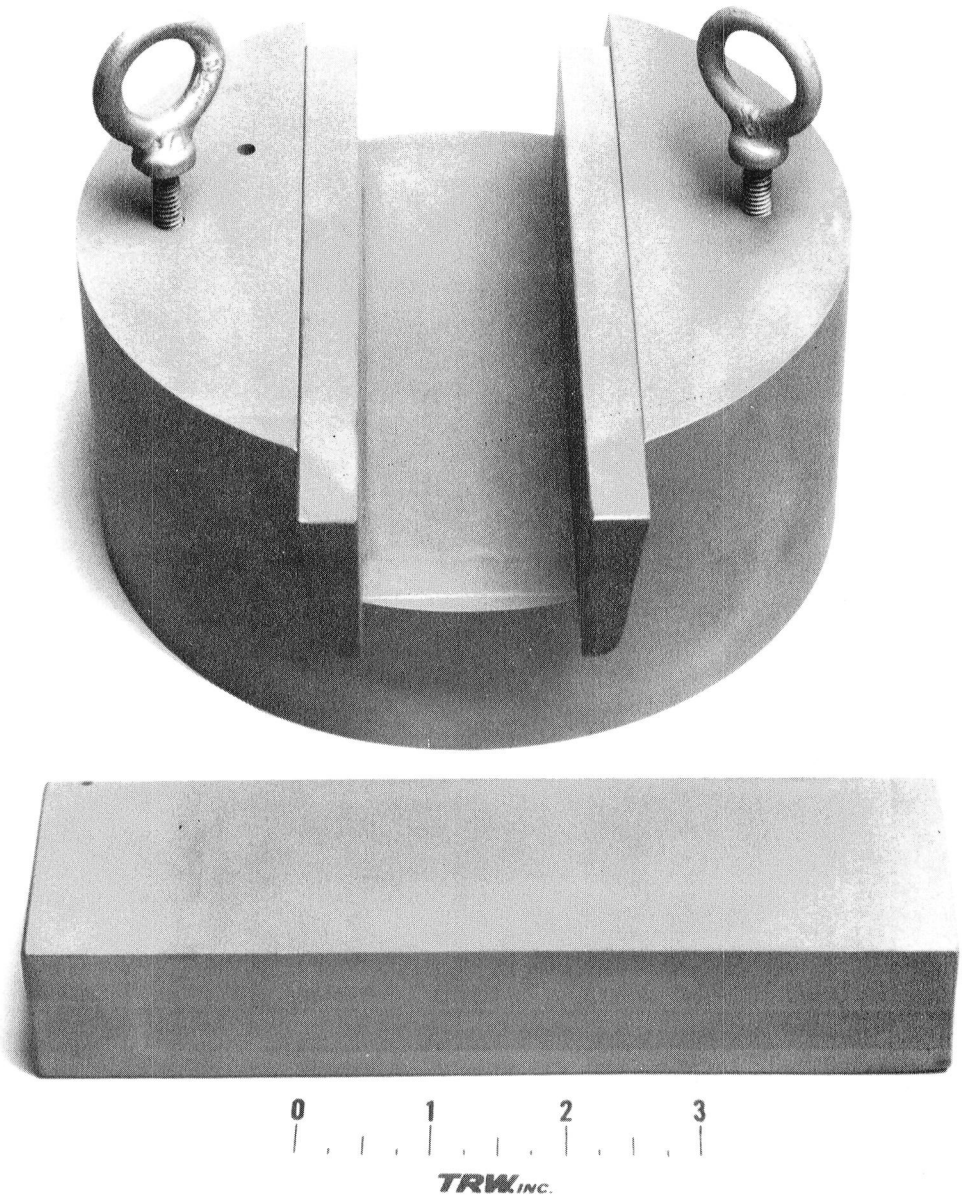


Figure 1. Typical TZM Channel Die.

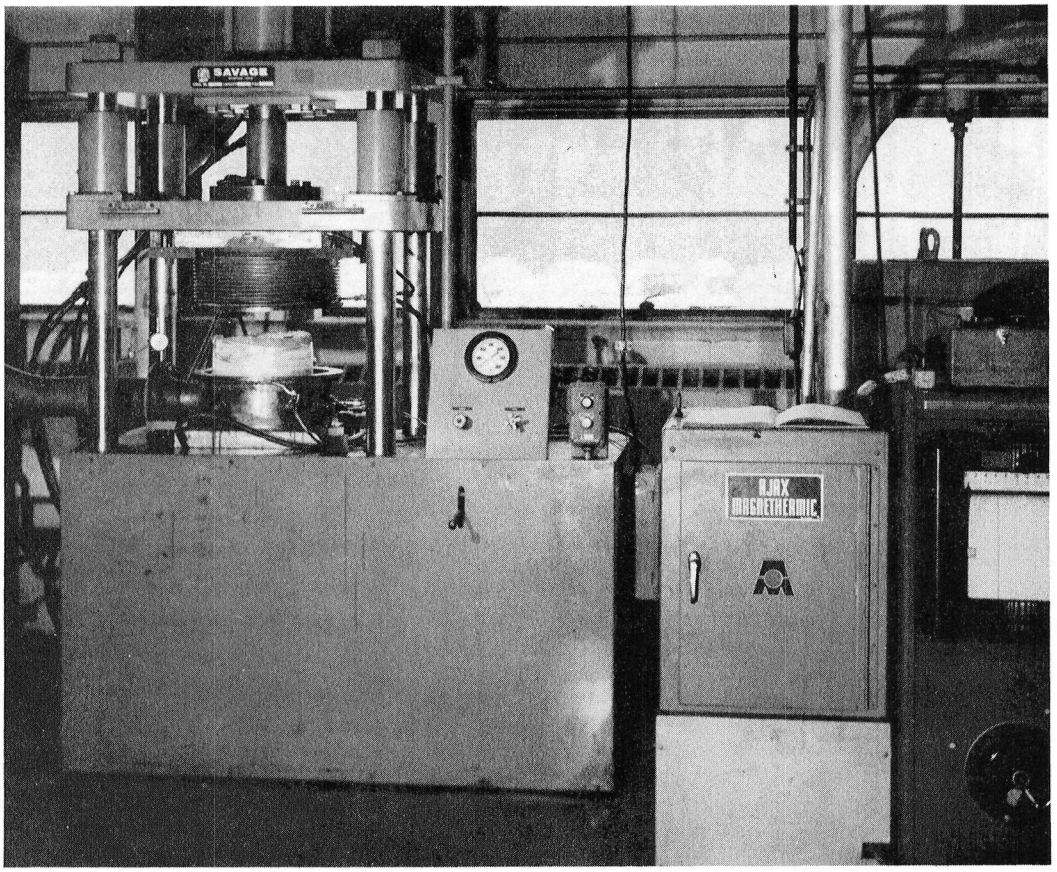


Figure 2. Hydraulic Press Equipped for Composite Bonding.

TABLE IV

Initial Processing Variables

A. Surface Preparations

1) 92 v/o HCl, 3 HNO_3, 5 H_2SO_4
2) 40 v/o HNO_3, 20 HF, 40 H_2O
3) 60 v/o H_3PO_4, 20 HCl, 20 H_2SO_4 (add H_2O_2 to start reaction)
4) 12 ppg* HNO_3, 66 HCl, 3.5 H_3PO_4 plus 1.6 kg (3.6 lb) $FeCl_3$ and 1.5 ml (0.05 oz) wetting agent
5) Concentrated HCl + several drops of H_2O_2
6) Oakite 33 (Commercial Cleaner)

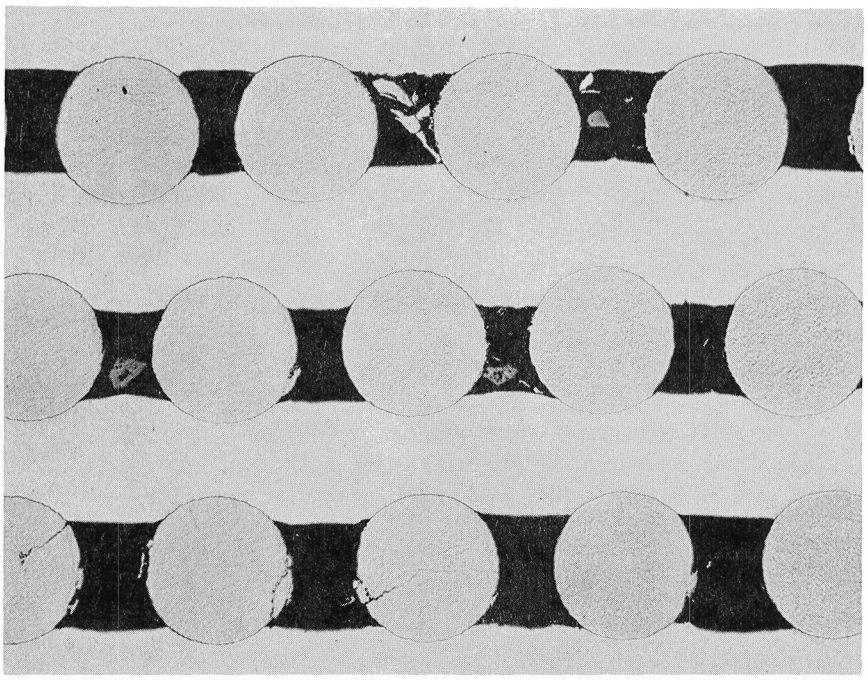
B. Bonding Aids

1) Electroplated Nickel
2) Electroplated Copper
3) Brushed-on Powder Layer (Ni-25W-15Cr-2Ti-2Al)
4) Powder Cloth Layer (Ni-25W-15Cr-2Ti-2Al)

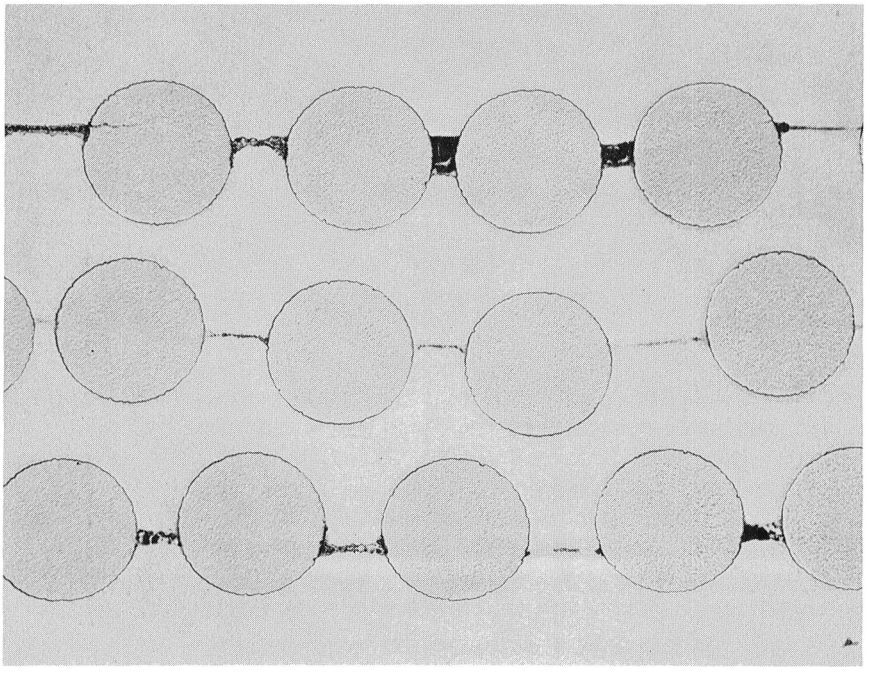
C. Bonding Cycles

1) Heat to 1010°C (1850°F), apply 138 MPa (20 ksi), hold 30 minutes.
2) Heat to 1093°C (2000°F), apply 138 MPa (20 ksi), hold 30 minutes.
3) Heat to 1010°C (1850°F), apply 193 MPa (28 ksi), then continue heating to 1093°C (2000°F), hold 20 minutes.
4) Heat to 1121°C (2050°F), apply 193 MPa (28 ksi), hold 20 minutes.
5) Heat to 1121°C (2050°F), apply 193 MPa (28 ksi), hold 90 minutes.

* Parts per gallon.

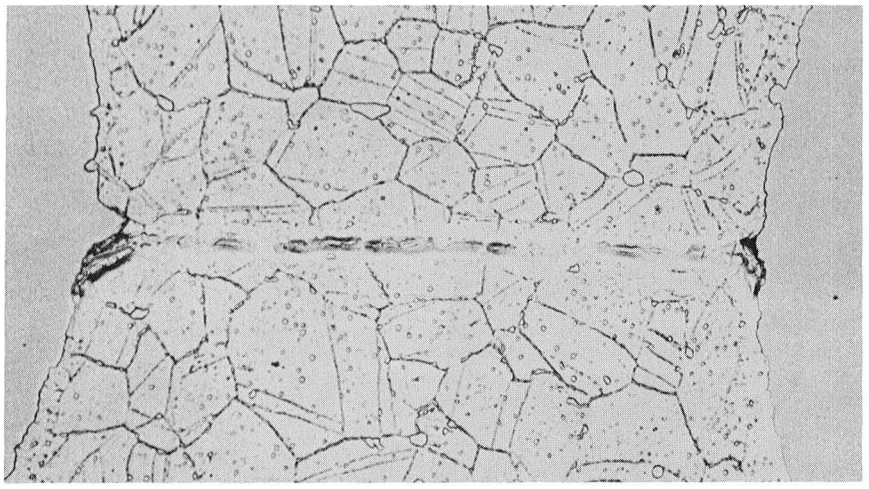


1850°F, 20 ksi, 30 Minutes 50X



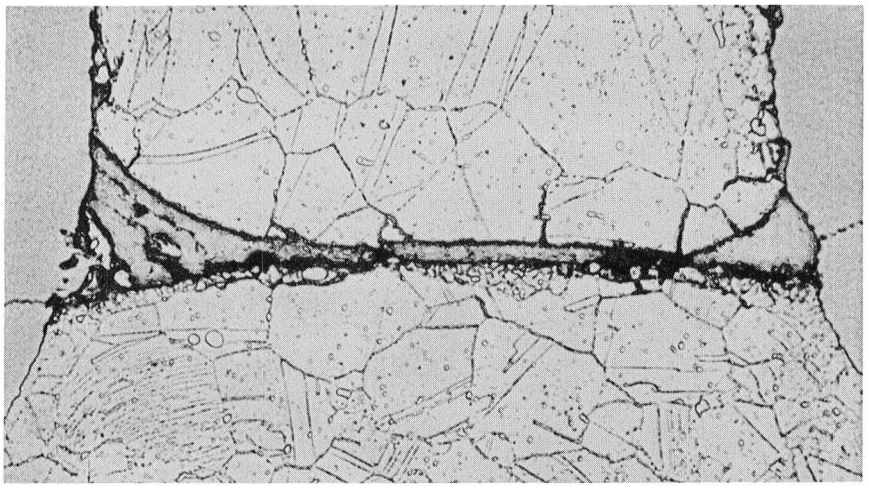
2050°F, 28 ksi, 20 Minutes 50X
(Cu-Plated Hastelloy X Foils)

Figure 3. Effect of Pressing Parameters on Consolidation of W/Hastelloy X Panels. 50X



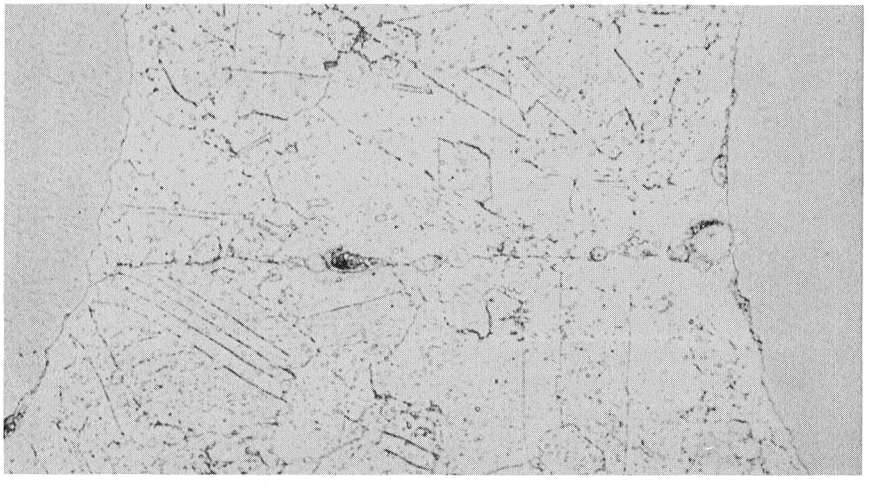
Fabrication Parameters
2050°F, 28 ksi, 20 Minutes.

Ni Plated 50X



Fabrication Parameters
2050°F, 28 ksi, 20 Minutes

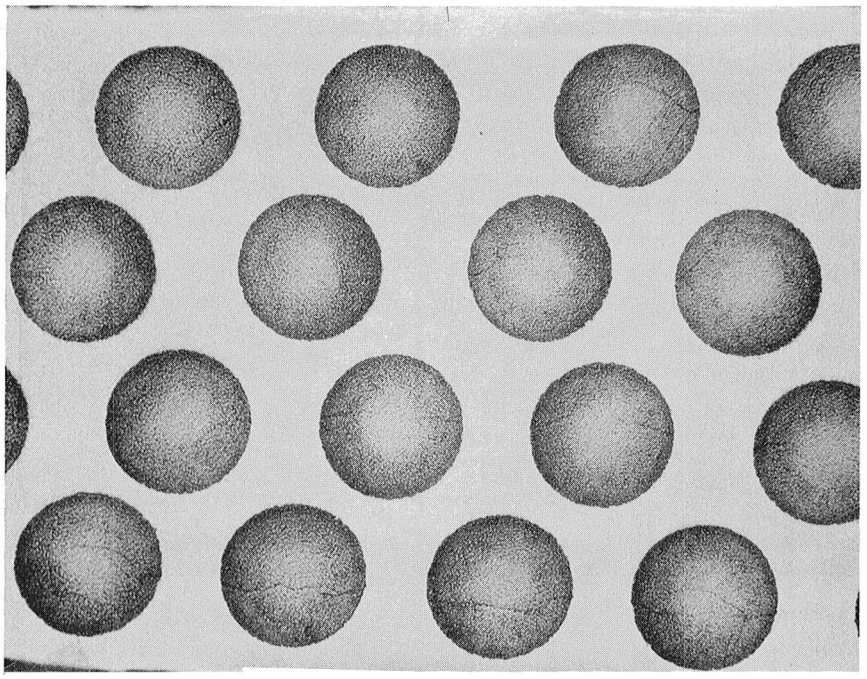
One Foil Cu Plated 500X



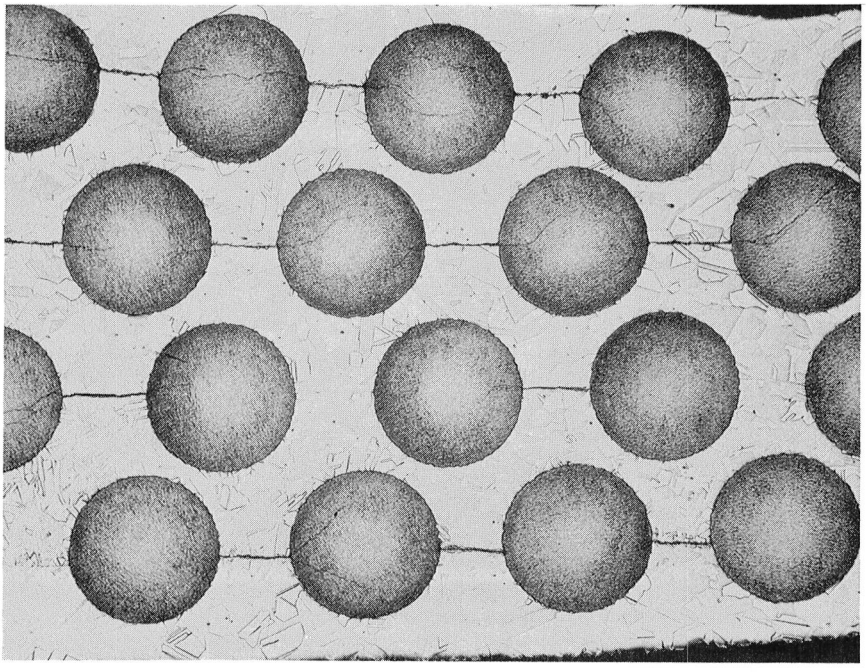
Fabrication Parameters
2050°F, 28 ksi, 90 Minutes.

Brush-Coated With 25 W Alloy 500X

Figure 4. Matrix/Matrix Interfaces in W/Hastelloy X Panels Consolidated at 1121°C, 193 MPa (2050°F, 28 ksi) for Different Times.

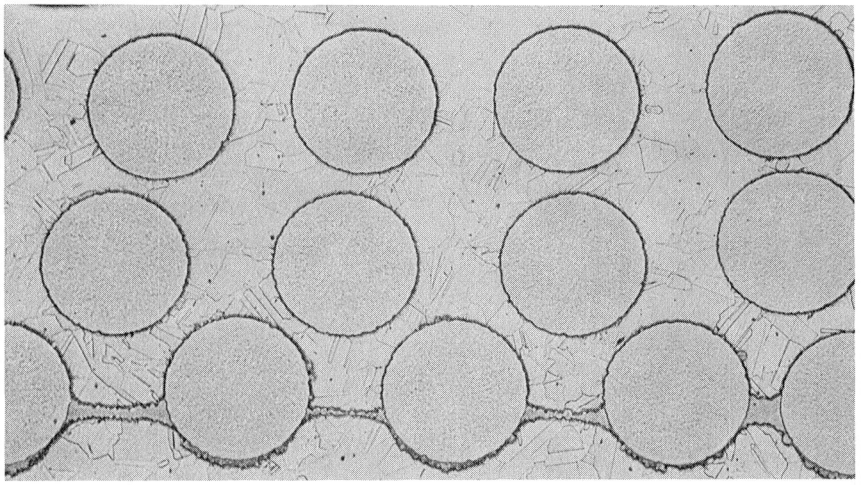


Matrix Unetched 50X

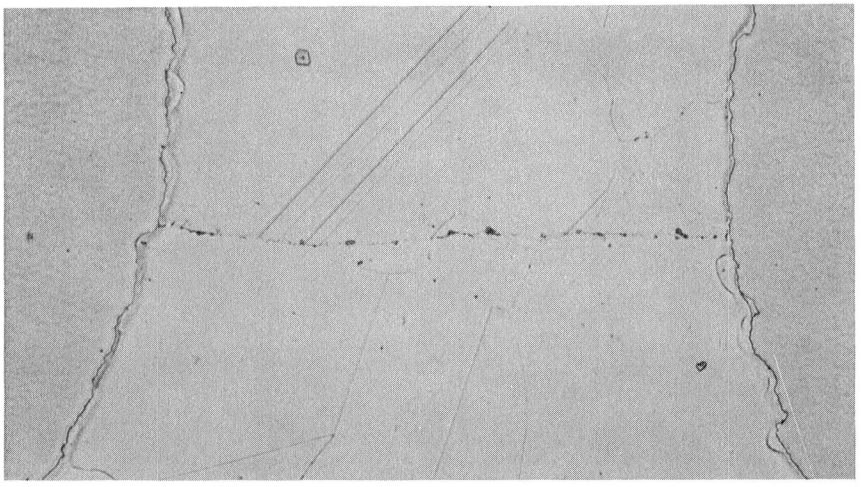


Matrix Etched 50X

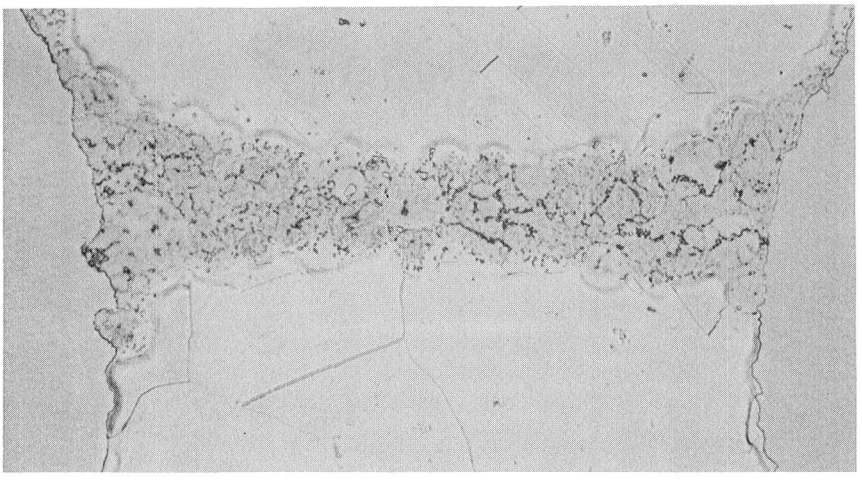
Figure 5. W/Nimonic 80 A Panel Fabricated at 1121°C, 193 MPa (2050°F, 28 ksi), 20 Minutes. Fully Consolidated but not Bonded - Foils Chemically Etched Prior to Panel Fabrication. 50X



50X

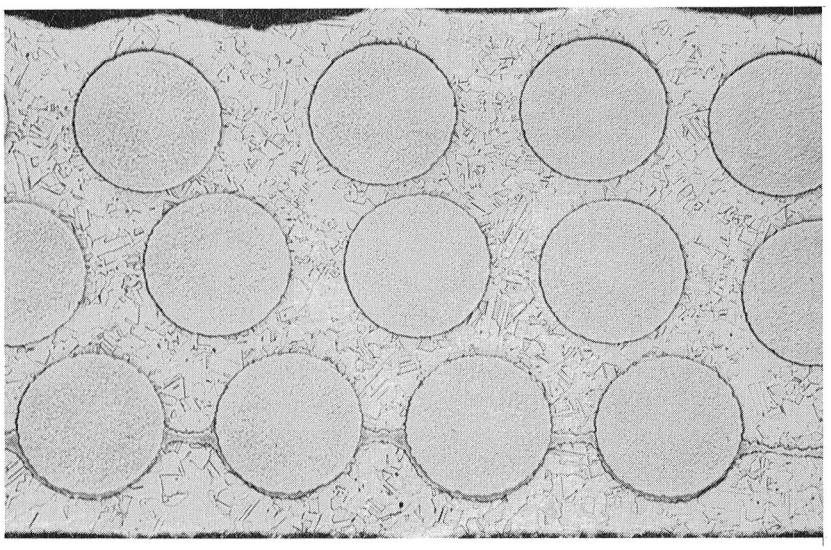


Etched Foils 500X



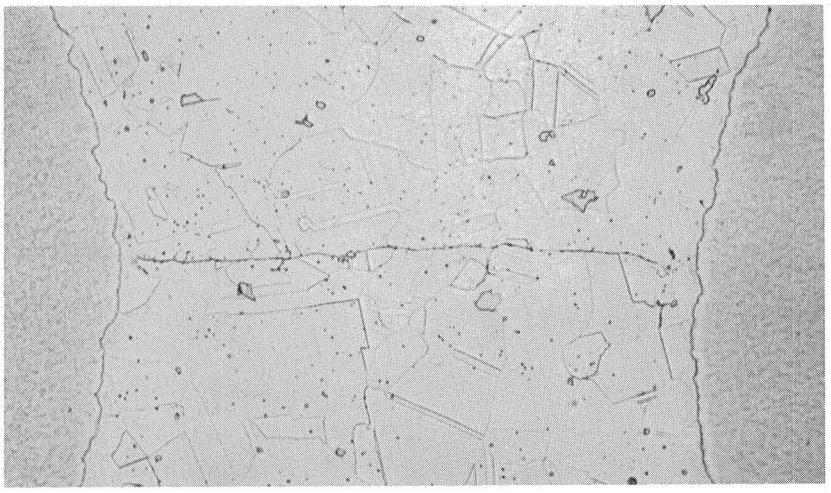
One Foil Brush Coated With 25W Alloy 500X

Figure 6. W/Inconel 600 Panel Fabricated at 1121°C, 200 MPa (2050°F, 29 ksi), 90 Minutes.

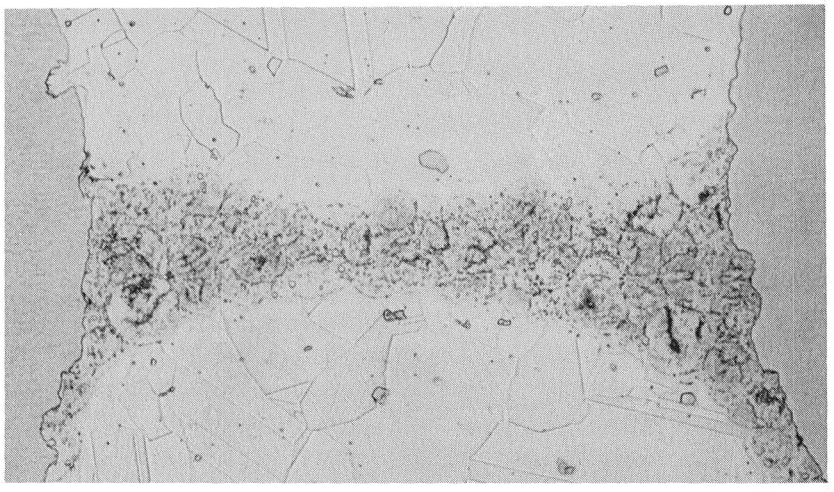


Etched Interface

Brush-Coated with Ni-25W-15Cr-2Ti-2Al 50X



Etched Interface 500X



Brush-Coated Interface 500X

Figure 7. W/IN-102 Panels.

Surface Preparation

The simplest surface preparation, viz., an Oakite chemical cleaning, appears to give acceptable matrix/matrix bonds with Inconel 600 and IN-102. Both Hastelloy X and Nimonic 80A require a surface conditioning. Preparation 3 (from Table IV) is recommended for the former and 4 for the latter. Prepration 3 is also effective on Inconel 600 and IN-102, but does not apply to the Fe-based alloy which was in powder form.

Bonding Aids

Nickel plating and nickel-alloy-powder coatings or thin layers of powder cloth* appeared to promote better matrix/matrix bonding in all four systems. The alloy powder bonding aid approach would appear to be more desirable than nickel plating in terms of fiber/matrix reaction and cost effectiveness. This approach also has some potential for improving fiber/matrix compatibility during the bonding cycle. No bonding aid is necessary for the powder cloth system (Fe-based alloy).

Bonding Cycle

Hastelloy X was the most difficult to consolidate (strongest at pressing temperature) matrix alloy and required long (60 minute) cycles for full consolidation at 1121°C (2050°F) and 200 MPa (29 ksi). The Nimonic 80A, IN-102 and Inconel 600 matrices appeared to be fully consolidated after a 20-minute cycle. However, a 60-minute cycle is suggested for all the Ni-based systems. A 30-minute cycle is adequate for the Fe-based alloy matrix at a temperature of 1093 to 1121°C (2000 to 2050°F).

4.1.3 Thermal Fatigue Tests

4.1.3.1 Specimen Fabrication

The five matrix alloys were made into composite panels using the Grade 218CS tungsten wire. The Inconel 600 and IN 102 composites were made from foil/filament lay-ups using cleaned and etched foils. In addition, Inconel 600 specimens were also prepared using foils brush-coated with -500 mesh Ni-25W-15Cr-2Ti-2Al powder. The Hastelloy X and Nimonic 80A foil/filament specimens were made only with NiCrAlY powder-coated foils. The FeNiCrAlY specimens were produced using powder cloth. Five-ply panels were manufactured at 1093-1121°C (2000-2050°F), 172-207 MPa (25-30 ksi), 45-90 minutes, except for the FeNiCrAlY composite panels, for this the bonding conditions were 1093°C, 172 MPa (2000°F, 25 ksi), 30 minutes. Rectangular specimens 13 x 0.6 x 2.5 cm (5 inches x 0.25 inch x t [$\sim$0.095 inch])were machined from the 3 cm (1.2 inch)-wide panels.

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* TRW uses a proprietary process for making cloth from metal powder. The cloth binder is driven off during the early stages of hot pressing, without reaction with the powder particles.

4.1.3.2 Testing

Thermal fatigue testing was performed in TRW's Gilmore Universal Testing Facility, shown in Figure 8. As is shown in Figure 9, thermocouples were attached directly to the specimen for temperature control during the test. The specimens were fixtured such that the distance between grips was 1.2 cm (3.1 inches). The specimens were resistively self-heated by passing an electrical current through the water-cooled grips. In order to maintain a locking load on the wedge-type grips, a slight tensile load of $\leq$10.3 MPa ($\leq$1.5 ksi) was maintained. The test atmosphere was argon. A representative time-temperature cycle is shown in Figure 10. Because water-cooled grips were used, there was a temperature gradient across the specimen as is shown in Figure 11. In subsequent discussions, reference is made to both the center portion of the specimen which experienced a uniform maximum temperature of 1093°C (2000°F) and the transition zone which experienced a gradient from ~300 to 1093°C (~570 to 2000°F).

All five composite systems survived 1000 cycle tests without catastrophic failure. However, only the W/Inconel 600 specimen survived without evidence of intermediate-temperature-zone cracking or appreciable internal damage. Table V summarizes the dimensional measurement data and visual observations. The Inconel 600 and FeNiCrAlY composites were the best in terms of dimensional stability.

Photographs of the specimens after testing are shown in Figures 12, 13, 14 and 15. Surface cracking in the transitional temperature zone accompanied by a growth in width at the gage section is evident in the Hastelloy X and Nimonic 80A specimens. No cracks were visible in the IN-102 specimen but some broadening of the width dimensions and surface roughening were observed. No significant macroscopic changes occurred in the Inconel-600 specimen.

There was no macroscopic distortion in the FeNiCrAlY specimen, but small surface cracks were visible over most of the surface. Few of these cracks propagated very deeply into the specimen in the hot zone. However, internal cracks and propagation along fibers did occur in the transitional temperature region. Two factors which may have influenced the behavior of this sample must be noted; these are:

1. Non-optimum powders were used because of procurement difficulty; the particle distribution included significant amounts of needle- and flake-like particles which worked through the 100 mesh (150 μm) screen.

2. The specimen slipped out of the grip after 250 cycles, which could have resulted in some arcing as electrical contact was broken. Visible examination of the specimen did not indicate a problem, however, and the test was continued.

These specimens were sectioned at two locations, viz., in the center of the hot zone and about 2.5 cm (~1 inch) away; this corresponded to a very severe

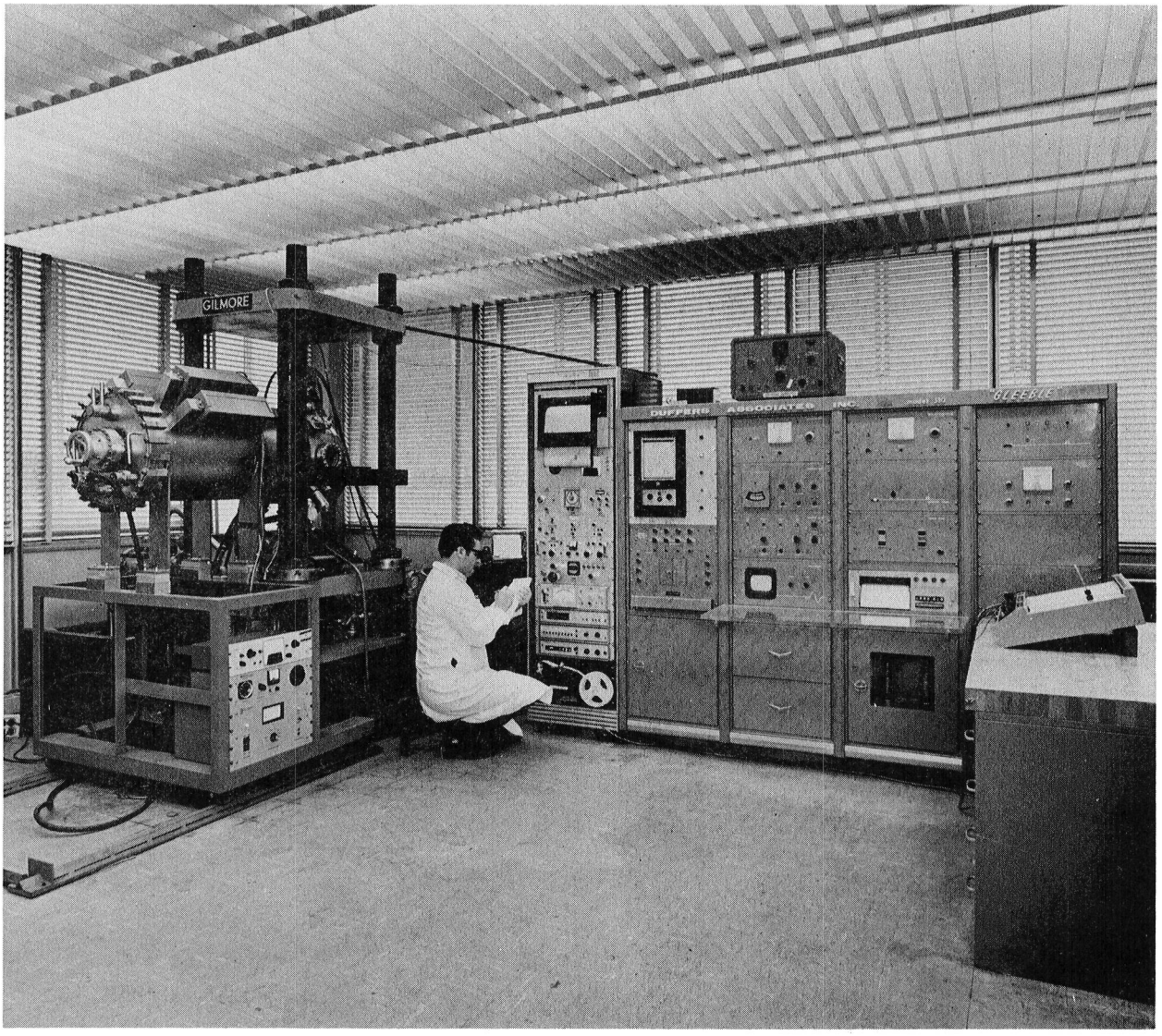


Figure 8. Gilmore Universal Testing Machine.

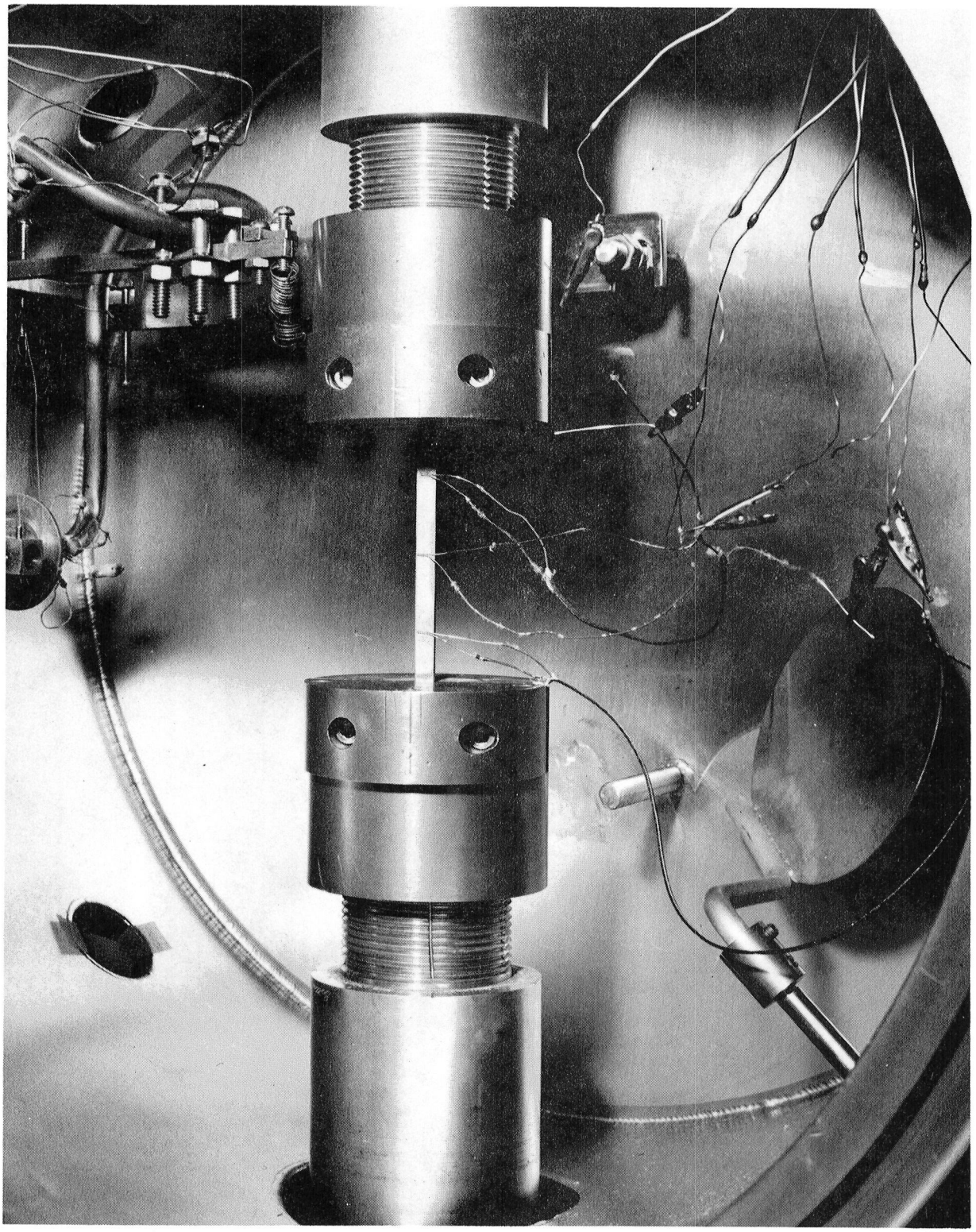


Figure 9. Close-Up View of Thermal Fatigue Specimen Mounted Between Water-Cooled Grips in the Test Chamber. Three Thermocouples are Welded on Specimen.

5748 c

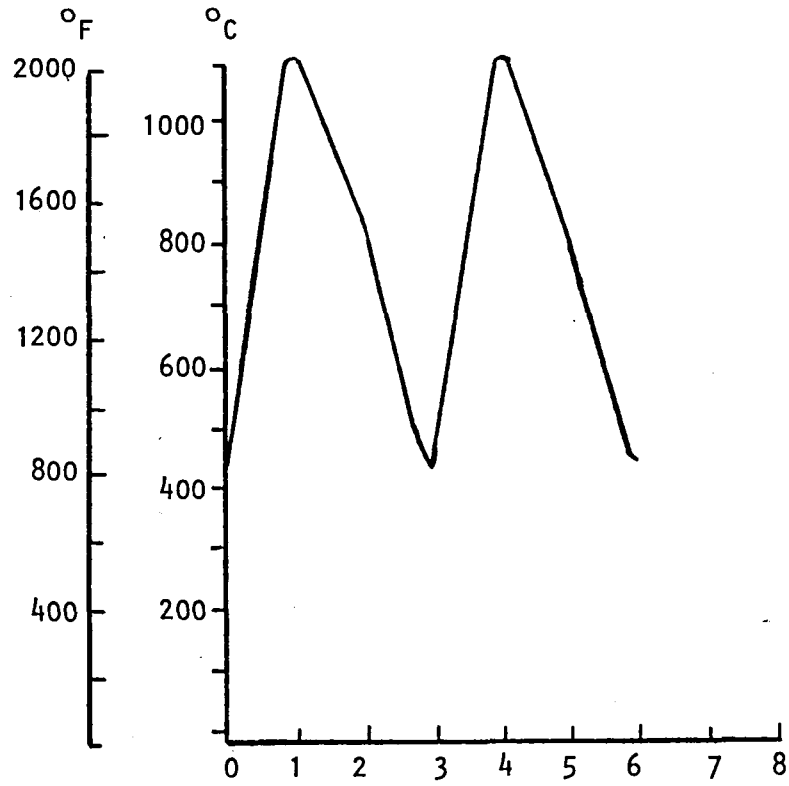


Figure 10. Time-Temperature Cycle Used in Thermal Fatigue (LCF) Tests.

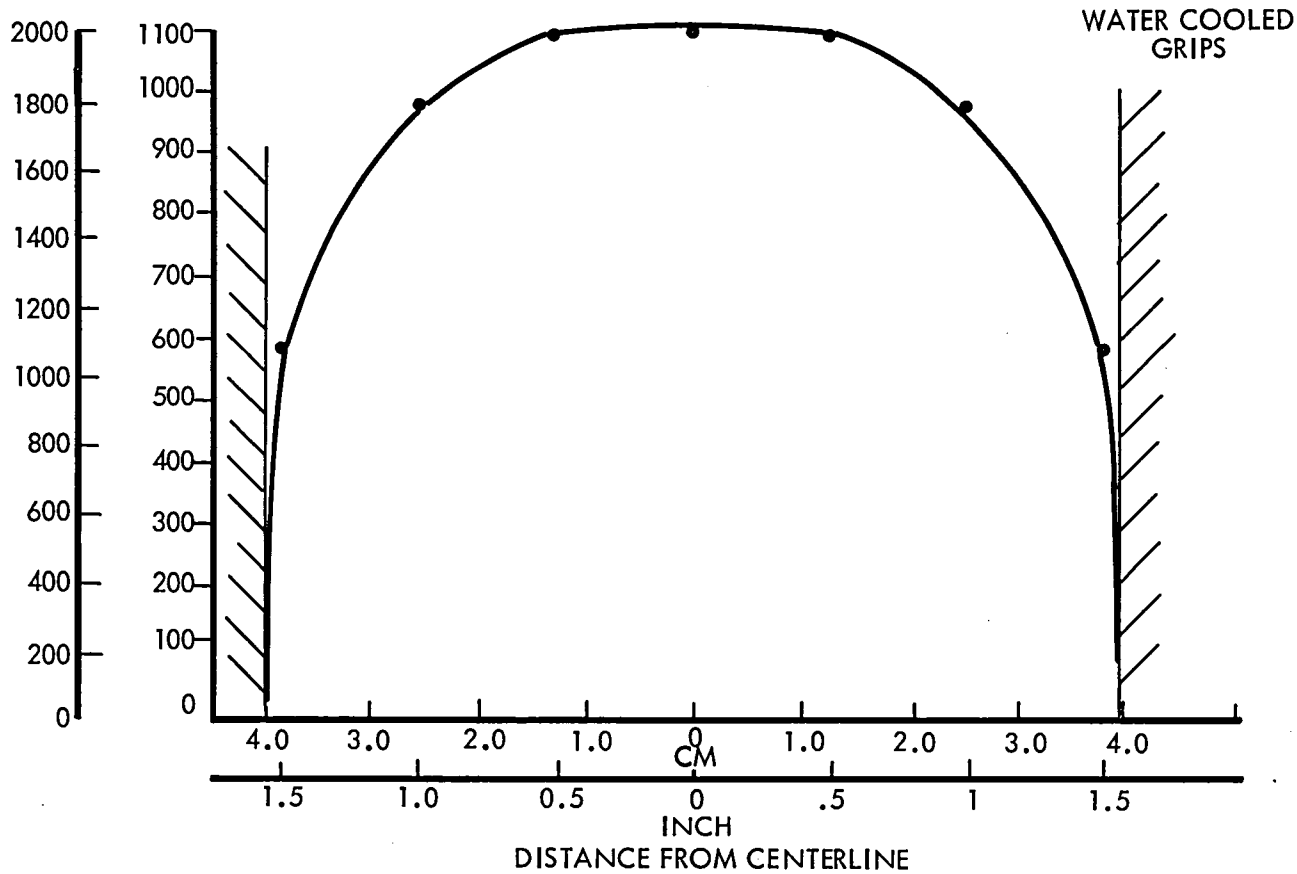


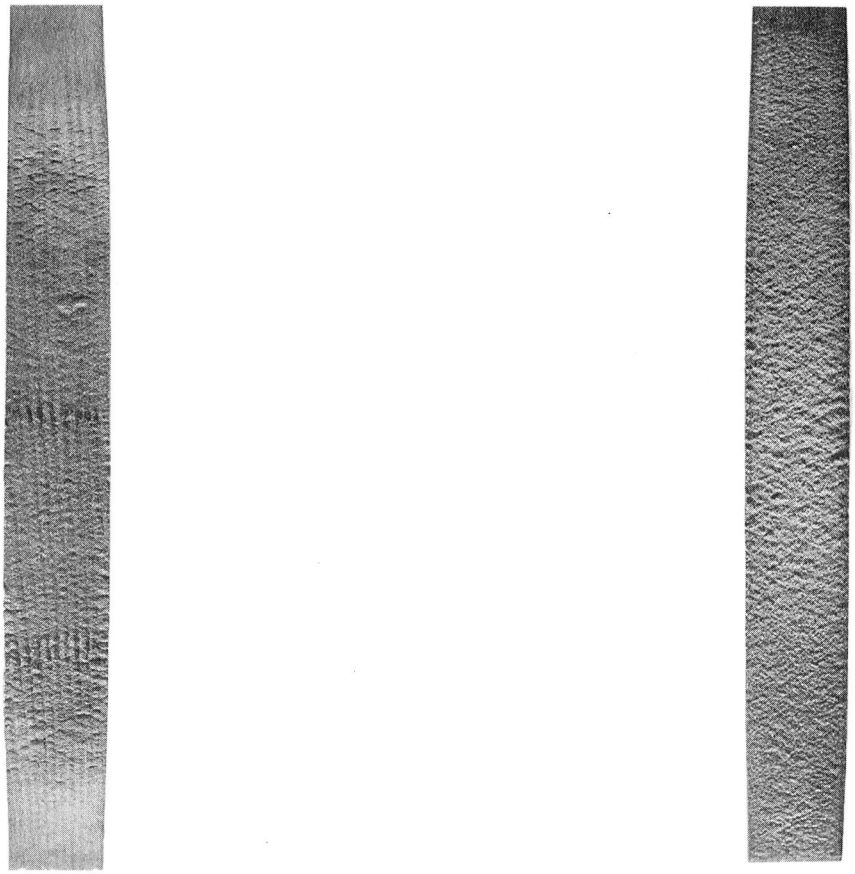
Figure 11. Temperature Gradient Along Specimen During Thermal Fatigue Test.

TABLE V

Dimensional Changes in Thermal Fatigue Specimens

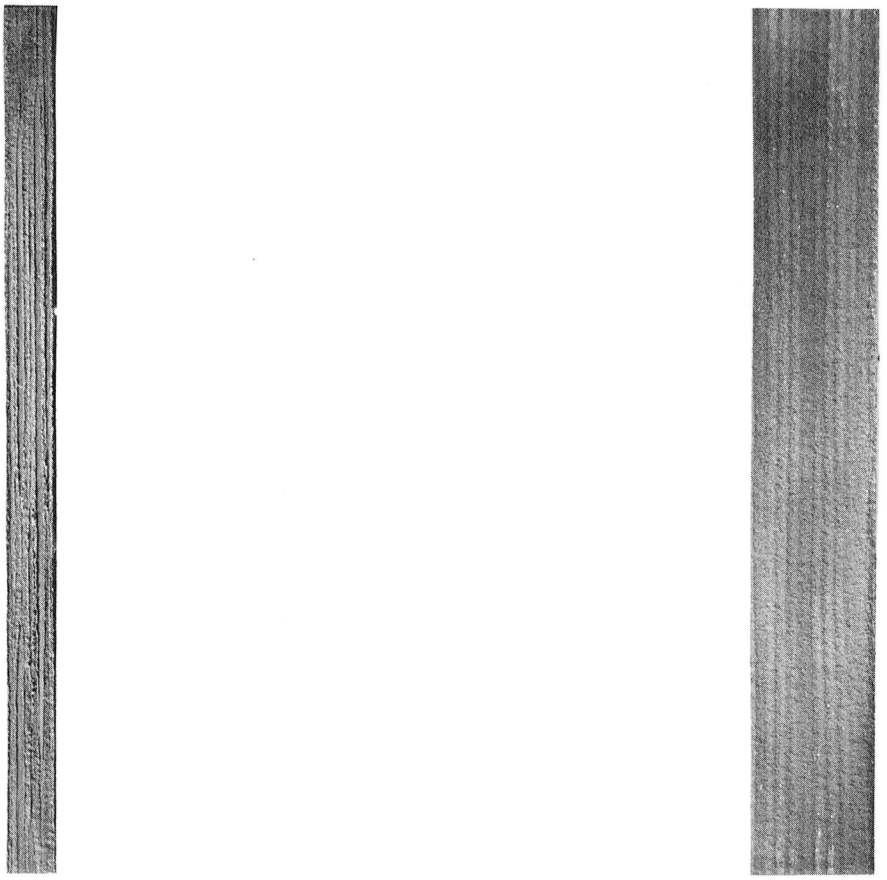
System	Δℓ (gage) Cm (mils)	Δℓ (gage) %	Δℓ (overall) Cm (mils)	Δℓ (overall) %	ΔW* Cm (mils)	ΔW* %	Δt* Cm (mils)	Δt* %	Observations
W/Inconel 600	.03 (11.8)	2.7	-.00127 (-1.0)	-0.1	.0076 (3.0)	1.2	.001 (4.0)	3.8	No surface cracks or distortion.
W/IN-102	.0063 (2.5)	0.51	-.001 (-4.0)	-0.084	.0056 (22)	8.8	.00114 (4.5)	4.7	Slight surface cracking Noticeable width increase.
W/Hastelloy X	.0493 (19.4)	4.2			.0064 (25)	9.9	.0013 (5)	5.3	Surface cracks visible in intermediate temperature zone. Noticeable width increase.
W/Nimonic 80A	-.0135 (-5.3)	-1.2			.0027 (10.7	4.4	.0078 (3.1)	3.5	Surface cracks visible over entire hot zone.
W/FeNiCrAlY	.0013 (0.5)	0.1	0	0	.001 (4.2)	1.7	.0012 (4.8)	4	Surface cracks visible. No distortion or dimensional change.

* Readings at center of gage.



W/Hastelloy X 2.5X W/IN-102

Figure 12. Thermal Fatigue Specimens after 1000 Cycle Test, 2.5X.



Edge View 2.5X Surface

Figure 13. W/Inconel 600 Specimen after 1000 Cycle Thermal Fatigue Test, 2.5X.

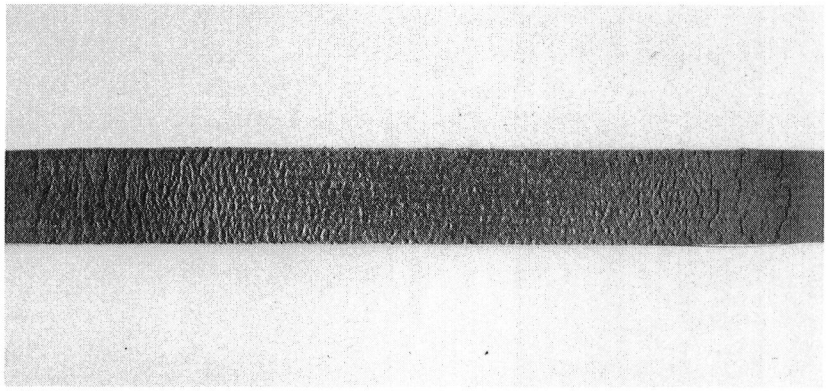
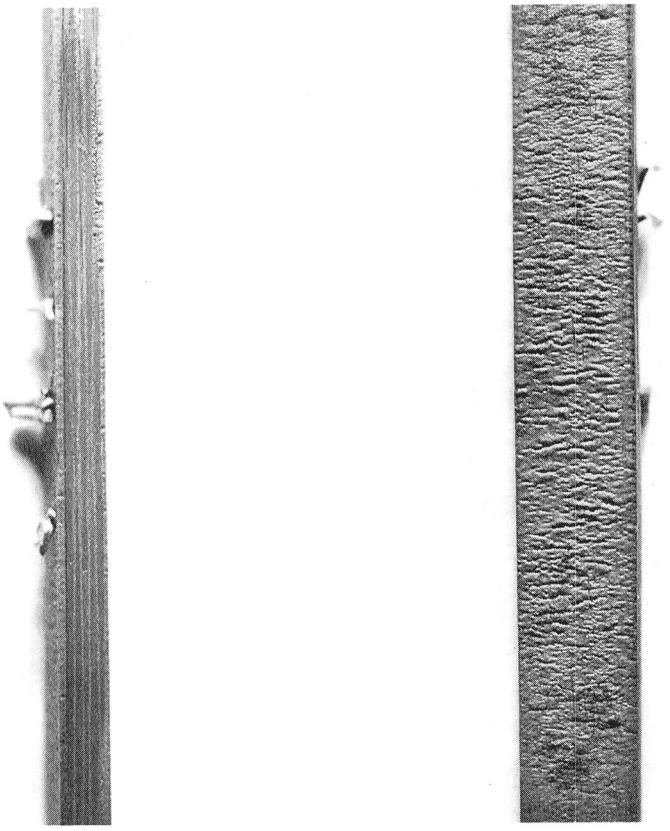


Figure 14. W/Nimonic 80A after 1000 Cycle Thermal Fatigue Test, 2.5X.



Edge View 2.5X Surface

Figure 15. W/FeNiCrAlY Specimen after 1000 Cycle, 426°-1093°C (800°-2000°F), Thermal Fatigue Test.

thermal gradient and a maximum temperature of about 930°C (1700°F). Metallographic cross sections are shown in Figures 16 through 20. The Hastelloy X system (Figure 16) shows severe fiber matrix disbonding at both locations. The IN-102 system (Figure 17) exhibits fiber-matrix disbonding in the hot zone, and both the Inconel 600 and the Nimonic 80A systems (Figures 18 and 19) exhibit fiber-matrix disbonding in the transitional temperature zone, although the latter is more severe, and some limited matrix-matrix disbonding is also in evidence. The FeNiCrAlY system (Figure 20) survived rather well in terms of resistance to internal damage.

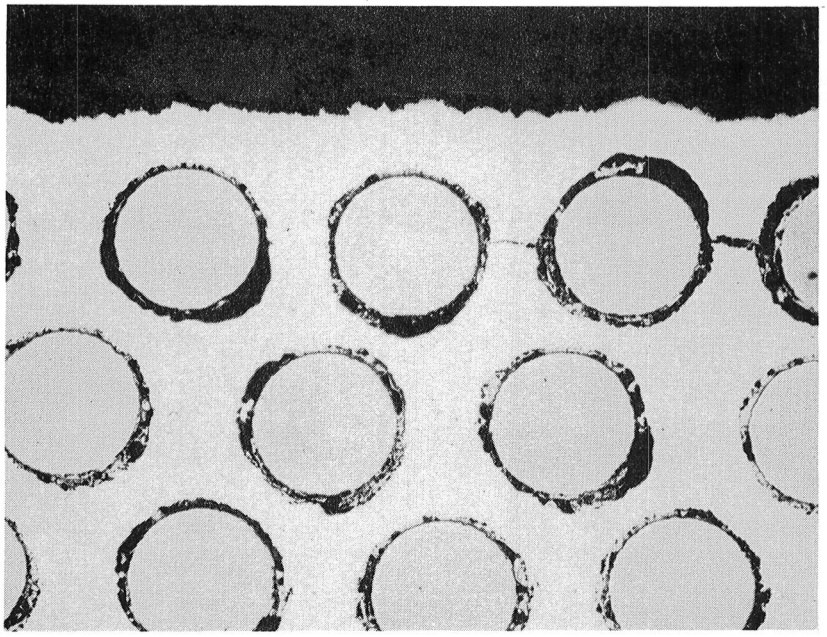
In addition to the chemical cleaning operation prior to bonding, a NiCrAlY powder bonding aid was used with the Hastelloy X, Nimonic 80A, Inconel 600, and IN-102 specimens. Non-optimum application of this powder layer may be at least partially responsible for debonding during the thermal-cycling tests. As shown in Figure 21, the powder particles were almost completely diffused away from the matrix/matrix and fiber/matrix locations after 1000 cycles to 1093°C (2000°F), but the powder particles and some associated voids were still visible in the intermediate temperature zone. These observations would suggest that, when a bonding aid is used, a post-fabrication thermal exposure at 1093°C (2000°F) might be beneficial to the composite properties. Based on the results of these evaluations, W/Inconel 600, W/FeNiCrAlY and W/FeCrAlY were selected by the NASA Program Manager for the preliminary property characterization work. Because of difficulties in obtaining the required FeNiCrAlY powder, Inconel 625 was then chosen as a replacement.

4.1.4 Fabrication of Test Specimens

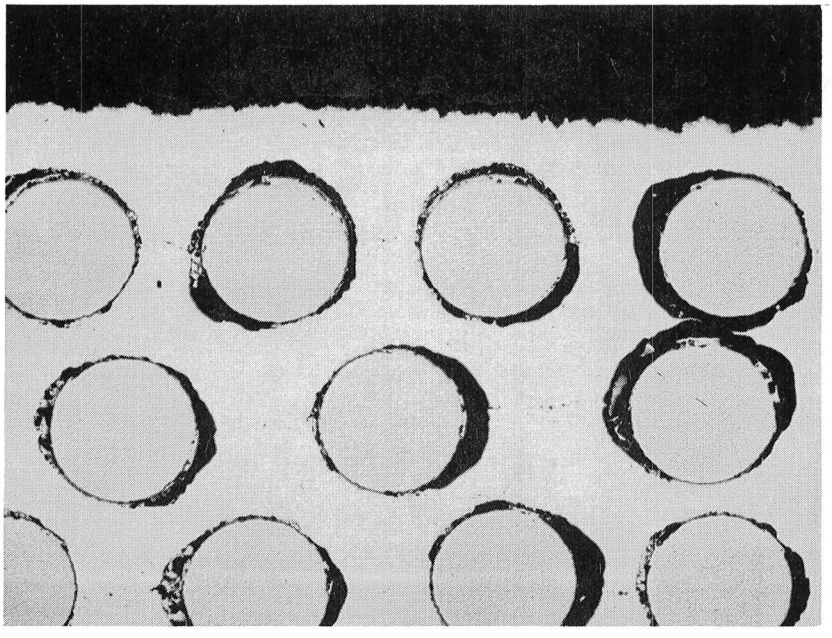
Inconel 600 and Inconel 625, the sheet alloy matrices, were obtained from Ulbrich Stainless Steel and Atek Metals Center, respectively. FeCrAlY (25-4-1) prealloyed powder was obtained from Federal Mogul Corporation. Five-ply panels were fabricated in each system with nominally 35 and 50 volume percent 218 W fiber to supply stress rupture and thermal fatigue specimens. Panels were also fabricated for miniature izod specimens (12-ply) and oxidation/compatibility (5-ply, protected fibers). The Inconel 600 and 625 matrix panels were produced by the foil-filament approach. Prior to bonding, foils were surface treated with the caustic permanganate-Oakite 33 procedure noted earlier. The FeCrAlY panels were prepared with powder cloth. Fabrication parameters were in the range 1093-1121°C (2000-2050°F), 30-60 minutes at 172-207 MPa (25-30 ksi). Specimens were subsequently machined using diamond grinding wheels.

4.1.5 Oxidation/Compatibility Tests

Composite specimens with 218 CS (cleaned and straightened) tungsten fibers in FeCrAlY, Inconel 625 and Inconel 600 matrices at 35 and 50 volume percent loading were subjected to 1093°C (2000°F) oxidation tests. Pre-weighed samples were placed in ceramic crucibles and held at temperature in static air for the prescribed period of time, after which they were reweighed. The weight gain data are summarized in Table VI. The FeCrAlY matrix composites had the greatest surface stability followed by Inconel 600, with the Inconel 625 system having the highest scaling and spalling rates.

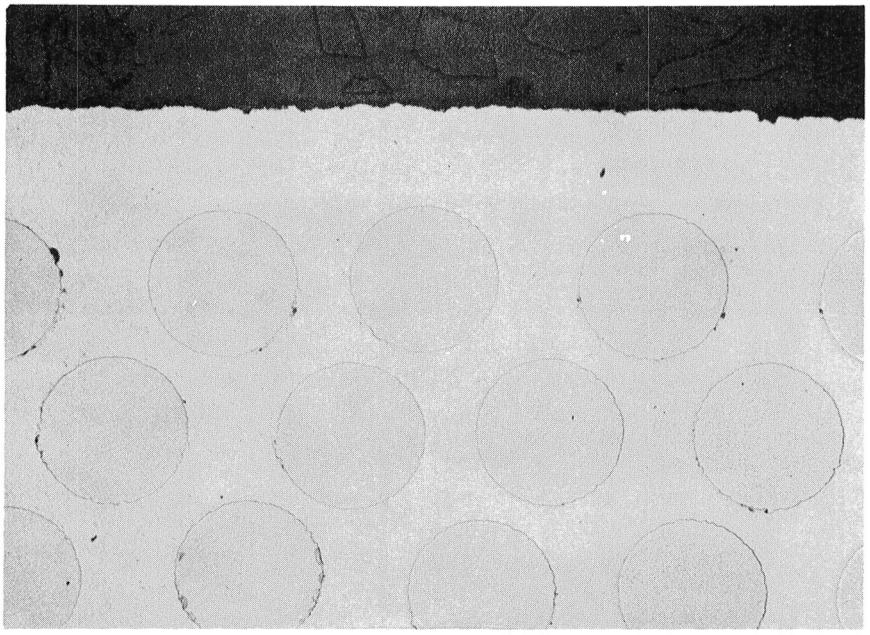


A. Intermediate Temperature Zone.

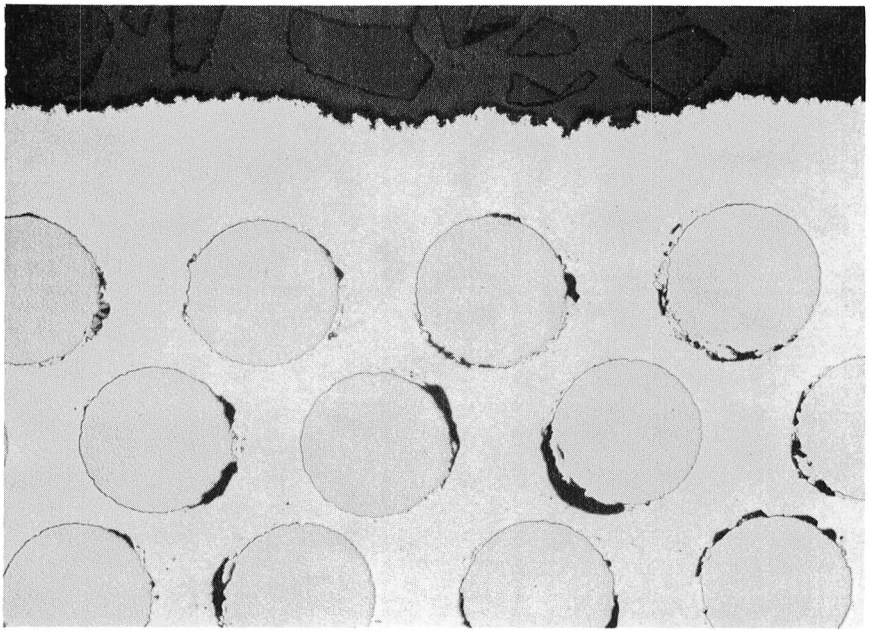


B. Hot Zone.

Figure 16. Metallographic Cross Sections Through W/Hastelloy X Thermal Fatigue Specimen. 50X

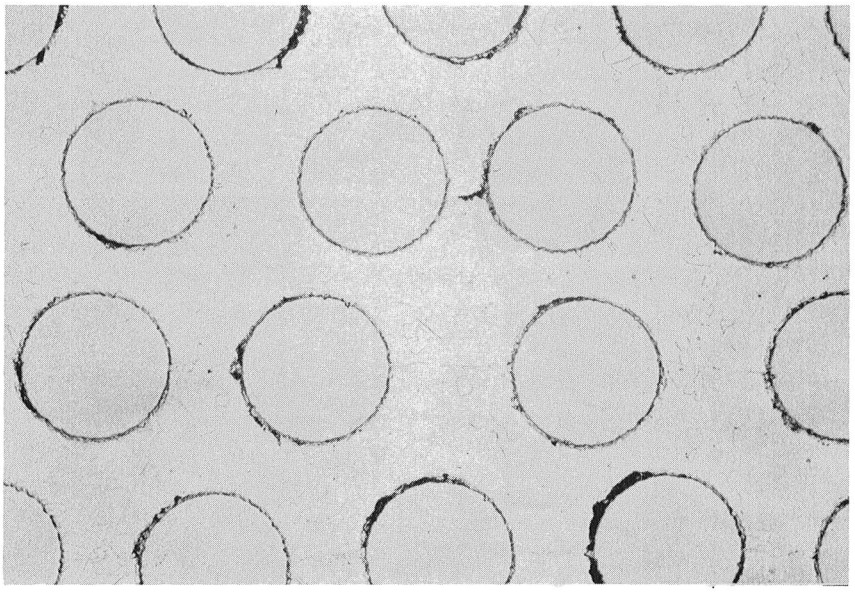


A. Intermediate Temperature Zone 50X

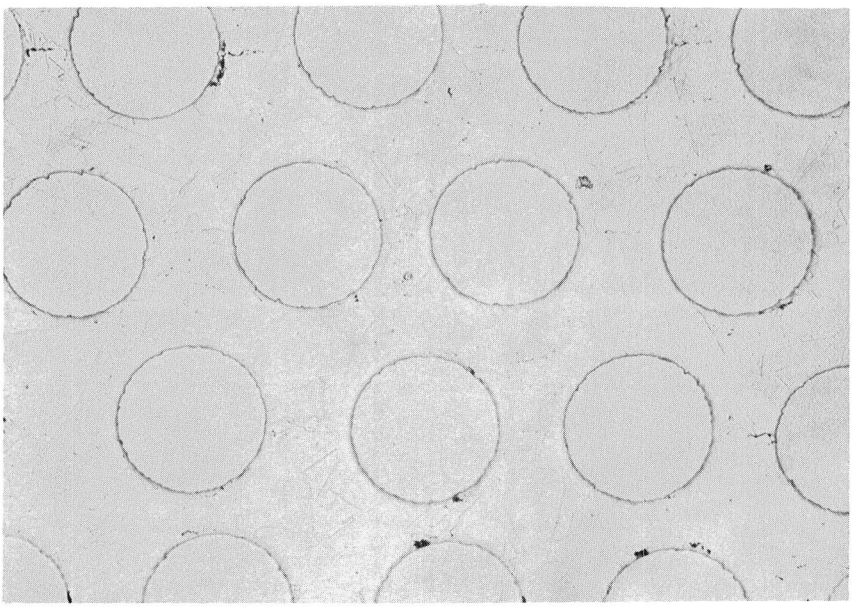


B. Hot Zone

Figure 17. Metallographic Cross Sections Through W/IN-102 Thermal Fatigue Specimens. 50X

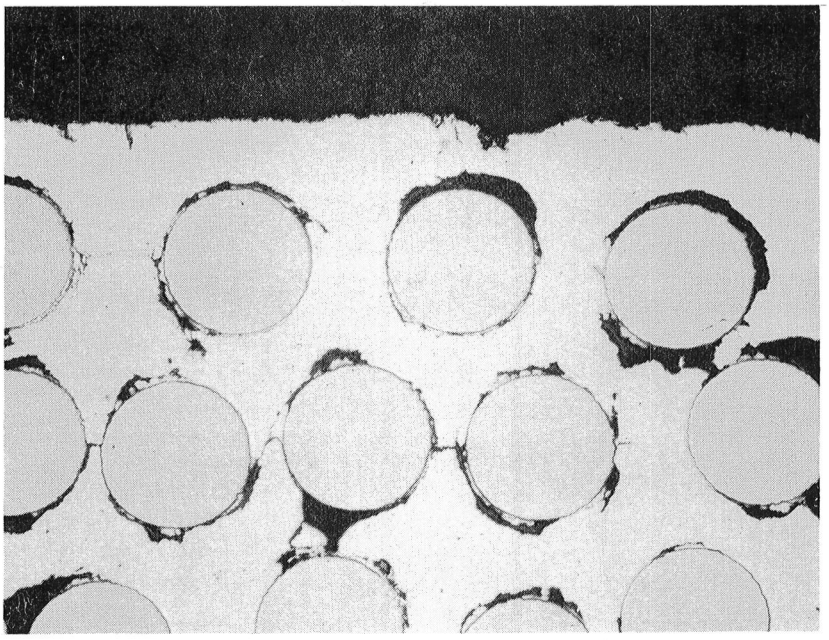


A. Intermediate Temperature Zone 50X

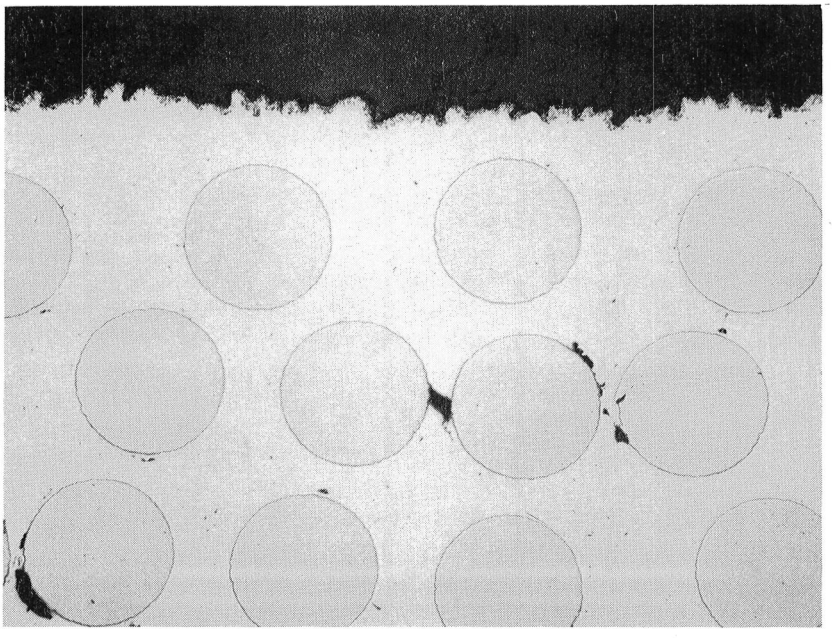


B. Hot Zone

Figure 18. Metallographic Cross Section Through W/Inconel-600 Thermal Fatigue Specimen. 50X

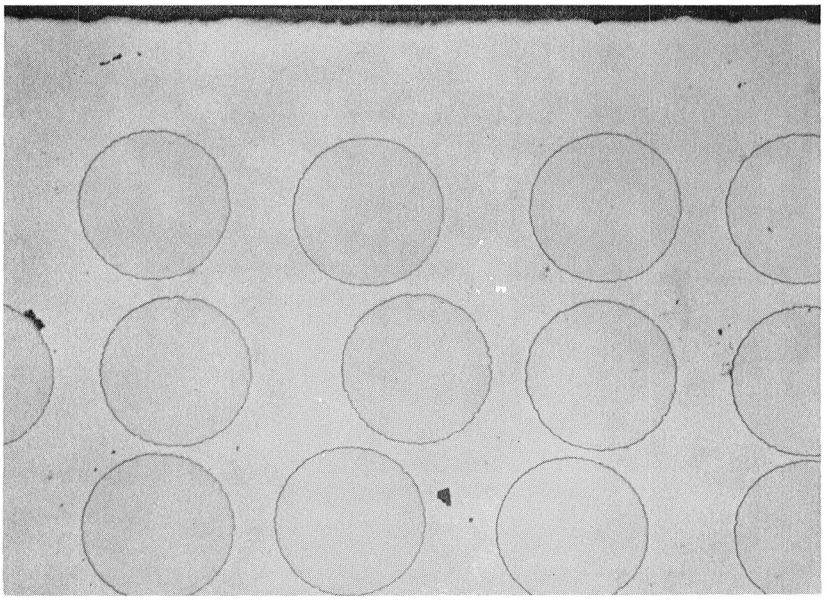


A. Intermediate Temperature Zone.

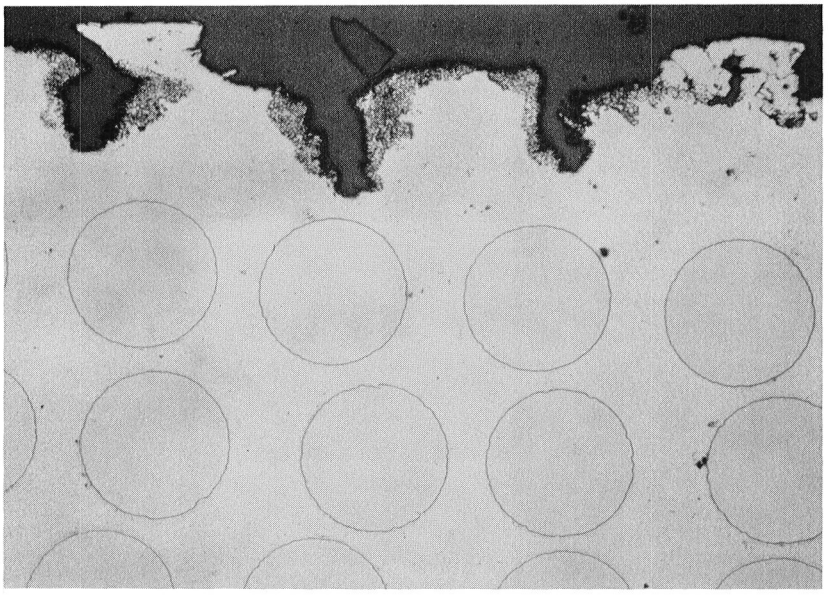


B. Hot Zone.

Figure 19. Metallographic Cross Sections Through W/Nimonic 80A Thermal Fatigue Specimen. 50X

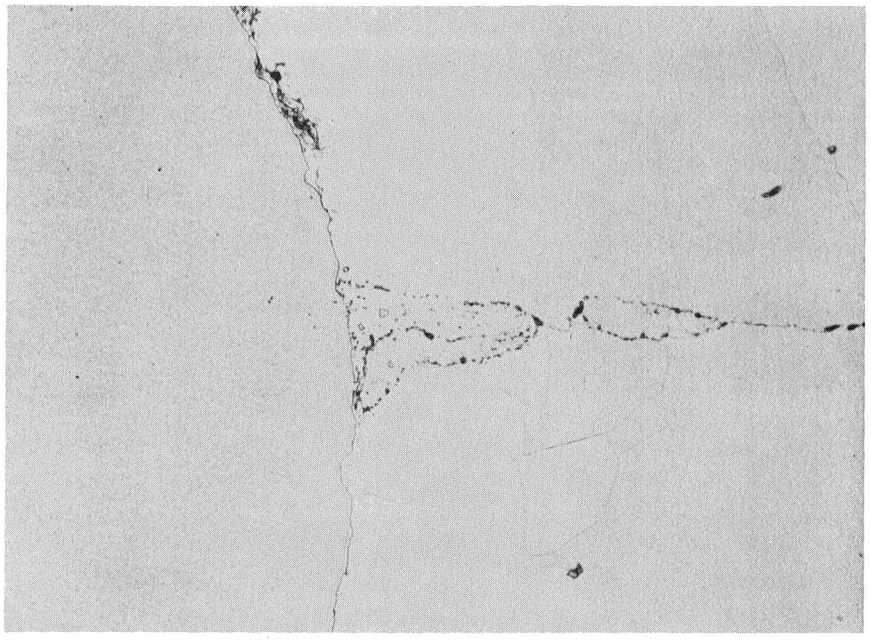


A. As Fabricated 50X

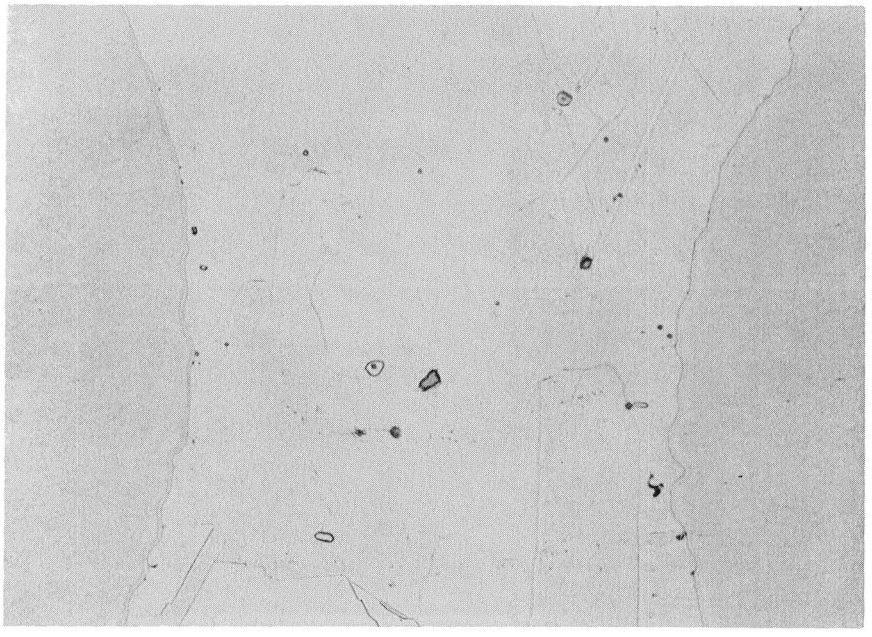


B. Hot Zone 50X

Figure 20. Cross Section Through W/FeNiCrAlY Thermal Fatigue Specimen. 50X



A. Intermediate Temperature Zone. 500X



B. Hot Zone

Figure 21. Effect of 1000 Cycles, 426°-1093°C (800°-2000°F) on Fiber/Matrix and Matrix/Matrix Interfaces in W/Inconel-600 Composite.

TABLE VI

1093°C (2000°F) Oxidation Rate Data on W/FeCrAlY, W/Inconel 600 and W/Inconel 625

Specimen (Vol. Percent)	Matrix	Total Weight Change, mg/cm^2					
		1 Hour	5 Hours	10 Hours	20 Hours	50 Hours	100 Hours
17 B1 (35 v/o)	FeCrAlY	0.19	0.33	0.44	0.54	0.73	0.90
17 B2 (")	"	0.21	0.34	0.44	0.56	0.77	0.92
18 B1 (50 v/o)	"	-0.27	0.32	0.42	0.53	0.74	0.74
18 B3 (")	"	0.42	0.55	0.56	0.68	0.87	0.98
19 B1 (50 v/o)	Inconel 600	0.62	0.89	0.96	0.92	0.83	1.17
19 B2 (")	"	0.59	0.92	1.07	1.22	1.44	1.88
20 B1 (35 v/o)	"	0.74	0.92	1.05	1.11	1.12	1.41
32 B1 (35 v/o)	Inconel 625	-7.19	1.00	0.67	0.74	-1.02	-1.85
32 B2 (")	"	1.02	1.21	1.14	1.06	-	-
32 B4 (")	"	1.20	1.44	1.33	1.42	0.47	-1.00
27 Bw (50 v/o)	"	1.28	0.40	1.87	1.96	2.12	-0.36

4.1.6 Impact Tests

Miniature (0.5 x 0.5 x 3.75 cm), Izod (notched) specimens (Figure 22) of W/FeCrAlY, W/Inconel 600 and W/Inconel 625 with 35 and 50 volume percent fiber were prepared for impact tests. Thermocouples were welded to 24 of the total 32 specimens within 0.1-0.2 cm of the notch for duplicate elevated temperature tests at 149, 260 and 1371°C (300, 500 and 700°F). Temperature calibrations were performed using an X-Y recorder and propane torches to heat a dummy specimen. Following procedures developed on a previous program [5], the specimens were heated to about 28°C (50°F) higher than the required test temperature and impacted during the cool-down cycle, impact occurring 3-5 seconds after removal of the torch. This procedure ensured that the measured temperatures were representative of the bulk specimen temperatures, rather than transitory surface temperatures.

Impact data are summarized in Table VII. Specimens which did not fail at the load range used (pendulum stopped completely) are indicated; i.e., specimen 6-35-3, > 2.8J (>25 in-lb), and 6-50-5, > 22.6J (>200 in-lb). The equipment used has four load ranges, the maximum of which is 22.6J (200 in-lb). The load range for each specimen was preselected so as to provide the greatest testing accuracy. However, as is shown in Table VII, several specimens exceeded the pre-selected limit. Also, a number of specimens exceeded the total capacity of the equipment.

At room temperature, i.e., below the DBTT of the tungsten wire, matrix properties influence the impact strength and, as is evident from these results, the stronger and tougher Inconel alloys result in impact properties superior to those of the FeCrAlY composites. Also, below the transition temperature, toughness is inversely proportional to fiber content. Above the transition temperature, fiber necking and matrix disbonding provide the greatest contribution to fracture energy so that toughness increases with fiber content.

The Inconel 625 matrix composite had the highest toughness over the entire range of test temperatures and had impact strength greater than 22.6J (200 in-lb) at test temperatures of 260°C (500°F) and above for both 35 v/o and 50 v/o fiber reinforcements. Tests were not performed on this system at temperatures greater than 260°C because of the load limitation.

Also shown in Table VII is a Normalized Impact Energy value. This was obtained by linear scaling of specimen size, since subsize specimens were used. On such a normalized basis, the impact strength of all of the systems above 371°C is greater than 92.3J (68 ft-lb).

4.1.7 Stress Rupture Tests

Stress rupture tests were performed on the FeCrAlY, Inconel 600 and Inconel 625 composites to define the 1093°C (2000°F), 100-hour stress rupture strengths of the various systems. The initial tests were carried out in an inert gas environmental chamber, mounted in a standard stress rupture frame, using water-cooled pull rods to allow loading through a Viton O-ring seal assembly. Temperature was measured by a Chromel/Alumel thermocouple which was tack welded the center of the specimen. The estimated grip temperatures were within 55-110°C (100-200°F) degrees of the specimen gage section. A total of five tests were performed: two

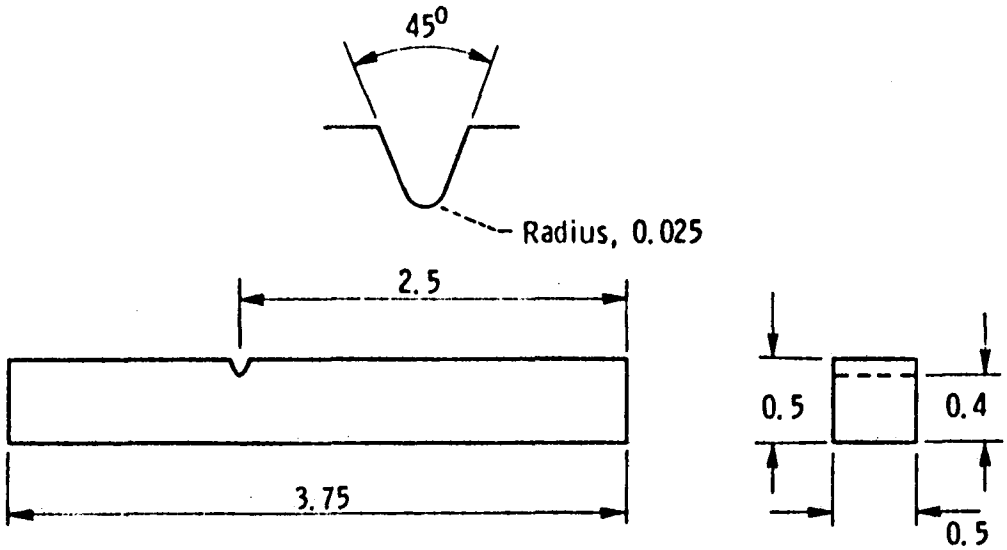


Figure 22. Miniature Izod Impact Specimen. Unnotched Specimens Have the Same Dimensions as Notched Specimens but No Notch. All Dimensions are in Centimeters Unless Otherwise Noted.

TABLE VII

Notched Miniature Izod Impact Strengths of W-Superalloy Composites

Spec. I.D.	System	Test Temperature °C	Test Temperature (°F)	Impact Strength Joules	Impact Strength in-lb	Normalized Impact Energy in-lb/in^2 (A)
6-35-3	35 v/o W/Inconel 600	21	(70)	>2.82	>25.0	>806 (B)
6-35-6	"	"	(")	4.26	37.7	1216
6-35-1	"	149	(300)	3.45	30.5	984
6-35-2	"	"	(")	3.32	29.4	948
6-35-8	"	260	(500)	3.87	34.3	1106
6-35-8	"	"	(")	3.77	33.4	1107
6-35-4	"	371	(700)	8.13	72.0	2322
6-35-5	"	"	(")	10.05	89.0	2871
6-50-1	50 v/o W/Inconel 600	21	(70)	1.24	11.0	355
6-50-7	"	"	(")	1.36	12.0	387
6-50-6	"	149	(300)	1.40	12.4	400
6-50-8	"	"	(")	1.29	11.4	368
6-50-2	"	260	(500)	2.64	12.3	755
6-50-4	"	"	(")	2.03	18.0	581
6-50-2	"	371	(700)	10.11	89.5	2887
6-50-5	"	"	(")	>22.60	>200.0	>6452 (C)
F-35-1	35 v/o W/FeCrAlY	21	(70)	0.50	4.4	142
F-35-3	"	"	(")	0.79	7.0	226
F-35-7	"	149	(300)	2.90	25.6	826
F-35-8	"	"	(")	3.62	32.0	1032
F-35-5	"	260	(500)	7.97	70.5	2274
F-35-6	"	"	(")	7.74	68.5	2209
F-35-2	"	371	(700)	6.84	60.5	1952
F-35-4	"	"	(")	8.14	72.0	2322
F-50-1	50 v/o W/FeCrAlY	21	(70)	0.86	7.6	245
F-50-3	"	"	(")	0.75	6.6	213
F-50-7	"	149	(300)	2.56	22.7	732
F-50-8	"	"	(")	2.98	26.4	852
F-50-5	"	260	(500)	>11.30	>100.0	>3226 (B)
F-50-6	"	"	(")	10.11	89.5	2887
F-50-2	"	371	(700)	18.53	164.0	5290
F-50-4	"	"	(")	>22.60	>200.0	>6452 (C)
I-35-1	35 v/o W/Inconel 625	21	(70)	10.85	96.0	3097
I-35-2	"	"	(")	>5.65	>50.0	>1613 (B)
I-35-3	"	149	(300)	15.14	134.0	4322
I-35-4	"	"	(")	>11.30	>100.0	>3225 (C)
I-35-5	"	260	(500)	>22.60	>200.0	>6452 (C)
I-35-6	"	"	(")		Not Tested	(D)
I-35-7	"	371	(700)		" "	(D)
I-35-8	"	"	(")		" "	(D)

(cont'd.)

TABLE VII (continued)

Notched Miniature Izod Impact Strengths of W-Superalloy Composites

Spec. I.D.	System	Test Temperature °C	Test Temperature (°F)	Impact Strength Joules	Impact Strength in-lb	Normalized Impact Energy in-lb/in^2 (A)
I-50-1	50 v/o W/Inconel 625	21	(70)	2.24	19.8	639
I-50-2	"	"	(")	3.03	26.8	864
I-50-3	"	149	(300)	7.17	63.5	2048
I-50-4	"	"	(")	5.65	50.0	1613
I-50-5	"	260	(500)	20.11	178.0	5742
I-50-6	"	"	(")	>22.60	>200.0	>6452 (C)
I-50-7	"	371	(700)	Not Tested		(D)
I-50-8	"	"	(")	"	"	(D)

A) Based on an average specimen area of 0.200 cm^2 (0.031 in^2)
1 J = 8.85 in-lb = 0.737 ft-lb.

B) Exceeded pre-selected load limit.

C) Exceeded equipment capability.

D) Value will be well in excess of equipment capability.

35 v/o W/FeCrAlY; two 50 v/o W/FeCrAlY and one 35 v/o W/Inconel 600, at applied stress levels of 275-345 MPa (40-50 ksi). All specimens failed during loading or within five minutes of applying the load, by pullout at the grips. During the course of these initial tests, unsuccessful attempts were made to reduce grip temperature by modifying the pull-rods to bring the cooling water closer to the specimen grips.

A successful test procedure was developed in the previous program[5] to conduct stress rupture tests at 1093°C in air, but this test procedure could not be used with these specimens since they had edge-exposed fibers. It was therefore decided to conduct the remaining tests in the creep-testing facility, which utilizes a vacuum/inert atmosphere furnace with a small tantalum element hot zone.

Stress rupture tests of the three composite systems were conducted in a Brew vacuum/inert atmosphere furnace using a specially designed tantalum heating element and heat shield assembly. The specimen configuration and grip assembly is shown in Figure 23. Temperature was monitored continuously by a Pt-Pt/Rh thermocouple attached to the center of the specimen. The temperature variation over the center 2 cm (3/4 inch) of the gage section has been shown in previous tests to be within 3°K (±6°F).

The data obtained are summarized in Table VIII and plotted in Figure 24. All three materials, when compared on the basis of fiber stress, show comparable behavior although the FeCrAlY has a definite superiority.

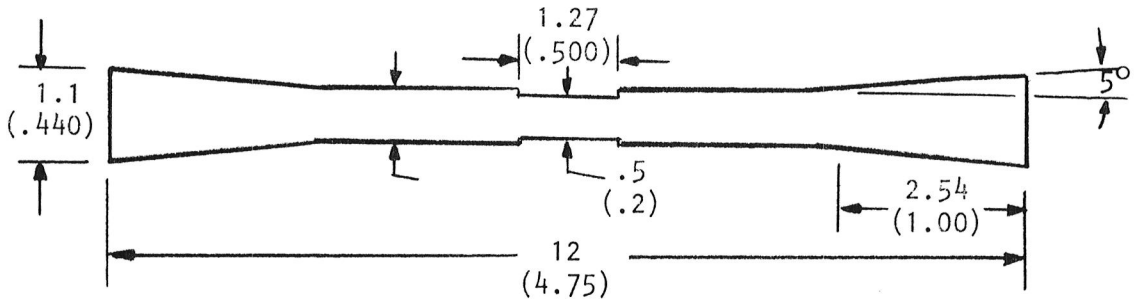
A problem was encountered due to an incorrect thermocouple replacement for two tests. A Pt-10% Rh thermocouple was inadvertently installed instead of Pt-13% Rh so that the indicated 1093°C (2000°F) temperature was actually 1182°C (2160°F) resulting in premature failures. A Larson-Miller extrapolation of the data for specimens 28-B-5 and 25-B-4 would give a predicted life of about 100 hours at 1093°C (2000°F). This is in reasonable agreement with the curves shown in Figure 24.

In addition to the W/FeCrAlY, composites of W-Hf-C/FeCrAlY were also fabricated and tested.

Problems were experienced in the testing of these W-Hf-C/FeCrAlY specimens, Series 35 B-2, which failed prematurely at the grip/fillet transition or by pullout. These tests were then repeated using specimens with a reduced gage section (from a nominal .64 cm (0.25 inch) width to 0.5 cm(0.20 inch). These results are included in the table as the 36B Series and are plotted in Figure 24. The advantage of the stronger fiber is clearly evident.

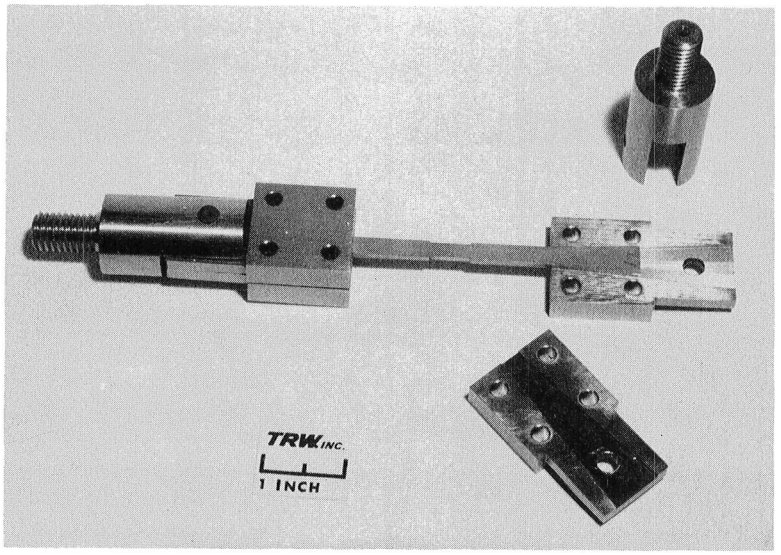
4.1.8 Stress Rupture Tests of Thermally Cycled Material

Stress rupture tests were performed at 1093°C (2000°F) on W/FeCrAlY specimens which had previously been subjected to 1000 cycles between 426 and 1093°C (800 and 2000°F). The results are shown in Table IX.



A. Specimen Design.

Dimensions in Centimeters (in.)



B. Grip Assembly and Machined Specimen.

Figure 23. Stress Rupture Specimen and Grips.

TABLE VIII

1093°C (2000°F) Stress Rupture Tests

Specimen	System	Composite Stress MPa	Composite Stress Ksi	Fiber Stress[1] MPa	Fiber Stress[1] Ksi	Time to Rupture Hours	Test Temp. °C	Test Temp. (°F)	
28 B-1	35 v/o W/Inconel 625	160	23	455	66	20.65	1093	(2000)	
28 B-2	"	160	23	455	66	20.85	"	"	
28 B-3	"	190	28	550	80	4.6	"	"	
28 B-4	"	190	28	550	80	0.25	"	"	
28 B-5	"	134	19.5	370	54	3.99	1182	(2160)	[2]
28 B-6	"	134	19.5	365	53	6.69	1093	(2000)	
25 B-1	50 v/o W/Inconel 625	280	40	530	77	6.59	"	"	
25 B-2	"	280	40	530	77	3.92	"	"	
25 B-3	"	210	30	390	57	29.39	"	"	
25 B-4	"	170	25	340	49	6.14	1182	(2160)	[2]
25 B-5	"	210	30	410	59	17.13	1093	(2000)	
25 B-6	"	170	25	340	49	101.20	"	"	
25 B-7	"	170	25	340	49	93.04	"	"	
16 B-1	50 v/o W/Inconel 600	270	39	515	74.6	3.49	1093	(2000)	
16 B-2	"	275	39.9	520	76	4.96	"	"	
16 B-3	"	212	30.8	405	58.8	25.79	"	"	
16 B-4	"	171	24.8	326	47.3	50.9	"	"	
16 B-5	"	168	24.4	321	46.6	13.62	"	"	
15 B-1	35 v/o W/Inconel 600	191	27.7	571	82.8	7.06	1093	(2000)	
15 B-2	"	174	25.3	521	75.6	6.91	"	"	
15 B-3	"	210	30.4	625	90.7	2.39	"	"	
15 B-4	"	132	19.1	392	56.9	27.04	"	"	
15 B-5	"	129	18.7	385	55.9	19.72	"	"	

(1) Based on a fiber count in the fractured cross section.

(2) Incorrect thermocouple used.

TABLE VIII (continued)

1093°C (2000°F) Stress Rupture Tests

Specimen	System	Composite Stress MPa	Composite Stress Ksi	Fiber Stress[1] MPa	Fiber Stress[1] Ksi	Time to Rupture, Hrs	Test Temp. °C	Test Temp. (°F)	Comments
14 B-1	50 v/o W/FeCrAlY	280	40	724	105	0.19	1093	(2000)	Specimens had lower than anticipated reinforcement levels.
14 B-2	"	280	40	724	105	0.14	"	"	
14 B-3	"	240	35	655	95	1.62	"	"	
14 B-4	"	240	35	655	95	1.1	"	"	
14 B-5	"	210	30	545	79	5.55			
35 B3-1	"	210	30	420	61	9.92	1182	(2160)	
35 B3-5	"	170	24	360	52	8.82	"	"	
35 B3-6	"	120	18	270	39	35.14	"	"	
35 B3-2	"	120	18	280	41	32.20	"	"	
31 B-1	35 v/o W/FeCrAlY	210	30	770	112	0.02	1093	(2000)	
31 B-2	"	170	25	548	79.5	22.50	"	"	
31 B-3	"	134	19.5	450	65	27.77	"	"	
31 B-4	"	110	16.0	414	60	49.15	"	"	
35 B1-1	35 v/o W-Hf-C/Fe	450	65	1180	171	-	"	"	Broke on Loading.
35 B2-2	" CrAlY	510	74	1125	163	-	"	"	Grip Pullout.
35 B2-3	"	480	69	1060	154	-	"	"	Broke on Loading.
35 B2-1	"	397	57.6	1060	154	1.79	"	"	Reduced Gage Section-Good Break.
36 B2-1	"	364	52.8	1093	158.5	2.30	"	"	
36 B2-2	"	287	41.7	864	125.3	9.09			
36 B1-1	"	294	42.7	884	128.3	31.51	"	"	
36 B1-2	"	209	30.3	627	90.9	213.87	"	"	

(1) Based on fiber count in the fractured cross section.

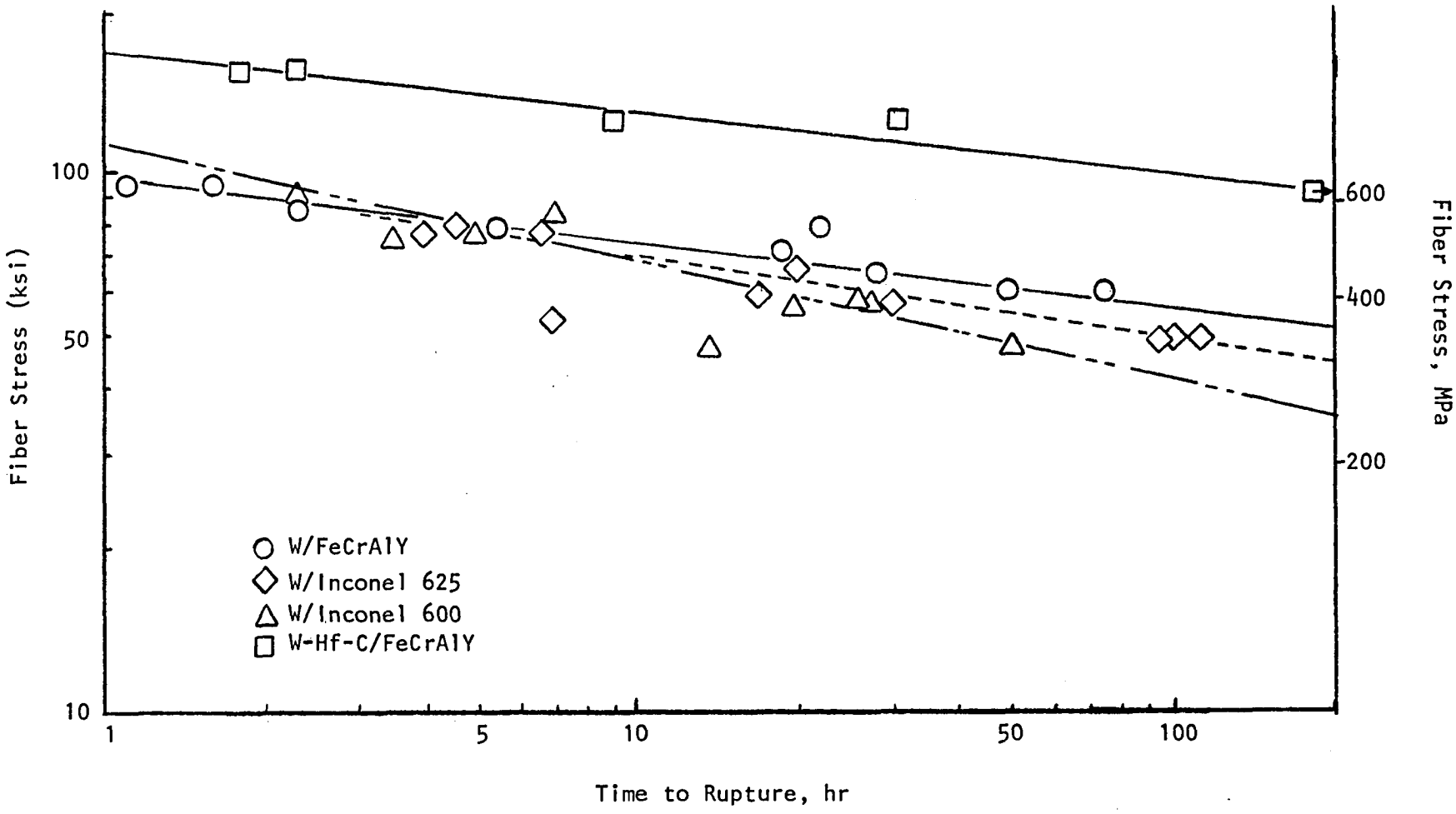


Figure 24. Stress-Rupture Comparison at 1093°C (2000°F).

TABLE IX

Stress Rupture of Thermally Cycled 35 v/o W/FeCrAlY

Specimen	Composite Stress, MPa	Ksi	Fiber Stress, MPa	Ksi	Time to Rupture, hours
31 B-1	221.6	32.14	586	85.0	2.30
35 B-3-3	188	27.3	498	72.2	18.50
35 B-3-4	160	23.2	416	60.4	73.92

These data are plotted in Figure 25 using a simple linear regression program (semi-log transform). For comparison, several data points for virgin panels (14B and 31B Series) are also shown. This clearly shows no stress-rupture degradation due to thermal cycling.

4.1.9 Thermal Fatigue Tests

Thermal fatigue tests, in which the variables were fiber orientation and load, were performed for W/FeCrAlY composite specimens as described below.

4.1.9.1 Effect of Wire Distribution

Three wire distribution patterns were investigated with the W/FeCrAlY system at nominally 45-50 v/o reinforcement. These combinations were selected as being most applicable to a possible blade reinforcement pattern.

1. $\pm 15^{o}$, fiber orientation, balanced ply configuration.
2. $\pm 15^{o}$, 0^{o}, balanced ply configuration, uniform 0.025 cm (0.010 inch) matrix cladding.
3. $\pm 15^{o}$, 0^{o}, balanced ply configuration, unbalanced cladding; i.e., the clad thickness on one side was 0.025 cm (0.010 inch) and 0.064 cm (0.025 inch) on the other.

Specimens were subjected to 100 cycles between room temperature and $1093^{o}C$ ($2000^{o}F$). Specimen dimensional changes are tabulated below.

TABLE X

Thermal Fatigue Resistance as Affected by Fiber Orientation in W/FeCrAlY

Specimen	Fiber Distribution	Appearance	Dimensional Change % Gage Length	Width	Thickness
33G-15-1	$\pm 15^{o}$	Excellent	+0.8	-2.1	+1.3
33B-15-2	$\pm 15^{o}$, 0^{o}	Delamination	0.17	-0.5	+4.7
33B-15-3	$\pm 15^{o}$, 0^{o} (Unbalanced Cladding)	Excellent	-0.34	-0.75	0.46

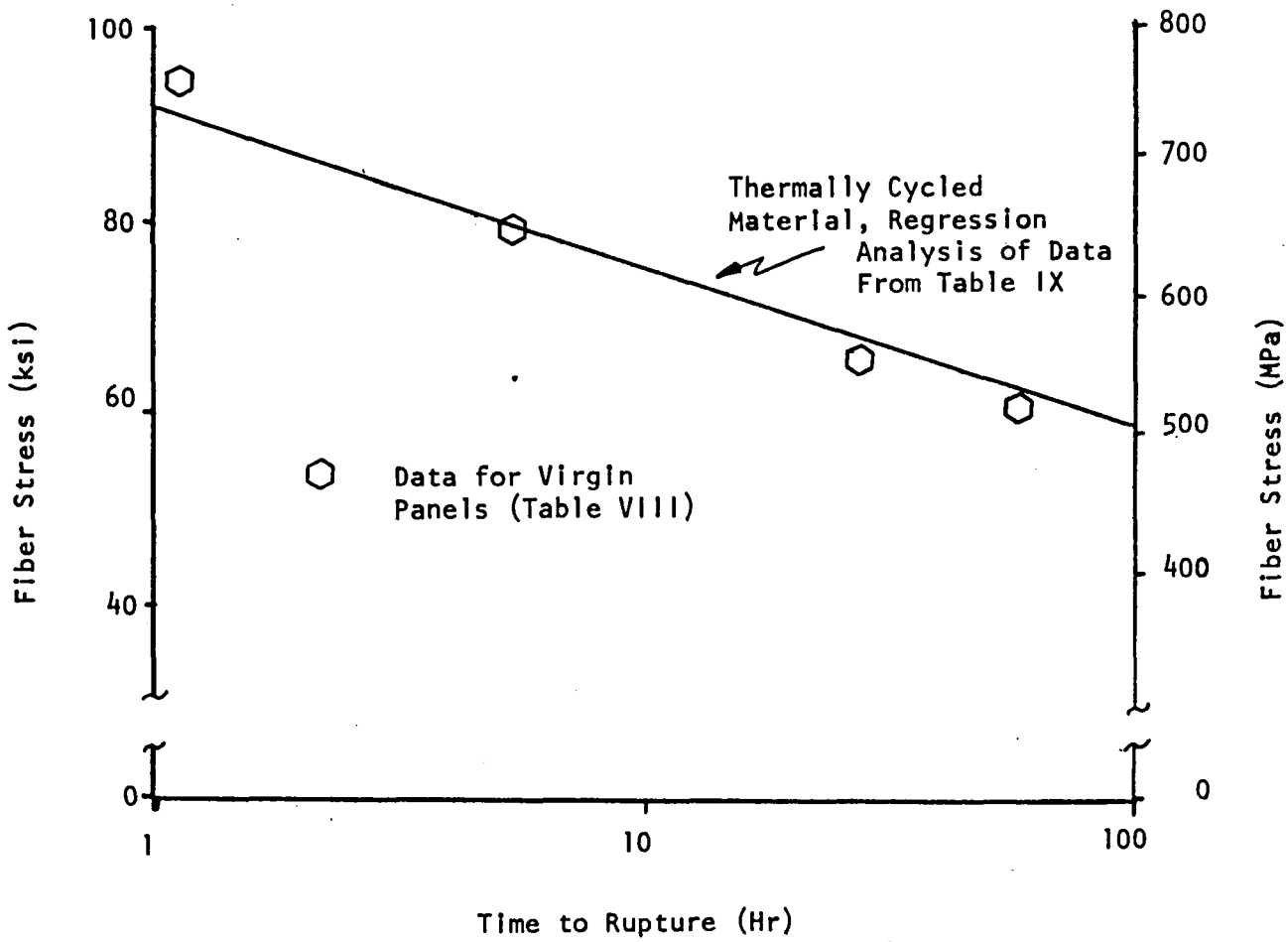


Figure 25. Comparison of Stress-Rupture Behavior of Virgin and Thermally Cycled W/FeCrAlY.

Only the ±15°, 0° specimen showed any sign of damage, and, as noted above, this did delaminate. Based upon the results with the other two materials, quality of this specimen is suspect. It should be noted that, despite asymmetric residual stresses in the specimen with the unbalanced clad, no distortion occurred. This indicates that the fibers are capable of accepting such stress imbalance at a 45 to 50 v/o level of reinforcement.

4.1.9.2 External Load Effects

Specimens of unidirectionally reinforced 50 v/o W/FeCrAlY were subjected to a static load during cycling from room temperature to 1093° (2000°F) with the object of defining stress for failure in 100 cycles. The data are summarized in Table XI. Fiber stress is calculated based on number of fibers in the fracture cross section and assuming no matrix contribution. Because of the dynamic test conditions, stress distributions are complex; it is reasonable to assume that the matrix is almost fully relaxed at the cycle peak temperature. The heating and initial cooling cycle are linear and calculations indicate that, in each cycle, the specimen is in the 1037-1093°C (1900-2000°F) range for about 5 seconds. A hundred cycles therefore correspond to 0.14 hours exposure above 1037°C (1900°F). Analysis of the fracture surface is complicated by the fact that localized melting occurs if failure takes place during the heating cycle. The possible effects of localized debonding and void formation on electrical resistivity and thermal conductivity need to be considered in analyzing data from this type of test.

Compared to conventional stress-rupture data (e.g., Figure 24), rupture lives of these specimens subjected to static load but cyclic temperature are significantly shorter (1 hour or less versus 10's of hours). This is an area which deserves further study. Two points should be noted, however:

1. Thermal cycling under a static load is an unlikely situation in an actual component, and

2. The test cycle is quite short and the response time of the controls on the apparatus is such that a temperature and/or stress overshoot cannot be ruled out.

4.1.9.3 Advanced Thermal Fatigue Characterization Studies

Specimens of Inconel 600 and Inconel 625 with nominally 35 and 50 v/o fiber reinforcement were subjected to 1000 cycle 426-1093°C (800-2000°F) tests. Dimensinal change data and comments on visual appearance after test are reported in Table XII.

Specimen 34B-2 suffered a possible temperature overshoot when the thermocouple became detached after 114 cycles. The test, however, stopped at the low temperature end of the cycle and was continued after a thermocouple was replaced. Both matrix systems with 50 v/o fibers cracked and delaminated.

Three machined 45-50 v/o W/FeCrAlY stress rupture specimens were subjected to 1000 cycles, 426-1093°C (800-2000°F), as above. Dimensional change in the gage section ranged from 0.1-0.7%. The appearance of all three specimens was good with no distortion, delamination or cracking.

TABLE XI

1093°C (2000°F) Thermal Fatigue Life
as a Function of Applied Load, W/FeCrAlY

Specimen	Applied Stress MPa	Ksi	Fiber Stress MPa	Ksi	Cycles to Failure
31B-2	280	40	650	95	7
31B-2	210	30	510	74	26
31B-4	170	25	455	66	140

TABLE XII

1000 Cycle Thermal Fatigue Resistance of W/Inconel 625 and W/Inconel 600

Specimen	Composite Composition	Dimensional Change, % Gear Length	Gear Width	Gear Thickness	Appearance
34B-1	35 v/o W/Inconel 625	-0.5	1.1	1.07	No distortion or cracking.
34B-2	50 v/o W/Inconel 625	-	12.2	12.4	Delaminated, badly cracked.
35B-1	35 v/o W/Inconel 600	0.23	3.1	3.8	No distortion; no significant cracking.
36B-1	50 v/o W/Inconel 600	-5.6	8.1	2.8	Growth in width dimension, microcracking.

4.2 Task II

4.2.1 Selection of Fiber/Matrix Combination for Task II

Up to this point, two fibers, 218 tungsten and W-Hf-C, and seven matrix alloys -- Inconel 600, Inconel 625, IN-102, Hastelloy-X, Nimonic 80A, FeNiCrAlY and FeCrAlY -- had been examined. Based upon all of the data to date, the W-Hf-C fiber clearly results in a superior composite. Also, in terms of combinations of properties, the Fe-25Cr-4Al-1Y matrix alloy is preferred. This was, therefore, the system chosen for further study in Task II.

4.2.2 Characterization of W-Hf-C/FeCrAlY

The W-Hf-C fiber, unlike the commercial grade 218W fiber, which is available in multi-thousand meter lengths, was delivered on spools of short lengths, 175 meters maximum, which necessitated making many splices during panel fabrication. The first panels manufactured contained 30 v/o fibers and were tested in tension at 982 and 1093°C (1800 and 2000°F), Table XIII. The specimens were tested in air and were held in water-cooled grips. The cross-head speed was 0.5 mm/min. The load-elongation curves were linear to failure and, hence, no yield strength could be determined.

Subsequently, additional panels containing 40-55% W-Hf-C were manufactured and tested. Table XIV lists the results of tensile tests at 982 and 1093°C (1800 and 2000°F).

The second 982°C (1800°F) specimen, T55-2, failed in a tensile-shear mode (see Figure 26). This may be due to the introduction of a bending moment during the test. This photo also shows the oxidation loss of a layer of exposed fiber. Actual specimen cross-sections are shown in Figure 27.

Two specimens were tested to determine the stress for 1% creep in 100 hours. The first specimen, C55-1, Table XV, was destroyed when it short circuited the furnace heating element, which caused the gage-section matrix to vaporize.

TABLE XV

Creep Tests - W-Hf-C/FeCrAlY

Specimen No.	Fiber Content v/o	Temperature °C	°F	Stress Composite MPa	(ksi)	Fiber MPa	(ksi)	Life hr	Elongation %
C55-1	55(1)	982	(1800)	113	(16.3)	210	(30.4)	>5(2)	
C55-2	39(3)	982	(1800)	113	(16.3)	290	(42.0)	30	
				380	(55.1)	972	(14019)	>3	>1.1(?)(4)

(1) Nominal
(2) Specimen destroyed; see text.
(3) By fiber count, excluding surface fibers.
(4) Specimen pulled out of grip; strain determined from photo.

TABLE XIII

Elevated Temperature Tensile Strength of 30 v/o W-Hf-C/FeCrAlY

Specimen	Temperature °C	°F	Composite MPa	Ksi	Fiber(1) MPa	Ksi	Elongation %	RA %
6-1	982	(1800)	430	62.4	1430	208	0.6	5.8
6-2	982	(1800	495	71.8	1650	239	0.5	7.5
6-3	1093	(2000)	386	56.0	1290	187	0.5	3.9
6-4	1093	(2000)	378	54.9	1260	183	0.3	1.4

(1) Fiber stress based on fiber content.

TABLE XIV

Elevated Temperature Tensile Test[1]

W-Hf-C/FeCrAlY

Specimen	Fiber Content v/o[2]	Temp. °C	Temp. (°F)	UTS Composite Fiber MPa	UTS Composite Fiber (ksi)	UTS Fiber MPa	UTS Fiber (ksi)	0.2% Y.S. Composite Fiber MPa	0.2% Y.S. Composite Fiber (ksi)	0.2% Y.S. Fiber MPa	0.2% Y.S. Fiber (ksi)	Elongation %	Modulus GPa	Modulus ($lb/in^2 \times 10^6$)
T55-1	55	982	(1800)	684	(99)	1240	(179)	615	(89)	1120	(162)	~3.2	152	(22.1)
T55-2	<38	982	(1800)	724	(105)	<1890[3]	(<274)	665	(96)	<1740	(252)	~2.4	151	(21.9)
T55-3	42	1093	(2000)	>548[4]	(>80)	>1300	(>188)	428	(62)	1020	(148)	>1.6	265	(38.5)[5]
T55-4	44.6	1093	(2000)	607[6]	(88)	1370	(199)	540	(78)	1210	(176)	3.0	85	(12.3)

(1) All tests conducted in still air.

(2) As determined by fiber count at specimen cross section in gage length and excluding all surface fibers.

(3) Some fibers may have been lost when specimen failed in shear.

(4) Specimen pulled out of grip.

(5) Questionable value due to slipping specimen.

(6) .38 cm (0.15 inch) wide gage; previous three specimens had 0.5 cm (0.2 inch) wide gage.

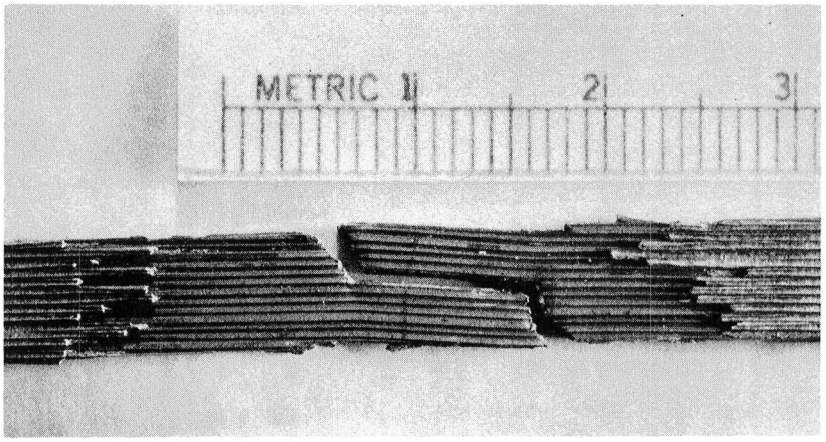
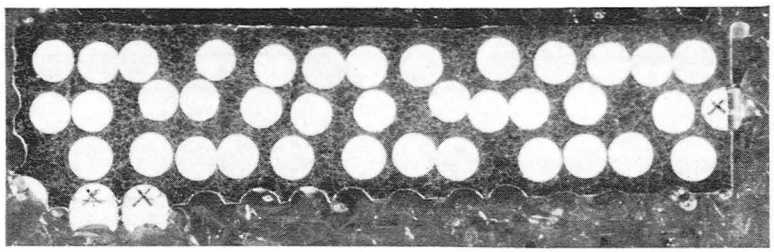
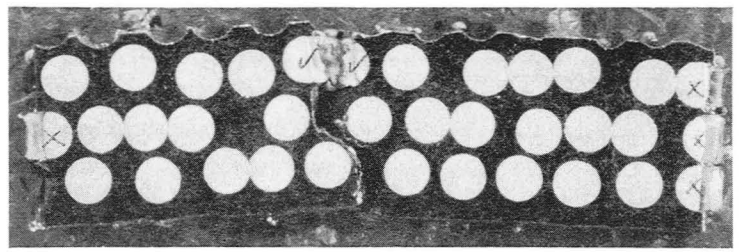


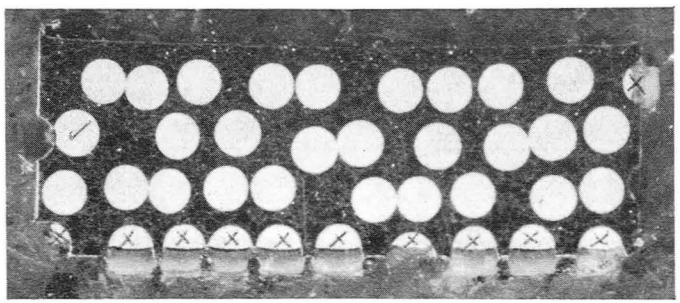
Figure 26. Typical Fracture Surface of W-Hf-C/FeCrAlY Elevated-Temperature Tensile Specimen.



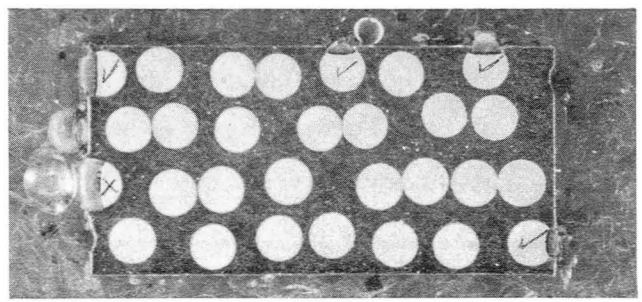
T55-1 55% 17.6X



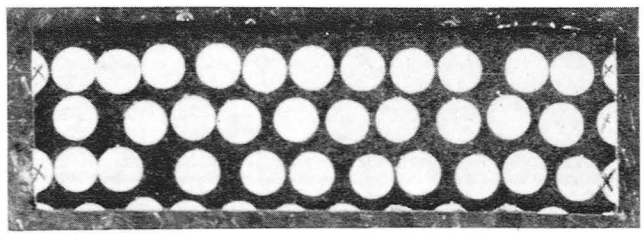
T55-2 >38v/o 18X



T55-3 42% 18X



T55-4 44.6% 16.7X



C55-2 39% 17X

Figure 27. 982°C (1800°F) Tensile Specimen No. 55-2 W-Hf-C, 55 v/o W-Hf-C, 38 mm Diameter/FeCrAlY.

The second specimen was at temperature for over a day at a fiber load of over 207 MPa (30 ksi) without showing any sign of creep over the 0.18 cm (0.070 inch) visible gage section. In contrast, three different viewers measured a small (approximately 1%) contraction in the gage region over the 30 hour period. Shortly, after we increased the fiber stress to a nominal 690 MPa (100 ksi), the specimen started to pull out of the grips. Subsequent examination of the specimen revealed that it had buckled transversely at one end. This bending may have caused the apparent contraction in the specimen gage section. The buckled end was forged flat at 260°C (500°F), sandwiched between shims, and reloaded in the furnace, but it slipped from the grip again after the application of the full 690 MPa stress at 982°C (1800°F). The specimen broke while it was being unloaded.

A subsequent 982°C (1800°F) creep test was performed on a (nominal) 55 v/o W-Hf-C/FeCrAlY specimen. The gage width had been reduced to 0.38 cm (0.150 inch) to further reduce the specimen load in hopes of preventing pullout from the grips. The test ended when the specimen pulled out of the grips. Reference lines on this specimen were photographed before and after the 220 hour test. Recognizing that no correction has been made for enlargement errors, the photos suggest that the sample has undergone 1-1/4% creep.

5.0 SUMMARY AND CONCLUSIONS

Various combinations of fiber and matrix materials were fabricated and evaluated for the purpose of selecting a specific combination that exhibited the best overall properties required for a turbine blade application. A total of seven matrix alloys, including Hastelloy X, Nimonic 80A, Inconel 600, Inconel 625, IN-102, FeCrAlY and FeNiCrAlY, were investigated reinforced with either 218CS tungsten or W-Hf-C fibers. Of major concern in this program was the composite's response to thermal cycling and much of the effort was devoted towards developing a composite having good thermal cycling properties. Based on preliminary screening studies FeCrAlY, Inconel 600 and Inconel 625 matrix composite systems were selected for extended thermal cycle tests and for property evaluations which included stress-rupture, impact and oxidation resistance. A specific fiber-matrix combination (W-Hf-C/FeCrAlY) was then selected for further property evaluation. The major conclusions from these evaluations are as follows:

1. Composites processed using powder as opposed to foil for the matrix material consistently resulted in better as-fabricated appearance and properties. In particular, there was less tendency to fail by matrix-matrix or fiber-matrix debonding.

2. Specimen dimensional instability resulting from exposure to thermal cycle testing was a function of both fiber volume fraction and matrix strength and ductility.

3. The effect of matrix alloy composition on the composites response to thermal cycling were complicated by the use of a NiCrAlY powder layer as a bonding aid to fabricate Hastelloy X, Nimonic 80A, Inconel 600 and IN-102 specimens. Non-optimum application of this bonding aid may be partially responsible for some disbonding during the thermal cycle tests. Thermal cycle test results indicated that a post-fabrication thermal exposure might be beneficial to composite properties when a bonding aid is used.

4. The FeCrAlY matrix material was the most thermal-cycle resistant material investigated. Composite specimens of W/FeCrAlY subjected to 1000 cycles of heating and cooling from 426 to 1093°C (800-2000°F) did not distort, delaminate or crack nor was any microstructural damage observed.

5. The FeCrAlY matrix material was the most oxidation resistant material investigated and the FeCrAlY matrix composite system was superior in stress-rupture compared to the other systems studied.

6. The Inconel 625 matrix composite had the best notched miniature Izod impact resistance over the range of temperatures investigated. Impact strengths of over 22.6J (200 in-lbs) were obtained at test temperatures of 260°C (500°F) and above.

7. Stress-rupture tests performed on W/FeCrAlY composite specimens previously cycled 1000 times between 426 to 1093°C (800 to 2000°F) indicated no degradation in strength due to thermal cycling.

8. No specimen distortion was observed after thermally cycling W/FeCrAlY specimens having a $\pm$ 15°, 0°, balance ply configuration with an unbalanced FeCrAlY cladding. This is significant since unbalanced claddings may be required for hollow turbine blades.

9. W-Hf-C/FeCrAlY was selected for further property evaluation. Tensile strength values of up to 724 MPa (105,000 psi) were obtained for this material at 982°C (1800°F) and 607 MPa (88,000 psi) at 1093°C (2000°F).

6.0 REFERENCES

1. Petrasek, D. W., Signorelli, R. A. and Weeton, J. W., "Refractory Metal Fiber Nickel Alloy Composites for Use at High Temperatures," NASA TN D4787, 1968.

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