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NASA Technical Memorandum 87172

Modifications of System for Elevated Temperature Tensile Testing and Stress-Strain Measurement of Metal Matrix Composites

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Prepared for the
First Symposium on Testing Technology of Metal Matrix Composites
sponsored by the American Society for Testing and Materials
Nashville, Tennessee, November 18–20, 1985

NASA

MODIFICATIONS OF SYSTEM FOR ELEVATED TEMPERATURE TENSILE TESTING AND STRESS-STRAIN MEASUREMENT OF METAL MATRIX COMPOSITES

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ABSTRACT

Composites consisting of tungsten alloy wires in superalloy matrices are being studied because they offer the potential for increased strength compared to current materials used at temperatures up to at least 1093 °C (2000 °F). Previous research at the NASA Lewis Research Center and at other laboratories in the U.S., Europe, and Japan has demonstrated laboratory feasibility for fiber reinforced superalloys (FRS). The data for the mechanical and physical properties used to evaluate candidate materials is limited and a need exists for a more detailed and complete data base. The focus of this work was to develop a test procedure to provide a more complete FRS data base to quantitatively evaluate the composite's potential for component applications. This paper will describe and discuss the equipment and procedures under development to obtain elevated temperature tensile stress-strain, strength and modulus data for the first generation of tungsten fiber reinforced superalloy composite (TFRS) materials.

Tensile stress-strain tests were conducted using a constant crosshead speed tensile testing machine and a modified load-strain measuring apparatus. Elevated temperature tensile tests were performed using a resistance wound commercial furnace capable of heating test specimens up to 1093 °C (2000 °F). Tensile stress-strain data were obtained for hollow tubular stainless steel specimens serving as a prototype for future composite specimens.

KEYWORDS

Metal matrix composites, stress-strain curves, tensile tests, elevated temperature testing, modulus of elasticity, ultimate tensile strength, yield strength.

E-2813

INTRODUCTION

Measurement of elastic modulus (E) and stress-strain behavior of materials at elevated temperatures is becoming increasingly important in aerospace design. The data base generated by this type of testing is especially important to designers working with metal matrix composites because of the versatility of fiber, matrix, and ply orientations available with these composites to meet the structural requirement of many potential applications.

High temperature stress-strain tensile tests for fiber reinforced superalloys (FRS) are needed for the generation of a data base of mechanical properties of these materials required for the quantitative evaluation of their potential application. Subsequent use of the data in an analysis/prediction computer model for the prediction of performance of potential applications will reduce the extent of the experimental work required for the FRS data base. Elevated temperature tensile stress-strain measurement of FRS will provide the designer with the elastic modulus (E), stress-strain behavior, yield strength (σ_{ys}), and ultimate tensile strength (σ_{ult}) data needed for the data base.

Preliminary experimental work at NASA Lewis has shown the feasibility to use tungsten fiber reinforced superalloys (TFRS) for elevated temperature applications between 760 °C (1400 °F) and 1204 °C (2200 °F) and offer a significant potential performance improvement when compared to other material systems. The principal potential application of TFRS has been identified as its use in blades of turbine engines. This potential has been the driving force of an extensive research effort at NASA Lewis Research Center.

Research underway at NASA Lewis can be divided in several areas. First has been the development of high temperature, high strength tungsten fibers. Second has been the selection of a good oxidation/corrosion-resistant matrix. Third, a great deal of attention has been directed toward the selection of the best fiber/matrix compatibility to minimize the reaction between fiber and the matrix, and as a result of that to minimize the degradation of the fibers properties. The fourth phase in this work has been to attempt to fabricate composite specimens in order to obtain preliminary mechanical properties. This phase was kept at laboratory scale due mainly to fabrication techniques which have been incapable of producing a large number of specimens with reproducible mechanical properties. Various fabrication techniques, involving the penetration of the matrix in the solid or liquid state into the bundles of fibers, have been applied to fabricate composite specimens and have been described in detail in a status review paper [1].

A newly developed arc spray fabrication process [2] has recently been developed at NASA Lewis and has provided a reliable technique to produce TFRS specimens for a test program to generate the needed data base for TFRS. TFRS specimens were tested at 760 °C (1400 °F), 727 °C (1700 °F), and 1093 °C (2000 °F) in tension, creep-rupture and thermomechanical fatigue in order to obtain the basic mechanical properties needed in the design of potential applications.

As part of this test program, a commercial averaging extensometry system, which is described in Ref. 3, was purchased to obtain the required stress-strain tensile data of TFRS composite specimens at elevated temperatures. Preliminary testing of composite specimens using the averaging extensometry system, revealed that problems associated with the existing apparatus impeded the accurate measurement of axial extension within the specimen gage length. To eliminate these problems and to be able to operate at higher operating temperatures, the commercial unit was modified significantly.

It is the objective of this paper to describe the equipment and test operation procedures used to obtain tensile stress-strain data for high temperature materials including metal matrix composites. Elevated temperature stress-strain data are presented to demonstrate the feasibility of the equipment modification.

DESCRIPTION OF TEST SYSTEM

An elevated temperature tensile test apparatus has been developed at NASA Lewis to conduct elevated temperature testing under axial strain control and at constant temperature. In this investigation a modification of a previously purchased elevated temperature averaging extensometry system, described in Ref. 3, was designed and built in order to obtain very accurate tensile data for the first generation of TFRS composite specimens. Although the new system required modifications to minimize problems associated with the operation of the commercial unit, the device described in this paper was based upon the system described in Refs. 3 and 4. The complete system consists of a tensile machine equipped with a resistance wound commercial furnace which is mounted on the moving crosshead, and a new strain measuring apparatus. The complete system is shown in Fig. 1 with associated controller and recorder. Loads were measured by means of an electronic load cell, while the extension of the specimen gage length was measured by two diametrically mounted strain gage extensometers located beneath the furnace. The design of the load-strain measuring apparatus and the means of controlling temperature are discussed below.

Load Application

The loading fixture, as shown in Fig. 2, consists essentially of an upper, middle, and lower pullrod, each with a coupling. The upper pullrod is attached to the load cell, which is secured to the stationary crosshead of the testing frame by means of a pin. Upper and lower threaded specimen coupling grips are used to hold the composite specimen between the upper and middle pullrods. The middle and lower pullrods are connected at two alignment couplings by means of another pin. A nut, which is threaded to one end of the lower pullrod and attached to the underside of the moving crosshead, is used to transfer the load from the constant speed moving crosshead to the loading fixture. A photograph of the assembled test fixture mounted on the testing frame is shown in Fig. 2. The furnace is shown on the side of the loading fixture.

For components operating within the furnace at elevated temperatures, which included the upper and middle pullrods and two threaded specimen coupling grips, a nickel base superalloy, MAR M246, was selected to be used because of its high strength and oxidation resistance at elevated temperatures. Stainless steel was used in the components operating outside of the furnace, which included the couplings, pins, and the lower pullrod.

The design criteria of the load train was as follows. The length of the upper pullrod was selected in order to maintain the center of the specimen at the hottest portion of the effective heating zone of the furnace to obtain a reproducible uniform temperature profile during testing. The length of the middle pullrod was designed to permit the attachment of the modified strain measurement apparatus and to limit the lower pin, which connects the alignment couplings of the middle and lower pullrods, to fall only a few milimeters before being stopped by the upper face of the moving crosshead, avoiding the damage of the thermocouples attached to the gage length of the specimen. The loading requirement is such that the pins must have a clearance fit to facilitate insertion but that such clearance be removed in order to eliminate backlash when the load frame is been calibrated. The threaded specimen coupling grips were flattened in two diametrically opposed sides to permit the strain measuring apparatus to transmit the axial extension to the strain gage extensometers avoiding metal friction which may affect the measurement of strain. Figures 3 and 4 show details of the specimen grips and the middle and lower pullrods assembly.

Modification of Strain-Measuring System

Preliminary testing using the commercial-averaging extensometry system revealed that problems associated with the strain-measuring system impeded the accurate measurement of axial extension within the specimen gage length. Three major problem areas were encountered: (1) excessive friction between the strain-measuring components, (2) inadequate fitting of these components, and (3) the fragile materials used in these components were easily broken, making the operating cost of the system prohibitive because of the cost of replacement parts.

In order to increase the clearance of the strain-measuring followers, the holes in the baffle plates were enlarged such that the follower rods were free to move without restraint. The original baffle assembly was also redesigned and shortened to eliminate angular mismatches to allow free movement of the strain followers.

The original assemblies to mount the extensometer strain ring mounting studs on the upper and lower ends of the gage length allowed excessive slack in the strain followers. The original strain rings were connected by two mounting screws which allowed rotation of the end plate when in contact with the strain followers. The system was redesigned and modified with a three-screw mounting system which reduced or eliminated rotation and provided a much more stable base for the strain followers, resulting in more accurate strain data determ- ination. These strain rings and mounting screws were made from Inco 600 alloy which allowed higher temperature operation.

The original strain-measuring system consisted of fused silica rods seated to the upper end of the gage length and fused silica tubes seated to the lower end of the gage length. The fused silica was very fragile and easily broken during insertion of the strain-measuring system into the furnace and also during fracture of the specimen. In addition, the fused silica also vitrifies at high temperatures and could cause strength and binding problems. New strain follower rods and tubes were designed and fabricated out of Inco 600, allowing higher temperature operation and eliminating breakage problems.

Strain Measurements

Strain measurements were performed by means of a modified averaging extensometer system that monitored the relative axial displacement of the ends of the specimen gage length. The apparatus, which is shown in Figs. 2 to 4 consists of the upper and lower gage length or strain rings, two sets of metal strain follower rod and tube assemblies, each with a pair of spring-loaded blocks, and a tubular baffle assembly having a base block mounted with two diametrically opposed ball-and-spring units. The two pairs of spring-loaded blocks, called the upper and lower guide blocks, are secured to the strain follower tubes and rods, respectively. The rods are lightly loaded between the ball-and-spring units inserted in the assembly base and the two tabs of the upper gage length ring. The tubes are similarly loaded between the upper spring-loaded guide block and the lower gage length ring tabs. Two strain gages, which are connected to the knife-edge supports of the guide blocks, are used to measure the axial extension of the specimen gage length at two opposite sides of the specimen diameter.

The two measured strain signals from the strain gages are fed into a dual input adaptor, which is connected to the strain data unit through the frame/console wiring, and electronically summed to give an average extension signal, which together with the load cell signal are continuously transmitted to a servo drive recorder that generates load/specimen axial extension curves for permanent record.

Specimen Heating and Temperature Control

In this investigation tensile tests were performed using a commercial furnace system, which is part of the existing system described in Refs. 3

and 4. The system consists of a vertical-bore type furnace and a modified temperature controller, which is used to provide and to control the power supplied to each of the three zones of the furnace.

The furnace consists of three independent 500 W heater windings with an overall heat zone of approximately 300 mm (11.81 in). Temperatures up to 1350 °C (2462 °F) are permitted in the furnace windings. Since the power was supplied and controlled independently in each winding, the end zones of the furnaces were used to compensate for heat losses, and together with the center zone winding provided a means of adjusting the temperature gradient along the specimen gage length. Since the furnace is mounted to the moving crosshead, a better temperature profile is maintained. The power supply and the specimen temperature were controlled by means of a solid-state electronics set point controller, which was modified to permit operation at higher temperatures. This system is described in detail in Ref. 4.

The furnace winding had a maximum operating temperature capability of 1350 °C (2462 °F), however, the set point controller and associated wiring had an electrical current limitation that restricted furnace operation to 1000 °C (1832 °F). The existing controller and associated interior wiring was modified to increase the temperature capability of the furnace to 1093 °C (2000 °F) which allowed higher temperature operation, while still retaining a safe operational life for the furnace winding.

Two platinum/platinum-13 percent rhodium thermocouples were used to measure the surface temperature at the specimen gage length when testing. Both thermocouples were attached to the specimen by means of a high temperature commercial steel wire as shown in Fig. 3. The specimen thermocouples, which were passed down along the baffle assembly of the loading-strain measurement apparatus, have their leads guided and isolated by ceramic beads and tubes. A digital readout, connected to the thermocouples leads by means of a polarized two-pole connector, was used to display the surface temperatures of the specimen. In practice, three thermocouples were used to obtain the initial surface temperature profile on the gage length of a dummy specimen.

MATERIALS AND EXPERIMENTAL PROCEDURES

The modulus and strength properties of unidirectional reinforced composites can be determined accurately with standard tensile test specimens. However, the testing of composites with angleplied fiber orientations becomes much more critical due to edge effects of angleplied fibers ending at the free edge of the gage length, rather than extending completely through the gage length. If the specimen width is increased to minimize these edge effects, the failure loads can become excessive for a specimen of reasonable thickness.

In order to obtain accurate design data for angleplied metal matrix composites, a tubular specimen was designed at NASA Lewis. The hollow tubular specimen eliminated edge effects because the plies are wrapped around each other, such that no free edge exists and the fibers are continuous along the entire gage length of the tube.

A newly developed arc-spray fabrication process developed at NASA Lewis provided a reliable technique to produce a large quantity of TFRS monotapes which were used in the production of composite specimens. In that process a TFRS monotape is produced by arc spraying the matrix material over a drum which has tungsten fibers wound on its surface. Hollow tubular composite specimens are produced by simultaneously consolidating the TFRS monotapes and diffusion bonding the grips in a high temperature press. The arc spray technique is described in details in Ref. 2.

The hollow tubular specimen geometry is shown in Fig. 5. Test specimen grips were machined using conventional tooling after completion of the consolidation process. The geometry was selected so that the anticipated tensile strength of the composite hollow tube was below the the minimum shear strength of the bonding between the composite tube and the grips. The required gripping length of 50.8 mm (2 in.) was determined based on that assumption, while a gage length of 25.4 mm (1 in.) was established arbitrarily. The grips are two stainless steel threaded ends which have a radius at the gage length to eliminate stress concentration effects. The threaded end grips have a circumferential groove in which the set screws of the gage length rings are fitted.

The procedure for a typical high temperature material test was as follows. The specimen threaded end grips were covered with a mixture formed by mixing 1000 ml of cellouse nitrate, 500 g of boron nitrate, and 200 ml of acetone to avoid binding between the specimen threaded ends and the load fixture threaded grips. The specimen and the load-strain measuring apparatus were then assembled as shown previously in Fig. 2. At that time, the gage length rings and the rod and tube assemblies were checked for accurate alignment. The furnace was swung out from that testing machine and was preheated to the test temperature. The specimen and load-strain measuring assembly was inserted into the furnace and the furnace was swung back into position within the loading frame and the assembly was connected to the stationary crosshead. The complete assembly was heated to test temperature without connecting it to the moving crosshead to allow free thermal expansion of its components. While the specimen was heating, the strain gage extensometers were calibrated against high magnification micrometer standards by adjusting the movement of the recording chart to the measured strain from the extensometer mounted in the external calibrator. After attaining test temperature, the lower pullrod was connected to the assembly and to the moving crosshead. The load calibration and zeroing were performed using the recorder chart to eliminate the compressive effects of the middle and lower loading elements on the specimen. The test specimen was held at the test temperature for a minimum of 5 min before the strain gage extensometers were connected to the knife-edges of the guide blocks and zeroed using the servo drive recorder chart. The moving crosshead was activated to provide the desired crosshead loading rate.

All tests were conducted using a crosshead speed of 0.25 mm/min (0.01 in/min). A very sensitive scale of 0.25 mm (0.01 in.) of specimen gage length axial extension per 508 mm (20 in.) of chart movement was used to record the elastic modulus portion of the stress-strain curve, while the remainder of the curve was recorded using scales from 0.25 mm (0.01 in.) to 5.0 mm (0.2 in.) of specimen gage length axial extension per 508 mm (20 in.) of chart movement. Load-strain curves were recorded for each test and converted to stress-strain curves using the initial measured gage length in strain calculations, and the initial calculated cross section area in stress calculations.

TEST RESULTS

The performance of the test system was checked using hollow tubular specimens of type 304 stainless steel described previously in this paper. Stainless steel was selected because of convenient availability and because of the extensive literature existing on its mechanical properties at elevated temperatures.

Tests performed on specimens have demonstrated that the heating time from the time the specimen is loaded into the furnace to the time the specimen temperature reached the set point temperature is approximately 45 min at 760 °C (1400 °F). The temperature can be held constant to less than ±2.8 °C (5 °F) at 927 °C (1700 °F) as long as the test lasts (5 to 10 min) when using the control system modified for this investigation. The axial temperature gradient along the specimen gage length was reduced to within ±°2 °C (4 °F) of the temperature at the center of the specimen as shown in Fig. 6.

Initial verification tensile tests were performed on type 304 stainless steel specimens at the 760 °C (1400 °F) in air. In this test, a crosshead speed of 0.25 mm/min (0.01 in./min) was used and the procedure explained previously was followed.

A stress-strain curve obtained from a hollow tubular type 304 stainless steel specimen tested at 760 °C is shown in Fig. 7. The low strain region is expanded in Fig. 7(a) and shows that good linearity was obtained in the elastic region and a modulus of elasticity of 138 GPa (20 Msi) was obtained. The specimen had an elastic strain limit of about 0.055 percent strain. The expanded portion of the stress-strain curve also shows that a 0.2 percent offset yield strength of 142 MPa (21 ksi) was obtained.

The full stress-strain curve obtained is shown in Fig. 7(b). Due to the procedure used to establish the initial balance of the extensometer, the stress-strain curve could be accurately measured to a strain of 4.5 percent. An ultimate tensile strength of 171 MPa (24.8 ksi) was obtained for this specimen. This extensometer strain measurement limit is sufficient to determine the plastic stress-strain behavior of composite materials and to allow determination of plastic work hardening coefficients if desired. For less ductile materials, the failure strain can be determined from this type of stress-strain curve, while for more ductile materials, failure strain can be measured directly from the failed specimens.

These data on hollow tubular type 304 stainless steel specimens show good agreement with literature of a modulus of 133.8 GPa (19.4 Msi) and an ultimate tensile strength of 202.7 MPa (29.4 ksi) at 760 °C (1400 °F) values published in Ref. 5. These specimens served as prototypes for hollow tubular TFRS specimens and were used to verify the load-strain measuring system. Future work will include the testing of TFRS composites to temperatures up to 1093 °C (2000 °F). The results obtained indicate that hollow tubular TFRS specimens can be successfully tested and an accurate determination of modulus of elasticity (E), elastic strain limit, yield strength (σ_{ys}), and ultimate tensile strength (σ_{ult}) can be obtained with this modified testing system.

CONCLUSIONS

A tensile test system has been developed to test hollow tubular TFRS composite specimens under axial strain control. The system consists of a conventional tensile test frame, a commercial resistance wound furnace, and a modified loading-strain measuring apparatus. The system is capable of performing tensile tests at temperatures up to 1093 °C.

The description and testing procedures of this apparatus have been presented. To demonstrate the capabilities of the equipment, tensile tests were conducted on hollow tubular specimens of type 304 stainless steel. These specimens were prototypes for future evaluation of the stress-strain behavior of metal matrix composites at elevated temperatures.

ACKNOWLEDGMENT

The author is grateful to Mr. David McDanels, Mr. Robert Signorelli, Mr. Donald Petrasek, and Mr. Leonard Westfall for many stimulating suggestions, to Mr. John Yuhas and Mr. James Nickle for making the components of the apparatus, to Mr. Allan Bollam for the modifications made to the furnace control system, and specially to Mr. Gerald Beremand for the preparation of the material used in this investigation and his help in carrying out the experiments; all from NASA Lewis Research Center.

REFERENCES

[1] Petrasek, D.W. and Signorelli, R.A., "Tungsten Fiber Reinforced Superalloys - A Status Review," NASA TM-82590, National Aeronautics and Space Administration, Washington, D.C., 1981.

[2] Westfall, L.J., "Tungsten Fiber Reinforced Superalloy Composite Monolayer Fabrication by an Arc-Spray Process," NASA TM-86917, National Aeronautics and Space Administration, Washington, D.C., 1985.

[3] "Instron High Temperature (In-Air) Furnace With Averaging Extensometry," Manual 1-7-46-2A, Instron Limited, High Wycombe, Buckinghamshire, England.

[4] "Instron 3-ZONE Temperature Controller," Manual 1-7-71-2, Instron Limited, High Wycombe, Buckinghamshire, England.

[5] Peckuer, D. and Bernstein, I.M., Handbook of Stainless Steels, McGraw-Hill, New York, 1977.

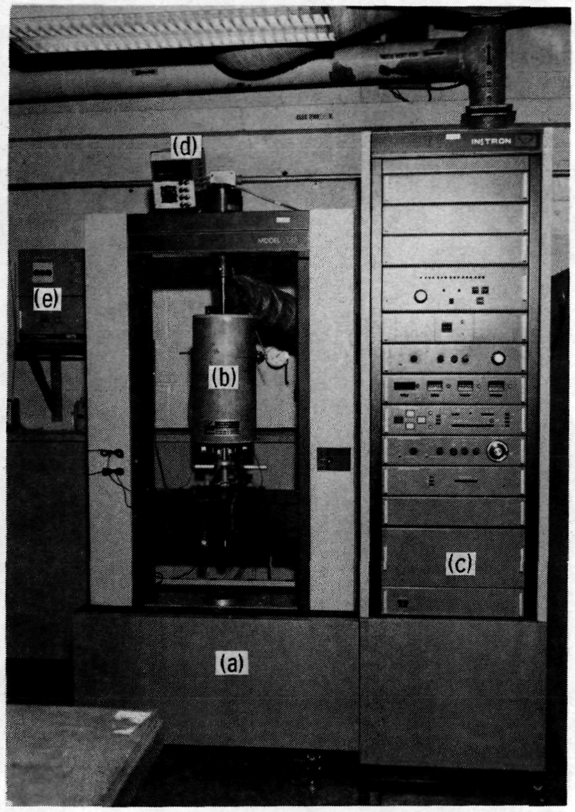

Figure 1. - Experimental setup: (a) Load frame, (b) furnace, (c) load control system and chart recorder, (d) temperature indicator and, (e) temperature controller.

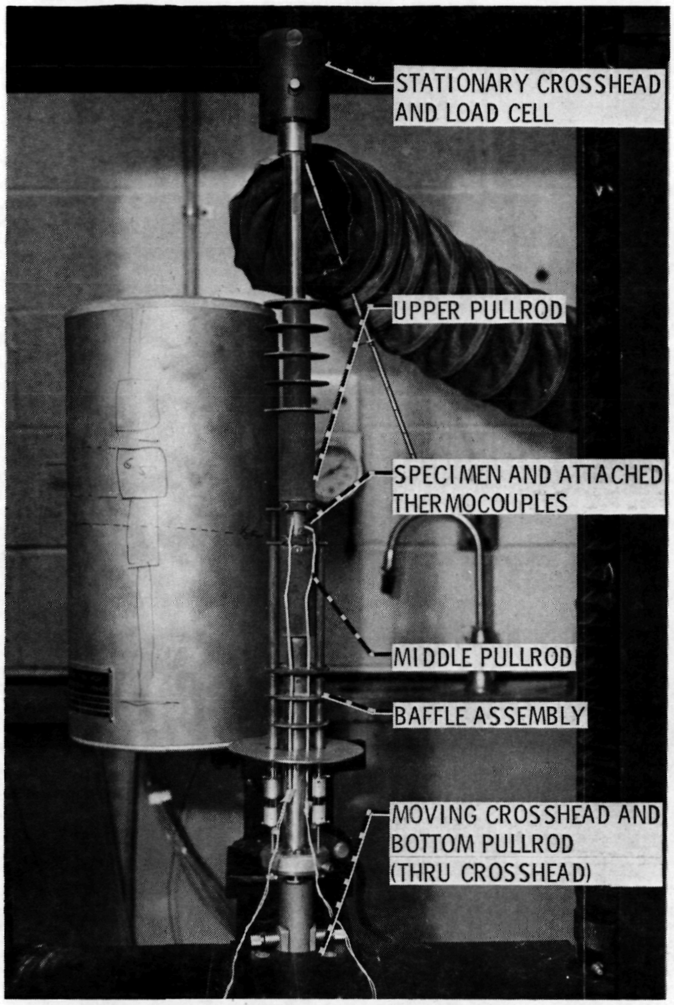

Figure 2. - Assembled text fixture mounted in load frame.

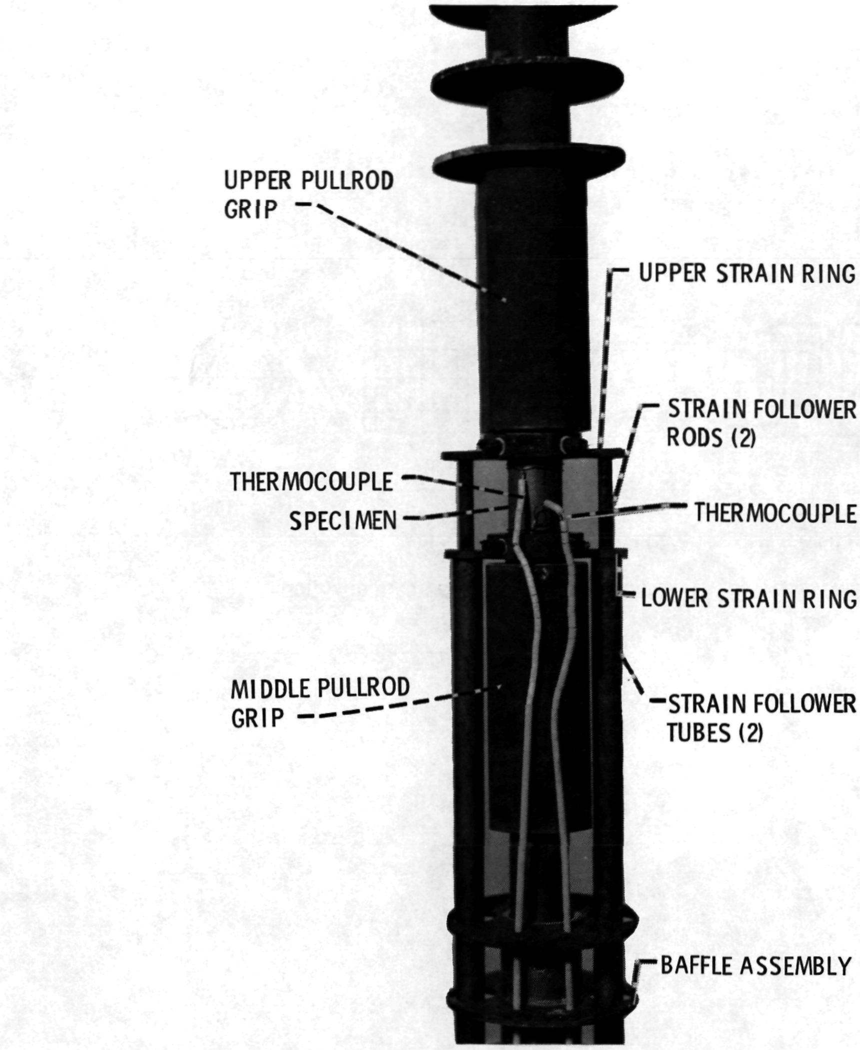

Figure 3. - Pullrods and averaging extensometry assembly with a specimen.

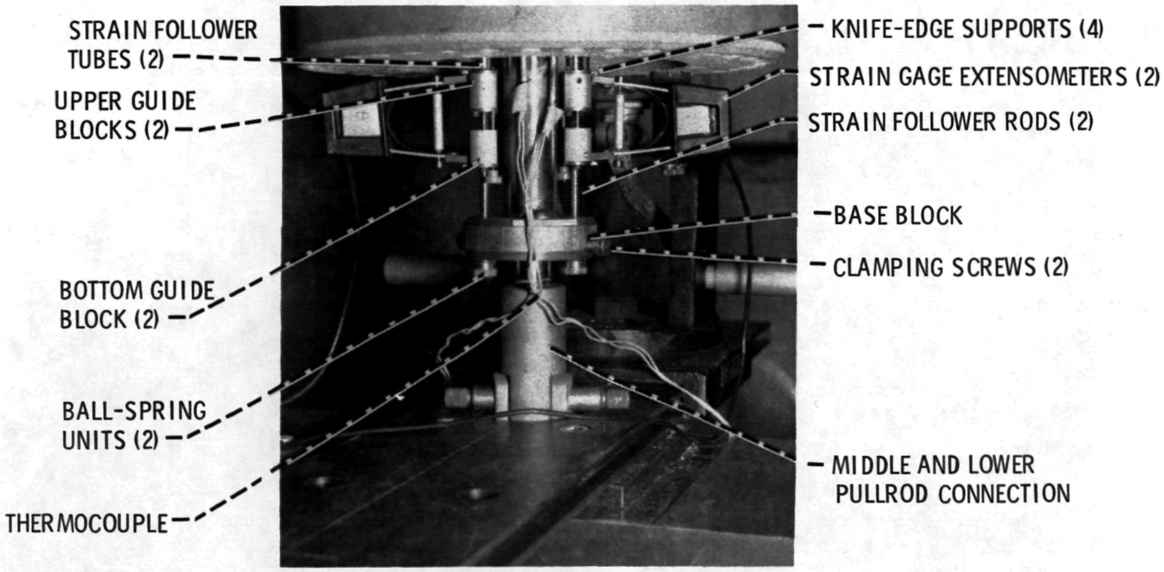

Figure 4. - Fitting of strain follower elements and strain gage extensometer.

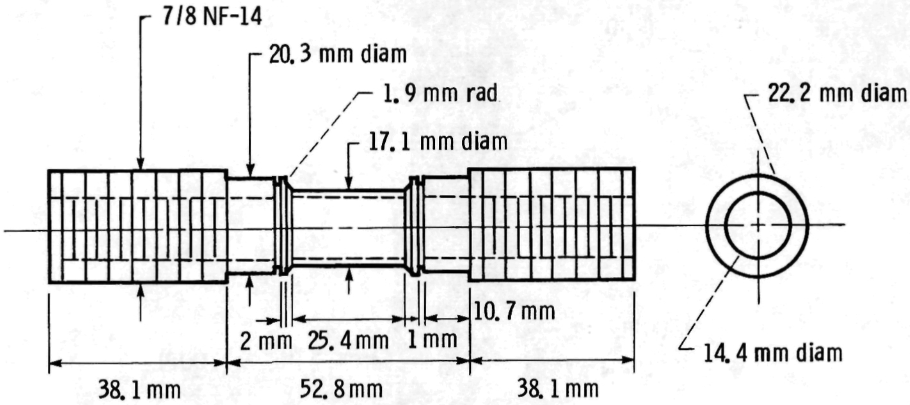

Figure 5. - Hollow tubular specimen configuration.

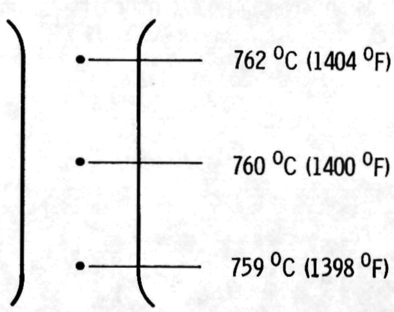

Figure 6. - Temperature profile in tubular specimens.

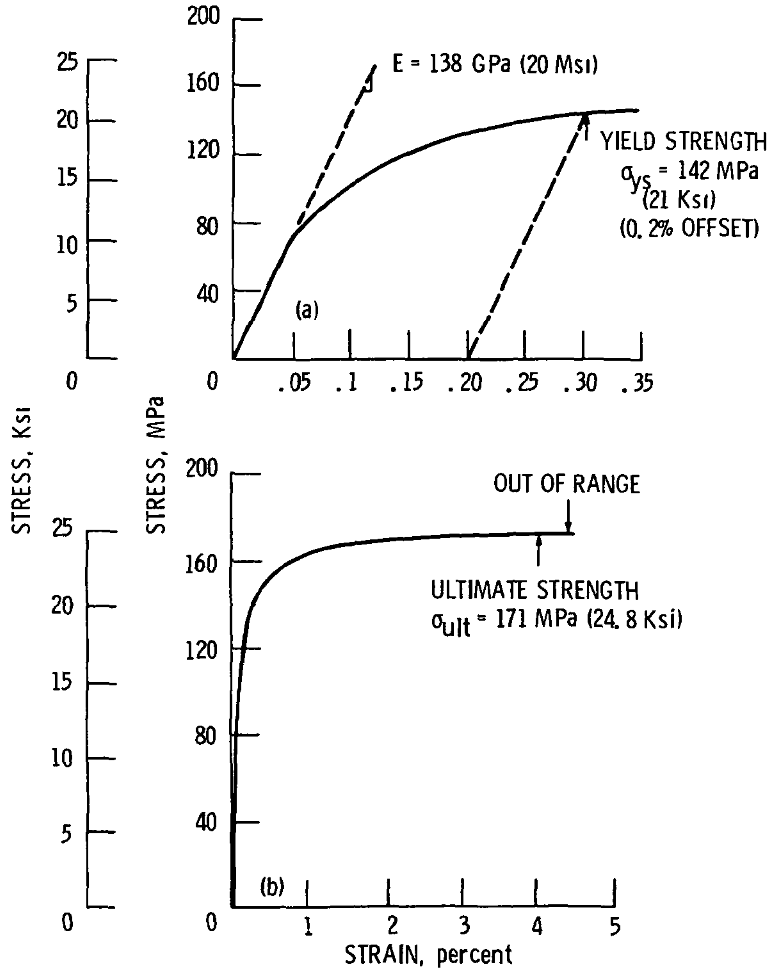

Figure 7. - Stress-strain curves of type 304 Stainless Steel tensile specimen tested at 760 °C (1400 °F).

(a) Enlargement of low strain region.
(b) Total measured stress-strain curve.

1 Report No NASA TM-87172	2 Government Accession No	3 Recipient's Catalog No
4 Title and Subtitle Modifications of System for Elevated Temperature Tensile Testing and Stress-Strain Measurement of Metal Matrix Composites		5 Report Date
		6 Performing Organization Code 505-63-81
7 Author(s) Juan O. Diaz		8 Performing Organization Report No E-2813
		10 Work Unit No
9 Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135		11 Contract or Grant No
		13 Type of Report and Period Covered Technical Memorandum
12 Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		14 Sponsoring Agency Code

15 Supplementary Notes

Prepared for the First Symposium on Testing Technology of Metal Matrix Composites sponsored by the American Society for Testing and Materials, Nashville, Tennessee, November 18-20, 1985.

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17 Key Words (Suggested by Author(s)) Metal matrix composites; Stress-strain curves; Tensile tests; Elevated temperature testing; Modulus of Elasticity; Ultimate tensile strength; Yield strength	18 Distribution Statement Unclassified - unlimited STAR Category 23

19 Security Classif (of this report)	20 Security Classif (of this page)	21 No of pages	22 Price*
Unclassified	Unclassified		

*For sale by the National Technical Information Service, Springfield, Virginia 22161

National Aeronautics and
Space Administration

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