FOREWORD

This volume is the thirty-fifth of the series WADD Technical Report 61-72 describing various phases of research and development on advanced graphite materials conducted by National Carbon Company, a Division of Union Carbide Corporation, under USAF Contract No. AF 33 (616)-6915.

The work covered in this report was conducted from July 1, 1961 through April 30, 1963 at the Advanced Materials Laboratory of National Carbon Company, Lawrenceburg, Tennessee, under the management of R. M. Bushong, Director of the Advanced Materials Project, and of R. C. Stroup, Manager of the Advanced Materials Laboratory.

The contract for this R&D program was initiated under Project No. 7350, "Refractory Inorganic Non-Metallic Materials," Task No. 735002, "Refractory Inorganic Non-Metallic Materials: Graphitic;" Project No. 7381, "Materials Application," Task No. 738102, Materials Processes;" and Project No. 7-817, "Process Development for Graphite Materials." The work was administrated by the Air Force Materials Laboratory, Research and Technology Division. Captain R. H. Wilson, L. J. Conlon and W. P. Conrardy acted as Project Engineers.

Other volumes in this WADD Technical Report 61-72 series are:

Volume I - Observations by Electron Microscopy of Dislocations in Graphite, by R. Sprague.

Volume II - Applications of Anisotropic Elastic Continuum Theory to Dislocations in Graphite, by G. B. Spence.

Volume III - Decoration of Dislocations and Low Angle Grain Boundaries in Graphite Single Crystals, by R. Bacon and R. Sprague.

Volume IV - Adaptation of Radiographic Principles to the Quality Control of Graphite, by R. W. Wallouch.

Volume V - Analysis of Creep and Recovery Curves for ATJ Graphite, by E. J. Seldin.

Volume VI - Creep of Carbons and Graphites in Flexure at High Temperature, by E. J. Seldin.

Volume VII - High-Density, Recrystallized Graphite by Hot-Forming, by E. A. Neel, A. A. Kellar and K. J. Zeitsch.

Supplement	-	High-Density Recrystallized Graphite by Hot-Forming, by G. L. Rowe and M. B. Carter.
Volume VIII	-	Electron Spin Resonance in Polycrystalline Graphite, by L. Singer and G. Wagoner.
Volume IX	-	Fabrication and Properties of Carbonized Cloth Composites, by W. C. Beasley and E. L. Piper.
Volume X	-	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Supplement	-	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Volume XI	-	Characterization of Binders Used in Fabrication of Graphite Bodies, by E. de Ruiter, A. Halleux, V. Sandor and H. Tschamler.
Supplement	-	Characterization of Binders Used in Fabrication of Graphite Bodies, by E. de Ruiter, J. F. M. Oth, V. Sandor and H. Tschamler.
Volume XII	-	Development of an Improved Large-Diameter, Fine-Grain Graphite for Aerospace Applications, by C. W. Waters and E. L. Piper.
Supplement	-	Development of an Improved Large-Diameter, Fine-Grain Graphite for Aerospace Applications, by R. L. Racicot and C. W. Waters.
Volume XIII	-	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and E. L. Piper
Supplement	-	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and R. L. Racicot.
Volume XIV	-	Study of High-Temperature Tensile Properties of ZTA Grade Graphite, by R. M. Hale and W. M. Fassell.
Volume XV	-	Alumina-Condensed Furfuryl Alcohol Resins, by C. W. Boquist, E. R. Neilsen, H. J. O'Neil and R. E. Putcher.
Volume XVI	-	An Electron Spin Resonance Study of Thermal Reactions of Organic Compounds, by L. L. Singer and I. C. Lewis.

ABSTRACT

This report describes methods and procedures for measuring short-time ultimate tensile, compressive, shear and flexural (modulus of rupture) strengths of graphite, and the stress-strain relationships in tension, compression and shear in the 20° to 2700°C range. It presents also the description of a method being developed for determining Poisson's ratio in tension and compression at room and elevated temperatures.

This technical documentary report has been reviewed and is approved.

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1. INTRODUCTION

Knowledge of the mechanical properties of graphite, particularly at high temperatures, is extremely important in the design of advanced weapon systems. The characterization of commercial and experimental graphite grades intended for use in this application must include ultimate short-time strength and stress-strain curves for uniaxially applied loads. The purpose of this report is to describe the techniques for the measurement of tension, compression, shear and flexural strength properties of graphite at room and at elevated temperatures used by the Advanced Materials Laboratory of National Carbon Company under U. S. Air Force Contract No. AF 33(616)-6915 in obtaining the data reported in other volumes of the WADD TR-61-72 series.

Orientation of the grain in graphite is of importance when measuring physical properties. In molded graphites, there are two with-grain and one across-grain orientations. The directions selected for study represent the maximum and minimum effects of the grain orientation on the strength of the graphite.

Manuscript released by the authors June 1963 for publication as an RTD Technical Report

2. TENSILE STRENGTH MEASUREMENTS

The short-time tensile strength of graphite is determined in the same manner as for most other materials; i.e., by applying a tensile load to a specimen of a given cross section and dividing the load at fracture by the cross-sectional area. Because graphite is a brittle material below 2000°C, the primary consideration in the design of a test to determine the short-time tensile strength of graphite is the alignment of forces on the specimen in such a way that only tensile stresses cause failure of the specimen.

Several techniques have been used for tensile measurements of graphite, but each has one or more of the following shortcomings:

1) Some techniques cannot be used readily at high temperatures because the specimen configuration necessitates use of very bulky grips.

2) Difficult or impossible alignment causes fracture to occur on the shoulder, rather than in the gauge length of the specimen.

3) Some techniques do not lend themselves to stress-strain measurements because the gauge length of the specimen is not well defined.

4) The overall method is too ponderous to make a large number of tests in a reasonable amount of time.

The main criteria for a good tensile test are:

1) Stresses other than tensile must be eliminated or reduced to a negligible amount.

2) The same degree of accuracy must exist at high temperatures as at room temperature.

3) The test must be capable of giving accurate stress-strain curves.

4) Variations in test results must be due primarily to the material, not to the test.

5) The test must be rapid enough to establish reliable levels of variation in a reasonable length of time.

2.1. Literature Survey

A literature survey revealed several techniques which might be used for measuring the tensile strength of graphite. The brittle-ring test[1] was considered most carefully and tests were made at room and at elevated temperatures to obtain data for comparison. Although the brittle-ring method apparently solves the alignment problem, it could not be used because of its inability to provide stress-strain data at high temperatures and because some controversy remains as to whether it is a valid tensile test.

The theta test method[2] was rejected because the configuration of the specimen is complicated and difficult to machine. This method also presents difficulties in obtaining stress-strain curves.

The cross-breaking technique of Roup and Fillmore[3] was also considered. This is a modified flexural test in which holes of various diameters are drilled through the width of the test bar along its transverse axis to concentrate the compressive and tensile forces respectively at the top and bottom of the bar. Since brittle materials are stronger in compression than in tension, the bar will in theory fail from tensile loading. The test is not suited for stress-strain measurements.

A test, designed after Bressman's method,[4] was used for the work described in this report and employed a specimen with conical ends. This test makes allowance for the viscoelasticity of graphite and the temperature dependence of graphite properties.

2.2. Specimen

The specimen adopted for routine testing is shown in Figures 1 and 2. Ultimate strengths obtained with this configuration are higher than those commonly reported in the literature because this type of specimen is more easily aligned which minimizes shearing stresses. Machining techniques used for the preparation of the specimen are of the utmost importance for a successful test. The photograph in Figure 1 shows the surface finish of a machined specimen before testing. The carbide cutting tool shown in Figure 3 proved to be superior to tools of conventional design for machining tensile strength specimens. This carbide tool is not precisely a contour tool because the horizontal cutting edge is half an inch shorter than the specimen gauge length.

Figure 4-A gives strain curves for a specimen machined with the tool shown in Figure 3, while Figure 4-B shows similar curves for a specimen machined with a conventional tool bit guided by a metal follower riding on a specimen profile cam. The latter tended to form a

helix running the full length of the specimen. Strain in these tests was measured by mounting two SR-4 strain gauges 180° apart on the gauge length of the specimen with the longitudinal axes of the gauges parallel to those of the specimens. The parallelism of the curves in Figure 4-A indicates good alignment of the forces, while the opposite is indicated in Figure 4-B.

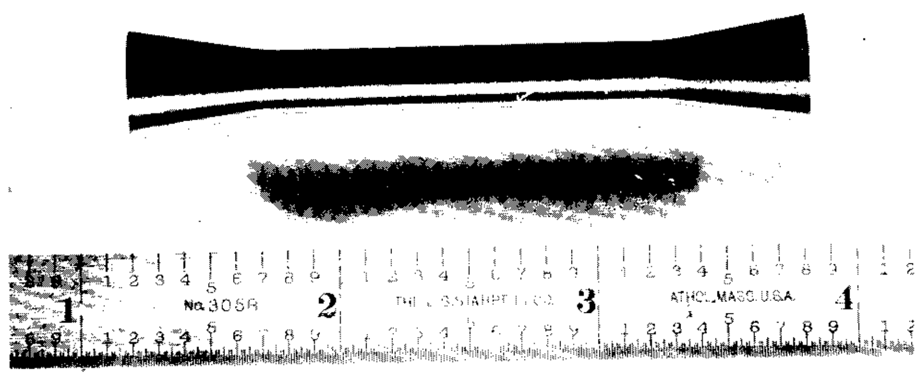

Figure 1. Photograph of Standard Specimen for Measuring Tensile Strength of Graphite

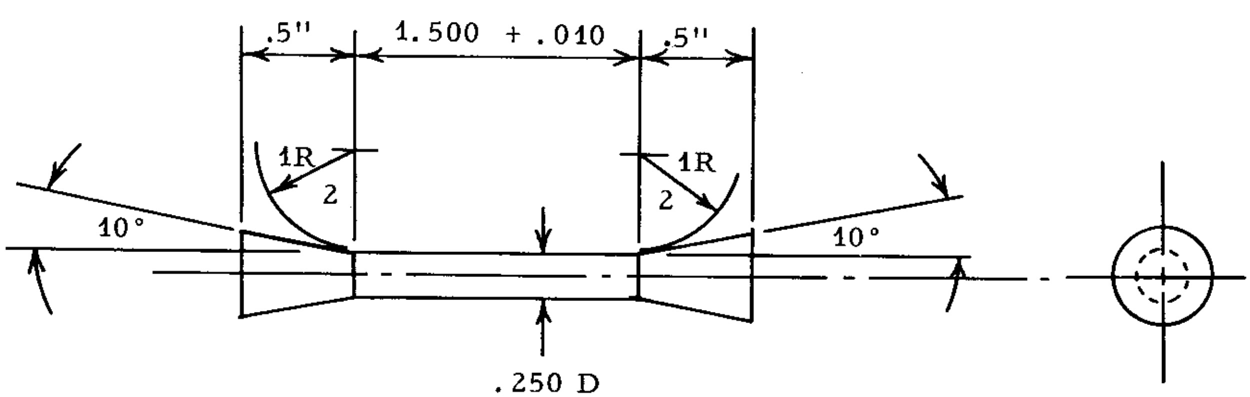

Figure 2. Drawing of Standard Specimen for Measuring Tensile Strength of Graphite

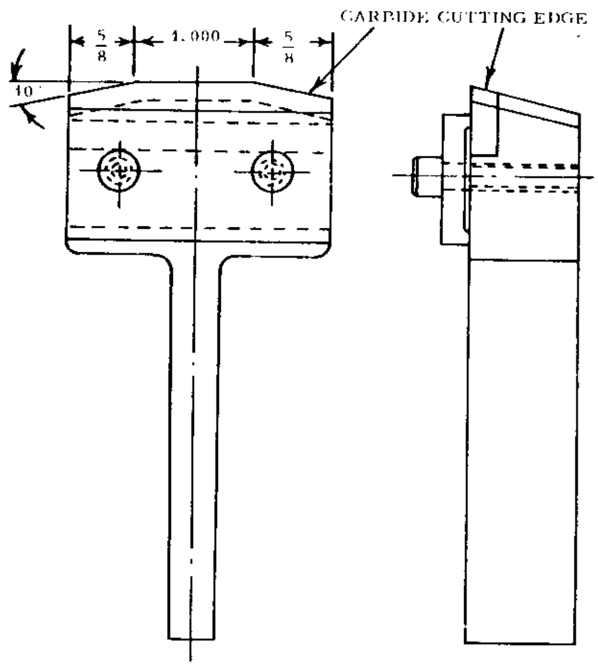

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Figure 3. Drawing of Cutter for Making Standard Tensile Specimen

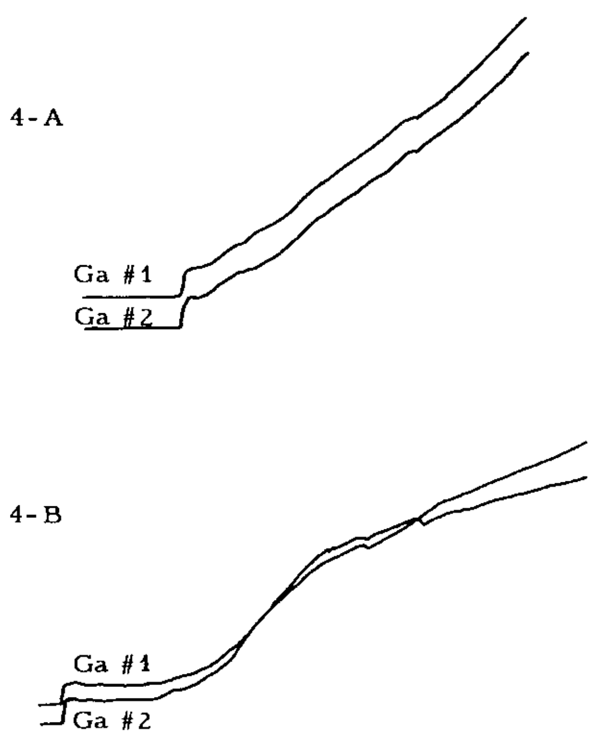

Figure 4. Strain Gauge Curves Showing Alignment of Forces in Tensile Specimens Cut by Two Methods

Surface blemishes on graphite can be very troublesome, even when these blemishes are outside the gauge length, as is shown in Figures 5, 6 and 7. The almost imperceptible notch in the cutting edge of the carbide blade (Figure 5) produced a ridge on the specimen shank (Figure 6), which caused the compressive break shown in Figure 7. Since it is imperative that the cutting tool be sharp and free of flaws it should be examined frequently by means of a magnifying glass.

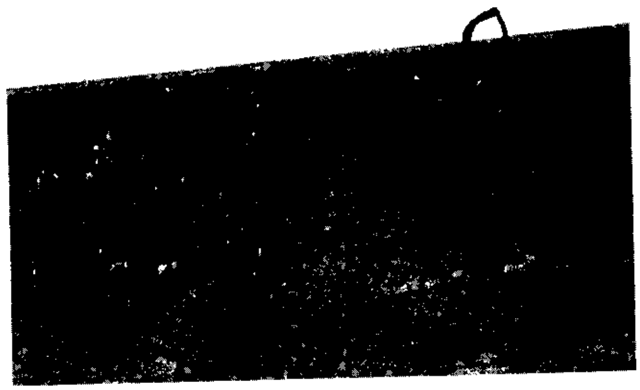

Figure 5. Macrograph (10 X) of Notch on the Cutting Edge of a Tensile Specimen Cutter

Figure 6. Macrograph (10 X) of a Ridge on a Specimen which was Made with the Cutter Shown in Figure 5

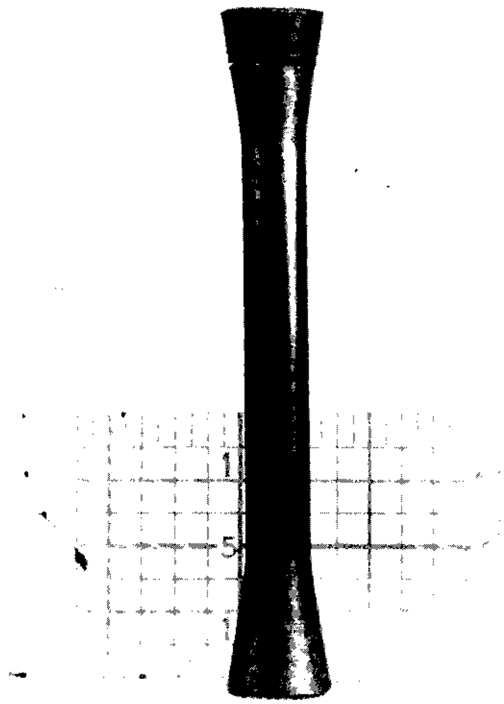

Figure 7. Photograph of Specimen With a Compressive Fracture Caused by the Ridge Shown in Figure 6

2.3. Equipment and Measurements

2.3.1. Furnace

High-temperature tensile measurements are made in a 3-inch OD by $2^1/_2$ -inch ID by 30-inch length graphite tube furnace insulated with 4 inches of lampblack carbon flour. The furnace is mounted on a lift so that it can be removed from the test machine to cool while a second furnace, mounted in the same manner, is put in use. Figure 8 is a photograph of a furnace set into place in the test machine.

Temperature measurements are made directly on the specimen, through a carbon sight tube, with an optical pyrometer. Thermal expansion of the specimen has no effect upon measurements because the specimen is not mechanically stressed until thermal equilibrium is reached.

To maintain a neutral atmosphere, to prevent oxidation of the hot graphite inside the furnace, and to keep a clear sight path for temperature measurement, argon gas is injected into the furnace and through the sight tube. Air is prevented from entering the furnace through the bottom by a boron nitride cover which is locked on with metal brackets.

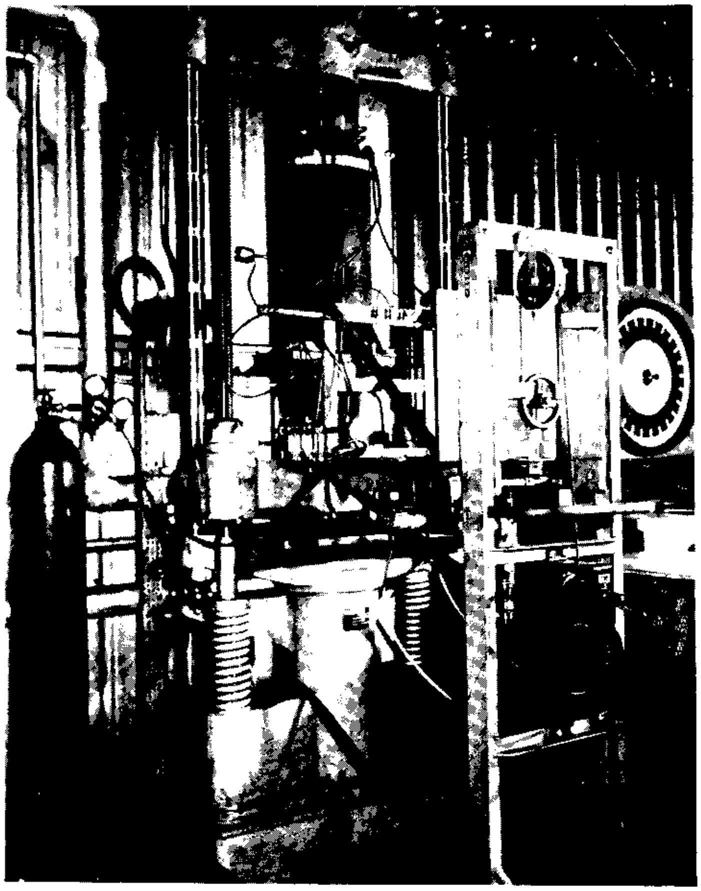

Figure 8. Photograph of a Vertical Tube Furnace Placed in the Test Machine in Preparation for a Test to be Made at Elevated Temperature

Power is supplied to the furnace by an autotransformer with a 480-v primary and a 9- to 560-v secondary connected to a 30-kva, single-phase, 60-cycle air-cooled dry-type transformer with 480-v primary, 9.60-v secondary at 3300 amperes. A circuit is made to the tube through water-cooled cables and water-cooled split copper-electrode blocks bolted to each end of the tube.

2.3.2. Grips and Pull Rods

The initial design of specimen grips and pull rods for high temperature tensile measurements is shown in the sketch in Figure 9. Stress concentration caused the grips and pull rods to crack at the threads and sharp edges. A new system of grips and pull rods was designed which had no sharp edges or corners. Figure 10 is a photograph of two of these assemblies; one assembled with pull rod and specimen in place, the other disassembled to show how the pull rod and specimen are held.

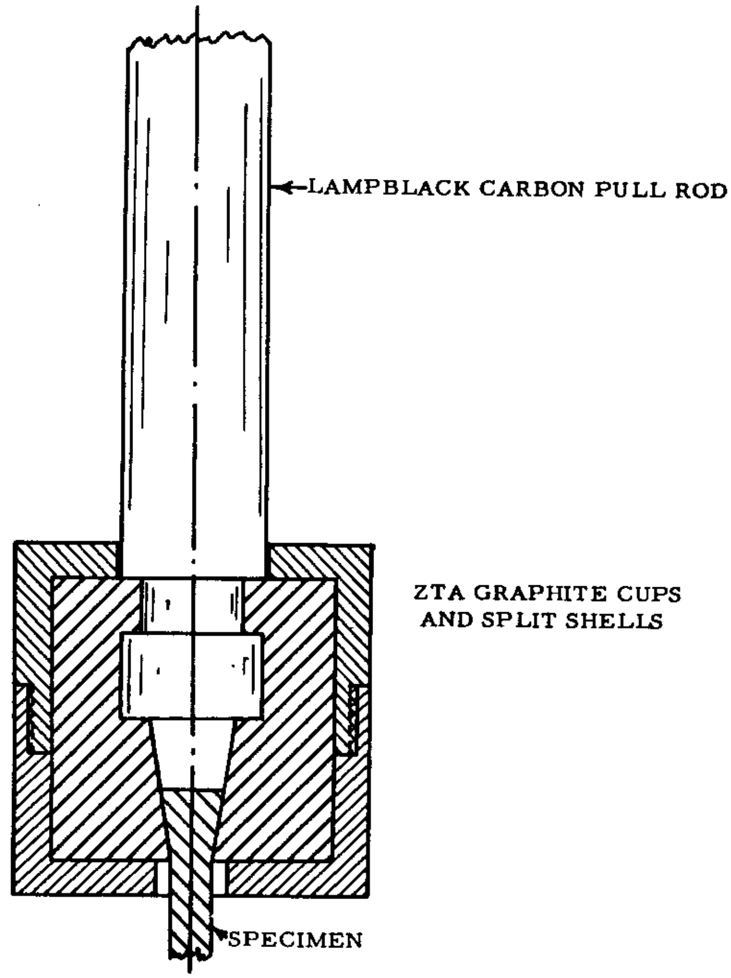

Figure 9. Sketch of Initial Design of Grips and Pull Rods Used for First High-Temperature Tensile Tests

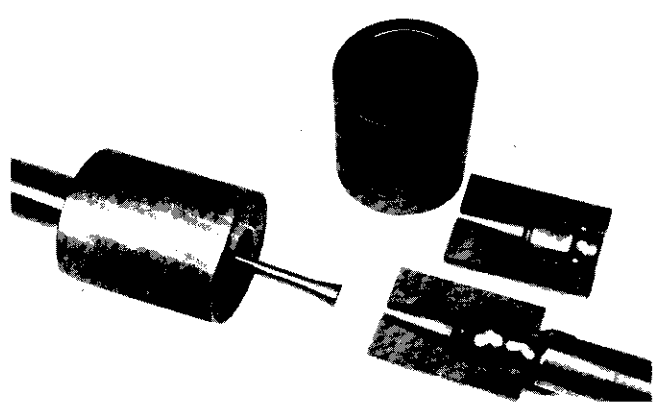

Figure 10. Photograph of Grips and Pull Rods Presently Being Used for Tensile Tests

Grips and pull rods (1 inch in diameter by 16 inches in length), used for high-temperature tensile tests, are made of grade ATJ graphite. To lessen the degree of mismatch in thermal expansion at elevated temperatures, all parts are machined from a single block of graphite. Care is taken to orient the grain direction of the graphite parallel to the axis of the load train; i.e., parallel to the tensile stress so that the highest tensile strength of the load train is utilized.

To obtain maximum alignment of tensile forces in the specimen, the pull rods are attached to the crossheads of the testing machine by box clamps which rotate on $^1/_2$-inch diameter steel balls so that both ends of the load train are free to move in any direction. Figure 11 is a close-up view of the box clamps; a schematic drawing of the entire assembly, mounted in the furnace for a high-temperature test, is shown in Figure 12.

A metal load train with short pull rods is used for room-temperature measurements.

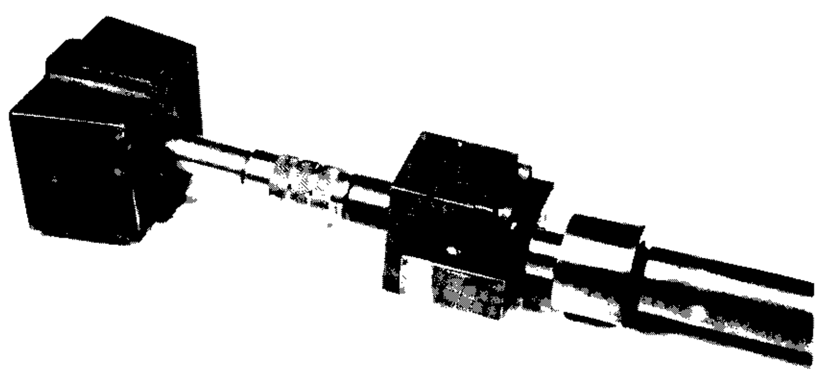

Figure 11. Photograph Showing Close-Up View of Box Clamps Used to Connect Tensile Pull Rods to V-Wedge in Test Machine Crosshead.

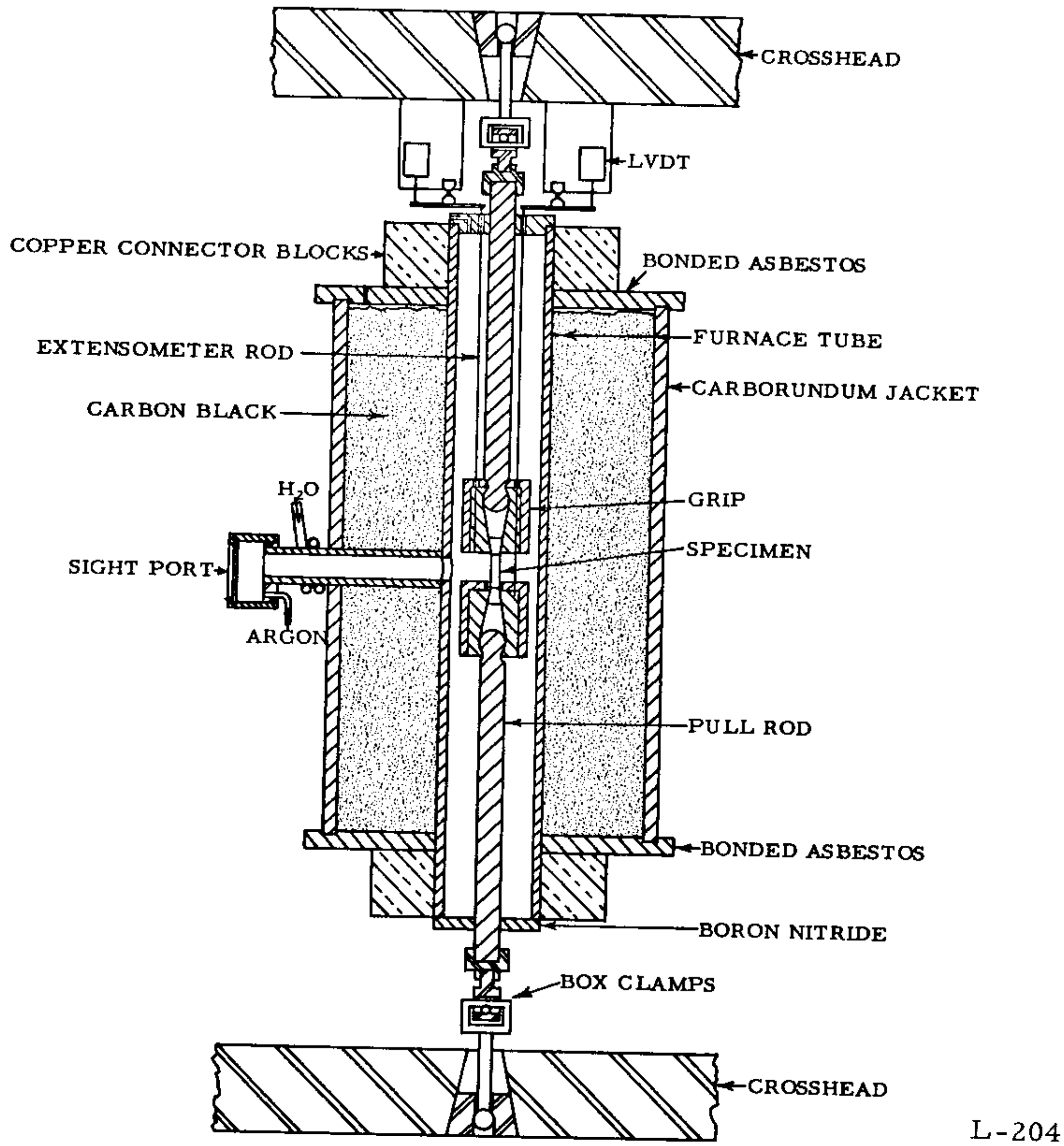

Figure 12. Drawing Showing Entire Tensile Load Train as Mounted in Furnace for High-Temperature Tests

2.3.3. Stress-Strain Measurements

2.3.3.1. Room Temperature

Strain measurements are relatively easy to make at room temperature by the use of a linear voltage differential transformer (LVDT) to follow the movable grip, or by a strain gauge attached to the gauge length of the specimen. Both methods have been used with comparable results.

Stress-strain curves are plotted directly on a recorder. The strain measurement is obtained by connecting the cores of two LVD transformers to the specimen grips through a system of levers. As the specimen is stressed, the resultant elongation activates the levers causing the LVDT cores to move. From the difference in the outputs of the two differential transformers, a signal is generated which rotates the drum of a stress-strain recorder. The stress measurement is obtained by means of a direct mechanical connection between the dial indicator of the test machine and the recording pen arm. The pen is pulled across the face of the revolving drum by the dial indicator, which is activated by the load applied to the specimen. A uniform strain rate is maintained by a strain pacer connected to the stress-strain recorder.

Measurements have been made by attaching SR-4 strain gauges to the gauge length of the specimen through a procedure recommended by the manufacturer of the gauges. The procedure is relatively simple, although care must be taken not to short out the strain gauge to the specimen. The surface to which the gauge is attached must be clean and the gauge must be applied with a uniform firmness.

Signals from the mounted strain gauges are amplified and recorded by means of a multichannel oscillograph which also records a signal from a load cell attached to one end of the pull train. The stress and strain curves are simultaneously recorded on one chart as a function of time; from this chart a stress-strain curve can be drawn. Figure 13 is a photograph of the stress-strain measurement equipment without the load cell.

Figure 13. Photograph of LVDT and Strain Gauge Test Assembly

2.3.3.2. Elevated Temperature

The strain-gauge method is not practical when a large number of tests are to be made and cannot be used at high temperatures. The LVDT method of measuring strain has therefore been adopted for this project.

If the assumption could be made that the strain in the pull rods is negligible, then the strain in the graphite specimen could be measured by using a LVDT to follow the movement of the test machine crossheads. This is not a safe assumption, however, since the strain in the pull rods may be considerable for high temperatures and high loads.

An extensometer method for measuring strain, developed by Martens, et al. [5] was adapted for the high-temperature stress-strain relationship. This method is shown schematically in Figure 14 and described in the discussion below. Graphite rods 3/16 inch in diameter

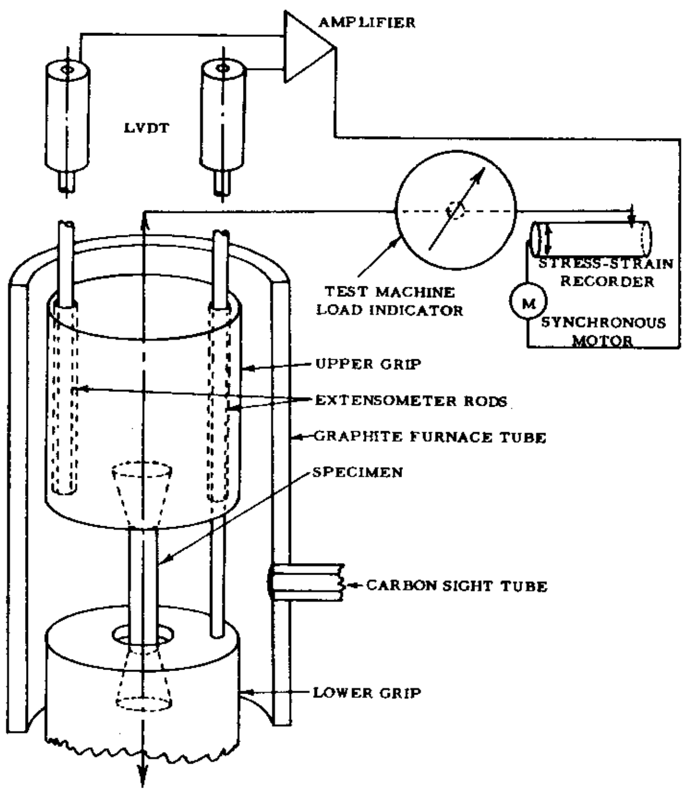

Figure 14. Schematic of Tensile Stress-Strain Extensometer for Elevated Temperature Tests

are mounted parallel to the top pull rod so that the bottom end of one rod is flush with the top of the specimen gauge length (bottom of the top grip) and the bottom end of the other rod is flush with the bottom of the specimen gauge length (top of the bottom grip). The top ends of the rods are directly connected to one end of balanced levers which are in turn connected to cores of LVDT's. The LVDT's are mounted in holders attached to the strain rods of the test machine. As the specimen is mechanically stressed, the extensometer rods move the levers which move the LVDT cores the same distance. A signal proportional to the movement of the rods is generated by the LVDT's as described in Section 2.3.3.1. Figure 15 is a photograph of the high-temperature LVDT extensometer and Figure 16 shows some high-temperature stress-strain curves made by the LVDT method. No reason can be given for the serrated curves shown in Figure 16, but a similar phenomenon has been observed in mild steel at elevated temperatures and in aluminum stressed beyond its initial yield point at room temperature.[6]

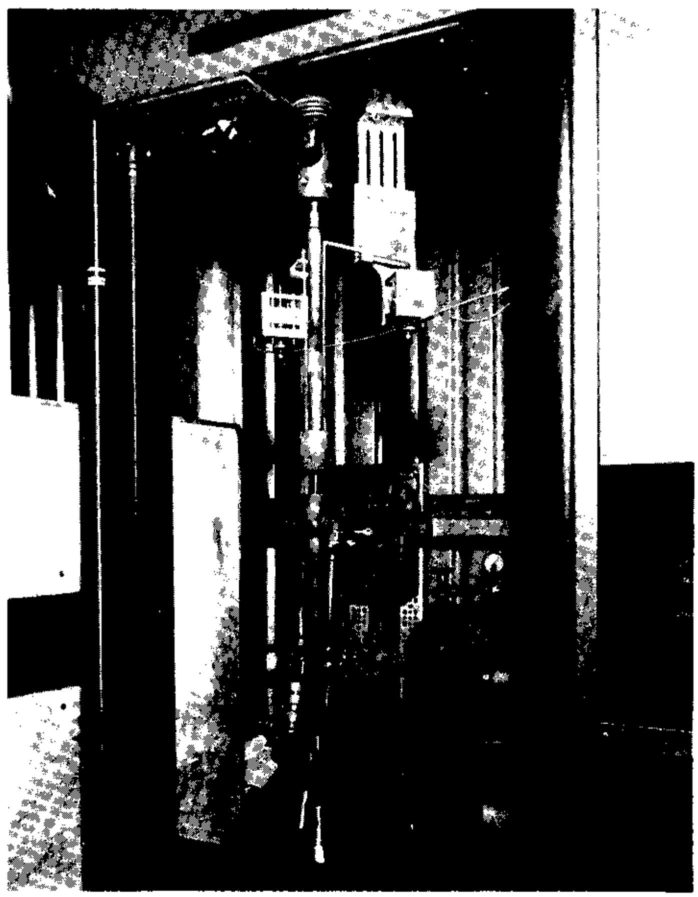

Figure 15. Photograph of Extensometer for Tensile Tests Made at Elevated Temperatures

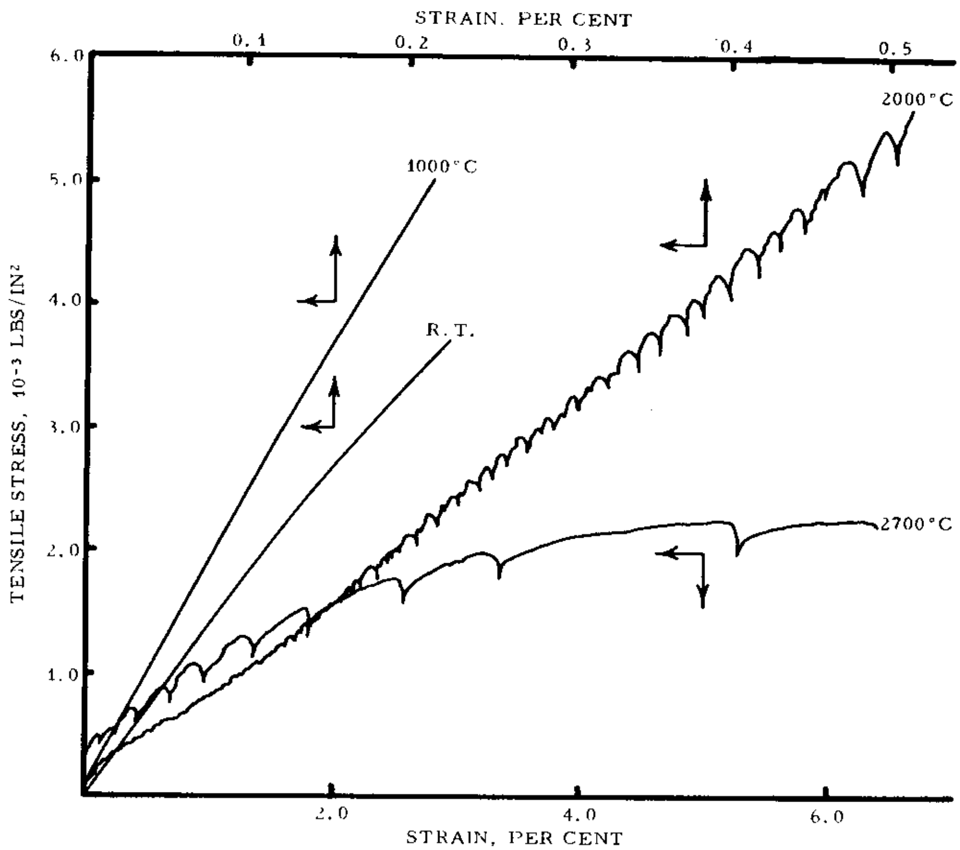

Figure 16. Tensile Stress-Strain Curves Made at Elevated Temperatures with Extensometer (LVDT)

A photographic method for measuring strain, developed by Fisher et al.[7] was investigated. Two reference points attached to the ends of the gauge length were periodically photographed while stress was being applied. The photographic method had four major limitations: (a) It was difficult to adapt to the type of furnace and testing machine used in this project because there was no direct time reference between strain and stress; (b) the contrast between the specimen and furnace wall above 2000°C was not sufficient to obtain a clear picture of the reference points; (c) processing of film and printing of photographs were too time consuming and would necessitate purchase of expensive equipment to reduce and store data; (d) the method would be costly and perhaps impossible to use for measuring lateral strain in two directions on a with-grain sample. The reasons for measuring lateral strains in two directions are discussed in Section 2.3.4.

2.3.3.3. Calibration of Extensometer

A direct means was used for determining the proportionality between drum rotation and movement of the extensometer rods. Dial indicators, graduated to 0.0001 inch over a 0.2000-inch range, were used to determine the linear motion of the rods which caused a recorded amount of drum rotation. Elongation of the load train at elevated temperatures was determined by use of a $^1/_2$-inch diameter rod threaded in a manner such that the grips were butted one against the other.

Strain-gauge test measurements were also used to calibrate the extensometer by measuring stress and strain as explained in Section 2.3.3.1 and comparing the resulting stress-strain curves with those obtained simultaneously on the stress-strain recorder by use of the extensometer method.

Although one LVDT could be used for the calibration tests a more stable signal was obtained with two LVDT's, assuring continuous operation of the stress-strain recorder throughout the test.

2.3.4. Poisson's Ratio

A test for determining Poisson's ratio (lateral strain divided by longitudinal strain) at high temperatures has been designed and equipment is being assembled. The test utilizes very high resolution (0.005 ± 0.00005 inch) LVDT's which will follow the movement of four graphite rods located in a lateral plane 90° apart and butted up against the sides of a tension or compression specimen. Four rods are used because graphite has two lateral strain values in the with-grain orientation such that the circular cross section of a with-grain specimen deforms elliptically. The across-grain specimen deforms in a circular manner. As the specimen is subjected to a uniaxial load applied normally to the plane of the lateral strain-sensing rods, the rods follow the movement of the sides of the specimen. This lateral movement is then measured by the LVDT's and the measurement is recorded on a multichannel oscilloscope. The method is illustrated in Figure 17.

Room-temperature measurements of Poisson's ratio were made by attaching strain gauges to the lateral and longitudinal axes of the tensile specimen and recording the strains with a multichannel oscillograph. The strain was measured in only one lateral direction because the specimens were too small to accommodate the required number of strain gauges. The results of this test are shown in Table 1.

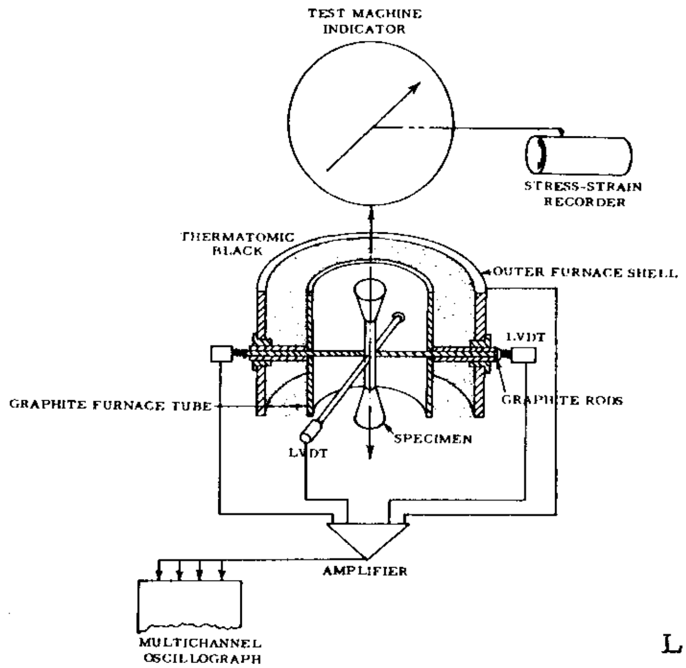

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Figure 17. Schematic Drawing of Apparatus for Measurement of Poisson's Ratio

Table 1. Room-Temperature Strain-Gauge Measurement of Poisson's Ratio

Type Graphite	Grain Orientation	Tensile Stress, lbs/in²	Strain, μ in/in Gauge No. 1*	Strain, μ in/in Gauge No. 2**	Poisson's Ratio ***
Fine Grain Molded	w.g.	3518	3150	520	0.165
Med. Grain Recrystallized	a.g.	1215	2420	229	0.095
Fine Grain	w.g.	4030	1940	223	0.117
Recrystallized	a.g.	2210	1944	884	0.046

* Elongation
** Lateral Strain measured in only one with-grain direction
*** Gauge No. 2/Gauge No. 1

2.4. Performance of Equipment

2.4.1. Sample Alignment

Strain-gauge measurements were made to check the alignment of the load train. Two SR-4 strain gauges were mounted on the gauge length of the specimen as described in Section 2.2. Signals from the gauges were amplified and recorded as described in Section 2.3.3.1. The results of this test are presented in Table 2. The two gauges recorded identical strain at two of the three applied loads. At the lowest stress the variation in strain was approximately ± 1 per cent of the mean. These results show that with reasonable care, stresses other than tensile may be held to a negligible level.

Table 2. Load Train-Alignment Test
Crosshead Rate=0.020 In/Min

Type Graphite	Grain Orientation	Tensile Stress, lbs/in^2	Strain, μ in/in Gauge No. 1	Strain, μ in/in Gauge No. 2	Coefficient of Variation, per cent Gauge No. 1	Coefficient of Variation, per cent Gauge No. 2
Med. Grain Molded	w.g.	1050	375	368	0.94	0.94
		1560	732	732	0	0
		2075	1058	1058	0	0

2.4.2. Type of Fractures

Theoretically, a specimen should fracture in the exact center of its gauge length when subjected to a pure tensile load. Such fracture does not always occur even in ductile materials. It perhaps occurs in lesser degree in brittle materials. Approximately 50 per cent of the specimens fracture within the central 1-inch section of the $1^1/_2$-inch gauge length, as is illustrated in Figure 18. The remaining 50-per cent fracture within $^1/_4$ inch from one of the conical ends. Only when the specimen is not properly machined do the fractures occur in the conical ends.

2.4.3. Coefficient of Variations

In order to determine whether or not variations which occur during the tensile-strength test are due to the test or to the material, the coefficients of variation were calculated from two grades of graphite: (a) A standard fine-grain molded graphite known to have variable properties;

(b) a medium-grain molded graphite developed with the specific purpose of reducing property variation. The coefficients of variation shown in Table 3 indicate that the latter stock shows a greater uniformity. Since all artificially made graphite is heterogeneous to some degree, very low coefficients of variation for tensile tests would be suspect because all properties of graphite in general, and tensile strength in particular, are sensitive to these intrinsic variations.

1 - ZTA, A.G., 2700°C

2 - ZTA, W.G., R.T.

3 - RVA, A.G., R.T.

4 - RVA, W.G., 2700°C

5 - ZTA, A.G., R.T.

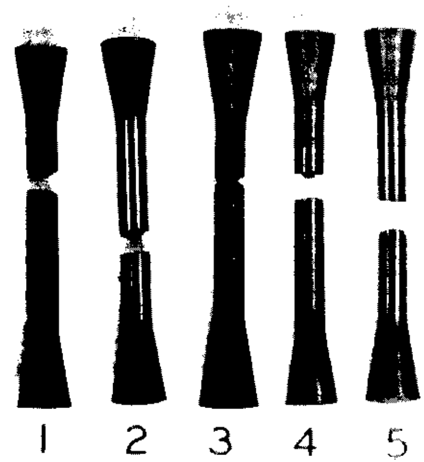

Figure 18. Photograph Showing a Group of Graphite Specimens which Fractured Toward Center of Gauge Length

Table 3. Coefficient of Variation, Ultimate Tensile Strength, Room-Temperature, Crosshead Rate = 0.020 In/Min

Type Graphite	No. Spec.	Grain Orientation	Ave. Ultimate Tensile Strength, lbs/in²	Coefficient of Variation, per cent
Fine Grain Molded	11	w.g.	3353	11.3
9- by 20- by	10	a.g.	2934	4.0
28-Inch blocks				
Med. Grain Molded	10	w.g.	3200	5.3
33-Inch diameter by	9	a.g.	2190	7.4
42-inch length				

A comparison of these coefficients of variation gives evidence that variations in the test results are probably due to the material and not to the test, provided that a reasonable amount of care has been taken to maintain equal conditions; i.e., specimen machining, specimen configuration, etc., throughout the testing program.

2.4.4. Comparison with Other Tensile Tests

Extensive high-temperature tests were conducted on ZTA graphite to determine ultimate tensile strength at temperatures ranging from 20°C to 2700°C. Table 4 gives the results of these tests.

Table 4. Ultimate Tensile Strength Versus Temperature ZTA Graphite 14-Inch Diameter, Crosshead Rate = 0.020 Inch/Min

No. Spec.	Blocks	Temp, °C	Ave. Ult. Str., lbs/in² w.g.	Ave. Ult. Str., lbs/in² a.g.
49	5	20	4405	
61	5	20		1530
10	5	1000	5090	
5	5	1000		1750
7	5	1500	6270	
5	5	1500		1925
10	5	2000	8540	
5	5	2000		2150
9	5	2250	8780	
5	5	2250		2355
12	5	2500	10075	
6	5	2500		2495
6	5	2700	7660	
4	4	2700		2390
1	1	2800		2080
1	1	3000		1765

The ultimate tensile strength of ZTA graphite throughout the same temperature range has been measured also by Southern Research Institute[8] and Aeronutronic[9] Division of Ford Motor Company. The comparison of results for the three series of measurements is shown in Figure 19. All data are based upon specimens with $^1/_4$-inch gauge diameter and a test machine crosshead rate of 0.020 inch/minute. Aeronutronic and Southern Research used specimens with threaded ends rather than conical ends as used by National Carbon Company.

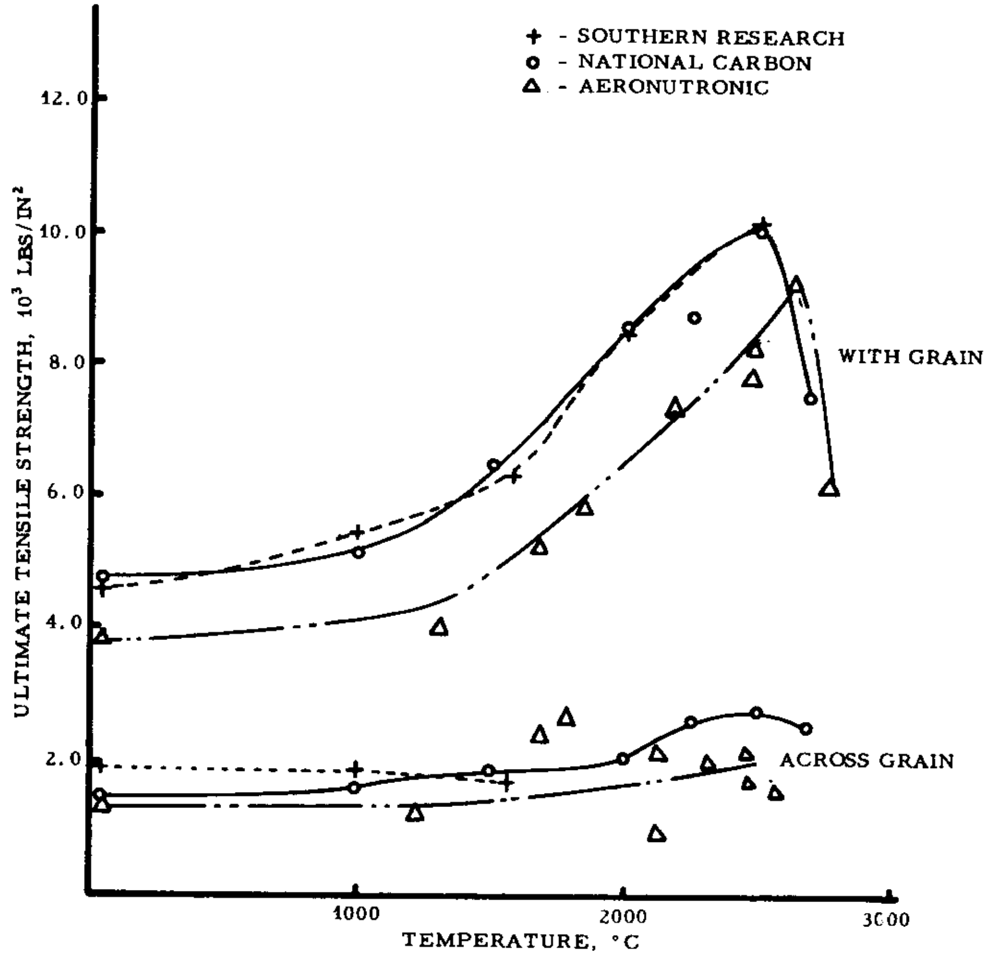

Figure 19. Graph of Ultimate Tensile Strength Versus Temperature, Grade ZTA, Comparison of AML Test with Other Tensile Tests

2.4.5. Pyrolytic Graphite Tests

Two specimens of pyrolytic graphite were tested for ultimate tensile strength at temperatures of 2650°C and 2950°C. A third specimen which was to have been used for room-temperature measurements delaminated while being machined. Insufficient stock remained for another specimen. At 2650°C the specimen failed by brittle fracture; at 2950°C the specimen did not fracture but elongated over 200 per cent.

Figure 20 is a photograph of a pyrolytic specimen in the grips before testing and Figure 21 is a photograph of the specimens before and after tests.

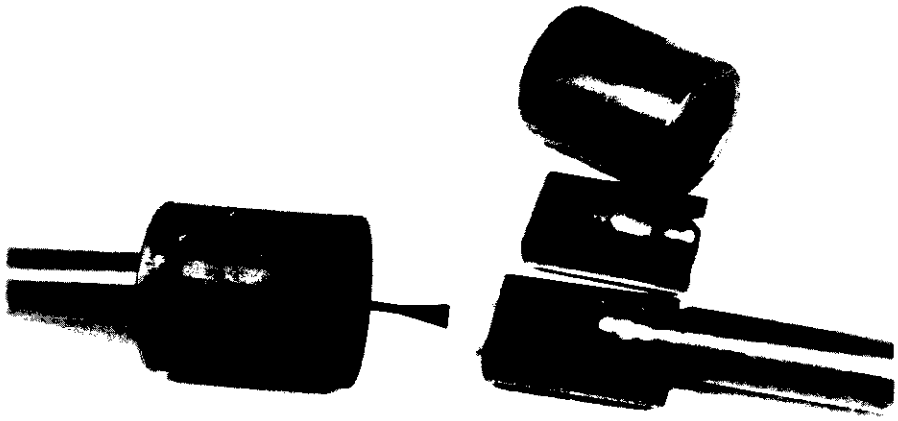

Figure 20. Photograph of Tensile Grips for Pyrolytic Graphite at Elevated Temperatures

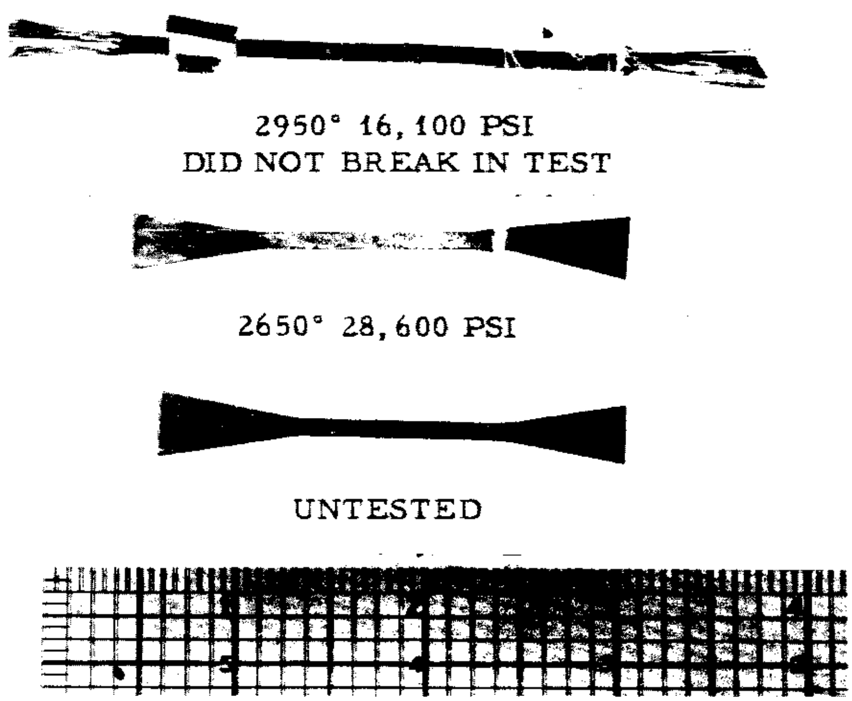

Figure 21. Photograph of Pyrolytic Tensile Specimen

3. COMPRESSIVE STRENGTH MEASUREMENTS

The short-time compressive strength of graphite under a uniaxially applied load can be determined at room and elevated temperatures, along with the resultant strain, by applying principles used to test ductile, homogeneous materials. For both compressive strength and stress-strain measurements, all that needs to be provided is a pair of rams to exert a measured force at a controlled rate.

The ultimate compressive strength is calculated by dividing the fracturing load by the cross-sectional area of the specimen.

3.1. Specimen

A cylindrical sample, $^1/_2$ inch in diameter, is recommended for determining the compressive strength of graphite at elevated temperatures for reasons explained in Section 3.2.2. The length may vary within reason. A $^1/_2$-inch gauge length was selected for this project primarily for ease of handling and for conservation of specimen stock.

The sample for room-temperature measurements may be a cube, a bar with square cross section, or a cylinder. A cylindrical sample may be preferred as it would be consistent with the shape recommended for high-temperature specimen measurements.

3.2. Equipment and Measurements

3.2.1. Room Temperature

Simple steel anvils, as shown in Figure 22, are used for the measurement of compressive strengths at room temperature. The force required to fracture the specimen is applied and measured, along with the resultant strain, by a universal testing machine.

3.2.2. Elevated Temperatures

The same furnace and techniques used for high-temperature tensile strength measurements (Section 2.3.1) are used for compressive measurements at elevated temperatures.

Graphite rams are required for measurements at elevated temperatures. The upper ram is mounted to the anvil with a transite coupling and the lower ram is set on carbon bricks which are placed flat on the test machine platen. Carbon bricks are used because of their relatively low thermal conductivity and because they can be precision ground on all

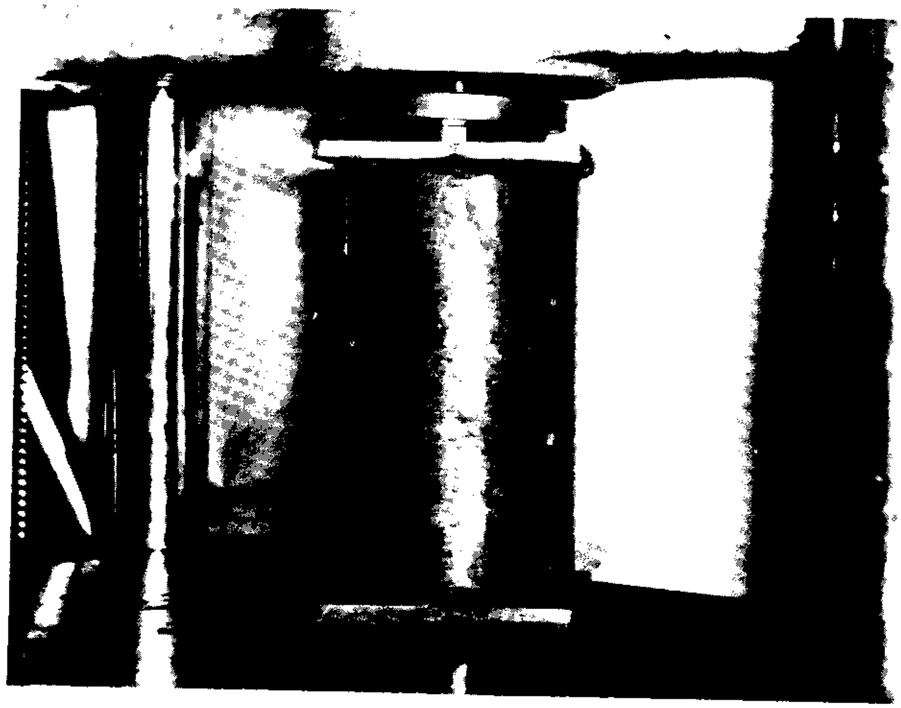

Figure 22. Photograph of Apparatus Used to Determine Room-Temperature Compressive Strength of Graphite

surfaces to make a flat, level base. Another advantage of the bricks is that they prevent the graphite tube furnace from grounding out through the test machine if one of the rams should buckle during a test. The bonded asbestos coupling serves the same purpose for the upper ram.

Initial work on high-temperature compressive strength was done with a $^1/_2$-inch, cube-shaped ATJ specimen. Grade ZTD graphite rams were used to convey the applied load, and a ZTD compression cage was used to maintain alignment of the rams. Figure 23 is a drawing of these parts of the equipment. If ZTA graphite had been available in sufficiently large stock, it would have been chosen as a ram material because of its higher room-temperature compressive strength and its assumed higher compressive strength at elevated temperatures.

The system was tested at 2500°C with the results shown in Figure 24. It can be seen that the compressive strength of ZTD graphite near the upper temperature limit is too low to allow the use of ZTD as ram material. It can be seen also that ZTD graphite undergoes too much deformation, as is evidenced by the imprint of the specimen on the face of the rams. The sharp corners of the specimen caused cracks to propagate in the rams from the corners of the specimen imprint to the outer surface of the rams, as can be seen in Figure 24. A cylindrical-shaped specimen was found to be more compatible with the circular cross section of the rams.

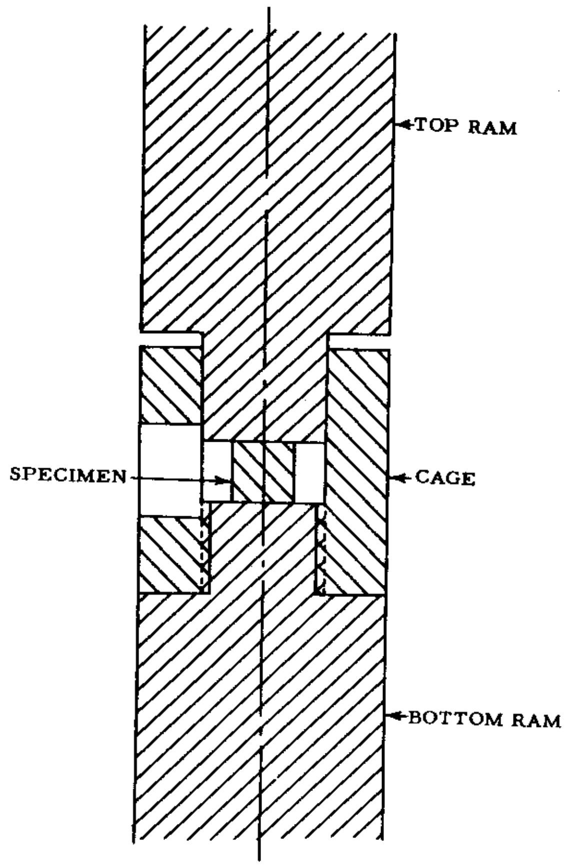

Figure 23. Drawing of Compressive Cage Used in First Compressive Tests at Elevated Temperatures

Figure 24. Photograph of Compressive Rams After First High-Temperature Test

Tests indicated that RVA graphite would function as well as ZTD for ram material. Since RVA is cheaper and is available in much larger sizes, it could be used to advantage for compressive rams. By capping the rams with short pieces of ZTA, the surfaces in contact with the specimen undergo minimum deformation.

3.2.3. Stress-Strain Measurements

Stress-strain curves are made by the extensometer method, as explained in Sections 2.3.3.1 and 2.3.3.2. The high-temperature rams and extensometer rod are shown in Figure 25. Figure 26 is a drawing of the entire compressive strength test apparatus as it is mounted in the vertical tube furnace.

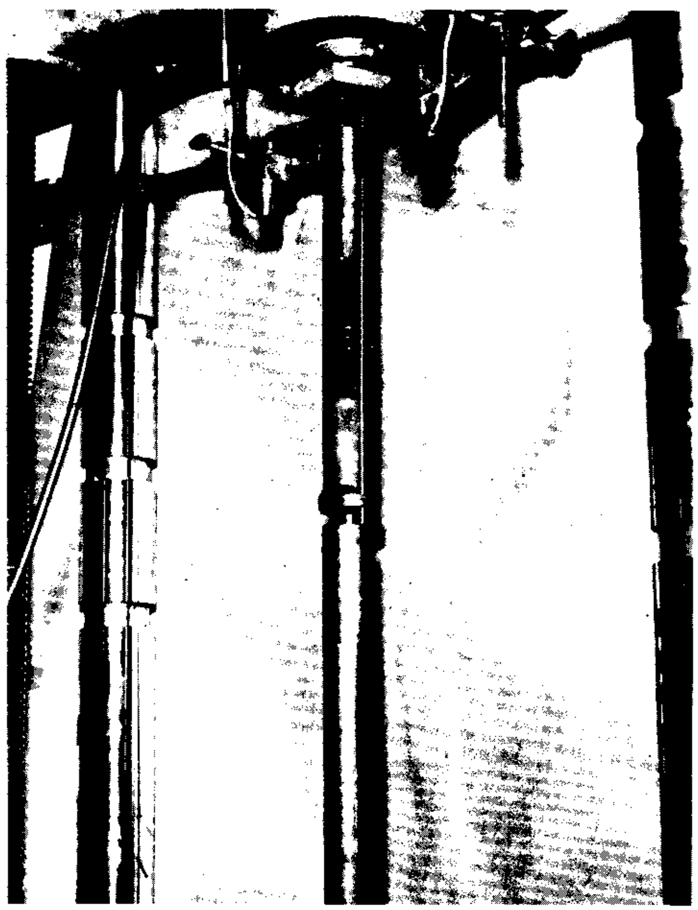

Figure 25. Photograph of High-Temperature Compressive Strength Apparatus Showing Capped Rams and Extensometer Rods

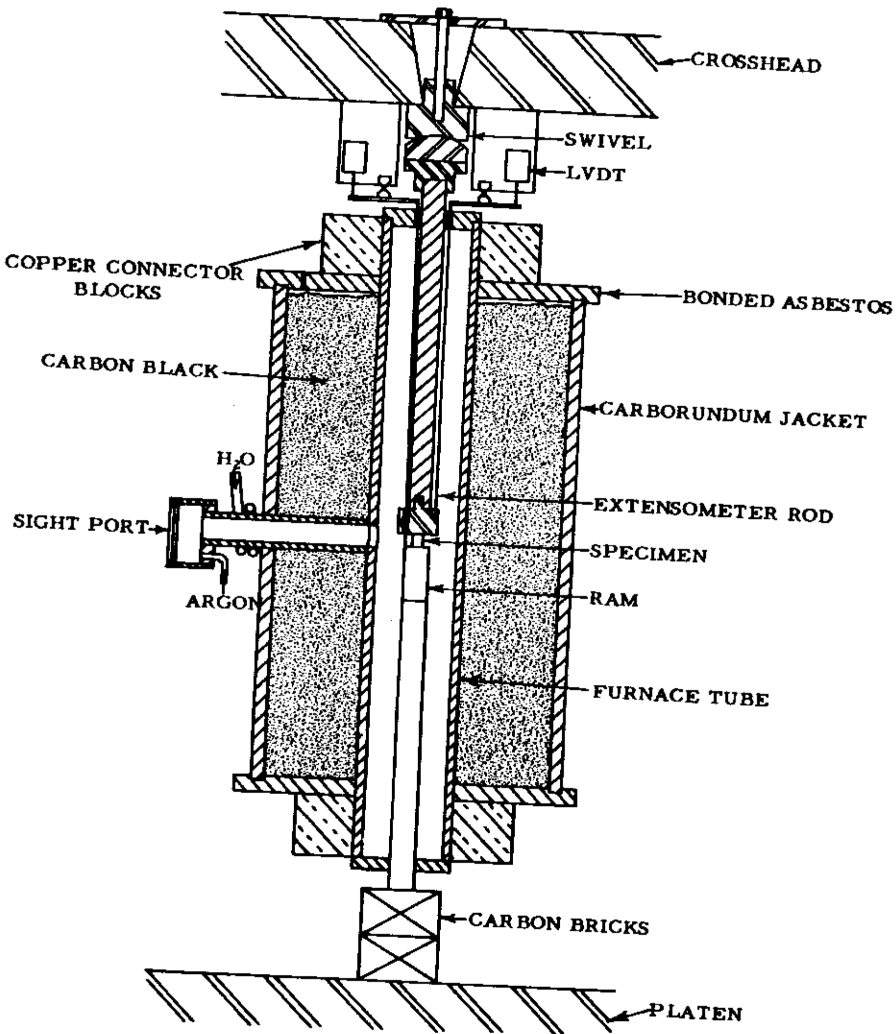

Figure 26. Drawing of Compressive Strength Apparatus as Located in Furnace for Elevated Temperature Test

3.3. Performance of Equipment

3.3.1. Specimen Size and Shape

Tests were made at room temperature to determine the optimum specimen size and shape. The results of these tests are shown in Table 5. Although there is a difference between the average compressive strengths as measured with the cylindrical and with the cubic specimens, neither shape is consistently higher or lower than the other. This seems to indicate that the difference is in the material tested.

Table 5. Effect of Specimen Size and Shape on Compressive Strength

Type Stock	Specimen Shape and Diameter	Grain Orient.	No. Spec.	Ave. Ultimate Compressive Strength, lbs/in²	σ lbs/in²	Coefficient of Variation, per cent
Medium	1-inch cube	a.g.	15	8,585	± 450	5.3
Grain	1-inch cube	w.g.	15	8,375	± 540	6.4
Molded	1/2-inch cube	a.g.	11	8,700	±1045	12.0
No. 1	1/2-inch cube	w.g.	12	8,300	± 855	10.3
Medium	1/2-inch dia.by 1/2-	w.g.	10	10,470	± 488	4.7
Grain	inch cylinders	a.g.	10	10,510	± 593	4.9
Molded	1-inch cube	a.g.	15	7,753	± 855	11.03
No. 2	1-inch cube	w.g.	11	7,430	± 627	8.4
Fine	1/2-inch dia.by 1/2-	a.g.	10	6,660	± 347	5.2
Grain	inch cylinders	w.g.	10	5,650	± 688	12.2
Molded	1-inch cube	a.g.	13	7,375	± 826	11.2
	1-inch cube	w.g.	10	7,005	± 643	9.2

3.3.2. Stress-Strain Curves

Figure 27 shows a typical family of high-temperature compressive stress-strain curves made by the extensometer method.

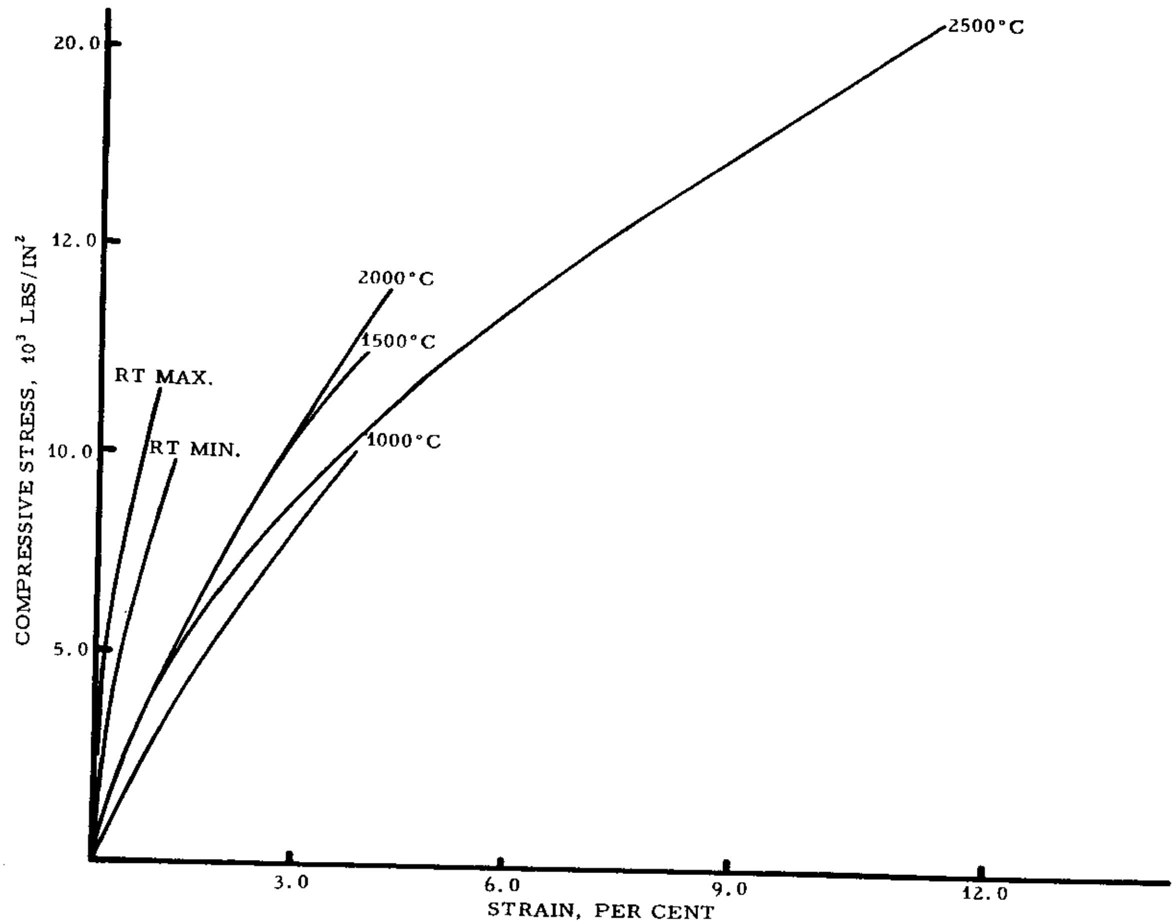

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Figure 27. Compressive Stress-Strain Curves Made with Extensometer in Elevated Temperature Tests

4. APPARENT SHEAR STRENGTH

Pure shear strength of graphite can be measured by twisting thin-walled hollow cylinders.[10] Since this method is time consuming, it could not be used to make the large number of determinations which were necessary for this program. The most practical means to make a number of tests which would give results with reasonable reproducibility was the clevis method in which a load is applied across the diameter of a cylindrical specimen at one or more points.

The load applied in the clevis tests probably is not a pure shearing force, because in these tests the initial fracture nearly always occurred in the geometrical center of the specimen. This fracture is a bending fracture and is disregarded for the purposes of determining the shear strength of graphite by this method. Because of the bending fracture the strength measured by the clevis method is called the apparent shear strength. In the test adopted for determining the apparent shear strength of graphite, a force was applied to the specimen at two points until the test machine indicator returned to the zero position, thereby indicating that the specimen had failed at the points where the load was applied. The shear strength of the specimen was calculated by dividing the applied load by twice the cross-sectional area of the specimen.

4.1. Specimen

The specimen used for the apparent shear strength test by the clevis method is a 0.250-inch diameter by 1-inch length cylinder. Blocks for with-grain specimens are marked with a line running parallel with the top of the block from which the specimen is cut. When the specimen is tested, the shearing load is applied perpendicular to this line as shown in Figure 28.

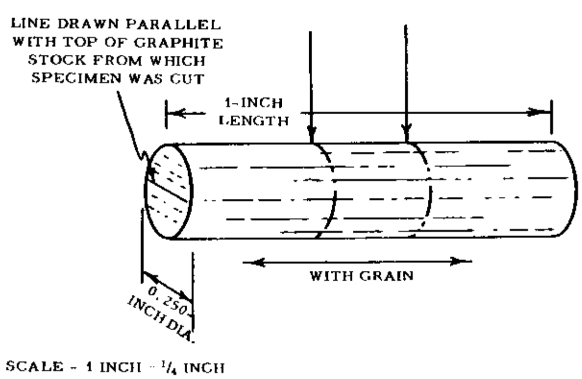

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Figure 28. Illustration Showing Relationship Between Grain Orientation and Shearing Load in a With-Grain Shear Specimen.

4.2. Equipment and Measurements

4.2.1. Furnaces

The same furnace and techniques used for high-temperature tensile and compressive measurements may be used for shear (see Section 2.3.1).

4.2.2. Clevis Fixture

The first clevis fixture evaluated, shown in Figure 29, sheared the specimen at one point and did not hold the specimen rigidly enough to prevent bending of the specimen. Because of this bending, a double-point shear clevis was adopted.

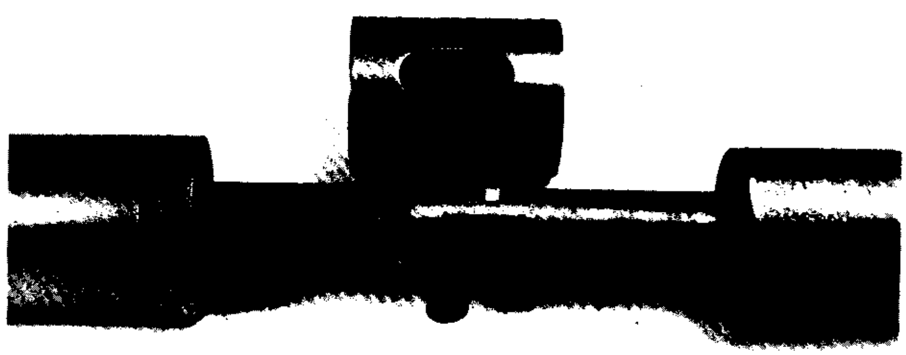

Figure 29. Photograph of First Clevis Fixture Used to Determine Apparent Shear Strength of Graphite at Elevated Temperatures

The fixtures for room- and high-temperature measurements are identical except for the materials from which they are constructed. Steel parts are used at room temperature and ZTA graphite parts are used for tests at elevated temperatures. The room-temperature fixture is shown in Figure 30. Blades in both fixtures are removable so that they may be replaced easily as they become distorted. After each test at 2500°C and above, the graphite blade must be replaced. The cylinder in Figure 30 is a sleeve which slips over the clevis, with specimen and blade in place, to maintain alignment of parts during the test. In room-temperature tests, the load is applied directly to the ends of the fixture by the crosshead and platen of the testing machine. At elevated temperatures the load is applied through RVA graphite compressive rams. Figure 31 shows the fixture mounted upon one of the graphite rams.

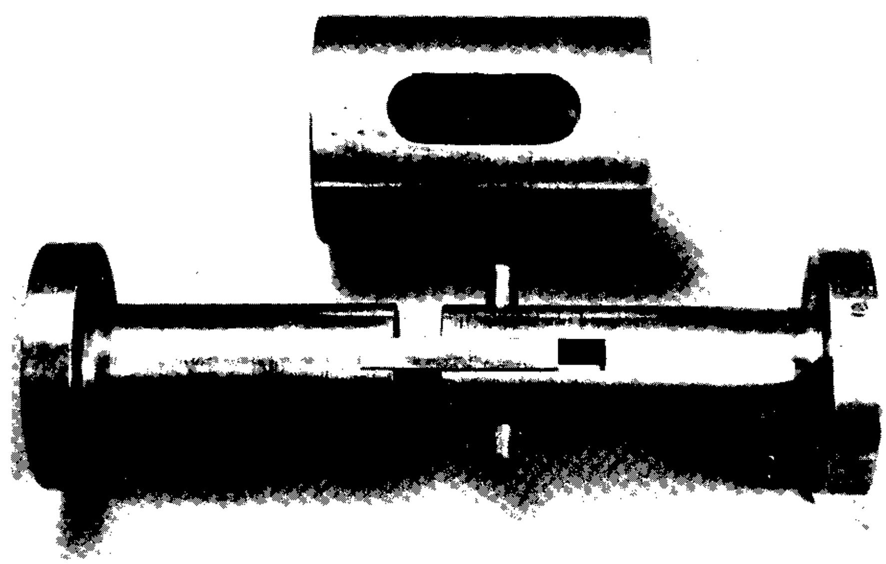

Figure 30. Photograph of Apparent Shear Strength Test Apparatus Used for Room-Temperature Determinations

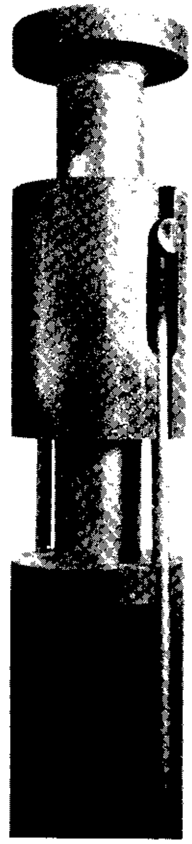

Figure 31. Photograph of Apparent Shear Strength Test Apparatus Used for High-Temperature Determinations

4.2.3. Stress-Strain Measurements

4.2.3.1. Room Temperature

When the shearing load is applied to a specimen of circular cross section, the cross section is distorted in direct proportion to the load. Figure 32 is an illustration of this distortion or strain in shear. Stress-Strain measurements made by use of the clevis apparatus are somewhat doubtful for reasons explained in Section 4.3.2. Room-temperature shear stress-strain curves are made by following the differential movement of the cross-head and platen with LVDT followers in the same manner as for tension and compression test explained in Section 2.3.3.1.

4.2.3.2. High Temperature

Stress-strain curves are made at elevated temperatures in the same manner as for room-temperature measurements described in Section

2.3.3.2. One extensometer rod is attached to the blade and the other to the yoke as illustrated in Figure 33.

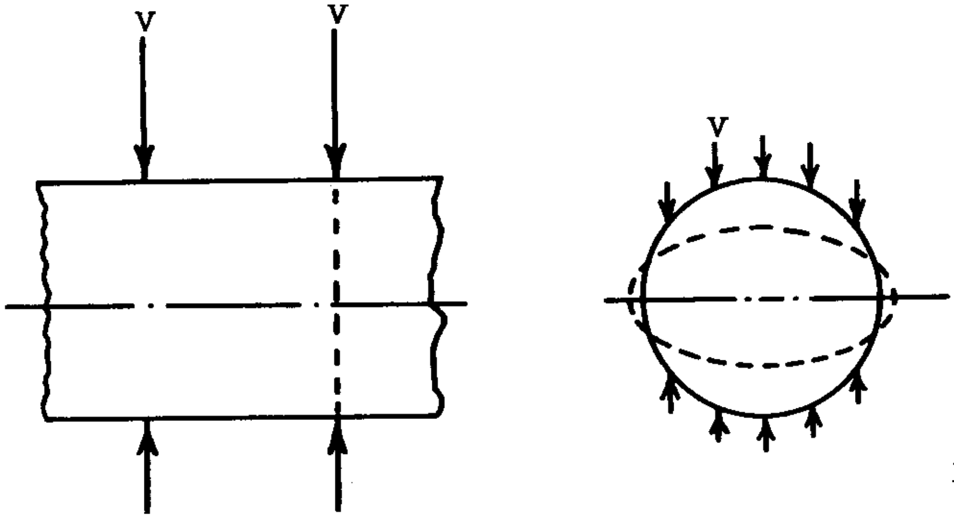

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Figure 32. Shear Specimen and Resultant Deformation in the Specimen Cross Section

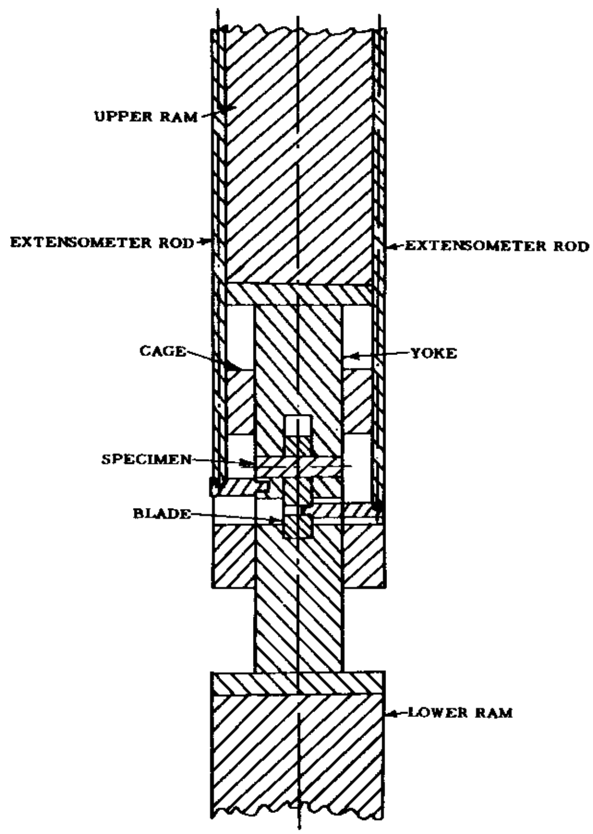

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Figure 33. Illustration of High-Temperature Shear Extensometer

4.3. Performance of Equipment

4.3.1. Apparent Shear Strength

Apparent shear strengths of a typical grade at two different loading rates (0.005 and 0.020 inch/minute) are given in Table 6 and the with-grain results are presented graphically in Figure 34. Above 2000°C, the shear strength of graphite is sensitive to load rate. In this temperature region the graphite specimen is changing from a brittle to a plastic state and part of the applied load is expended in deforming the sample (see Figure 35). At the higher load rate there is less creep or deformation before fracture.

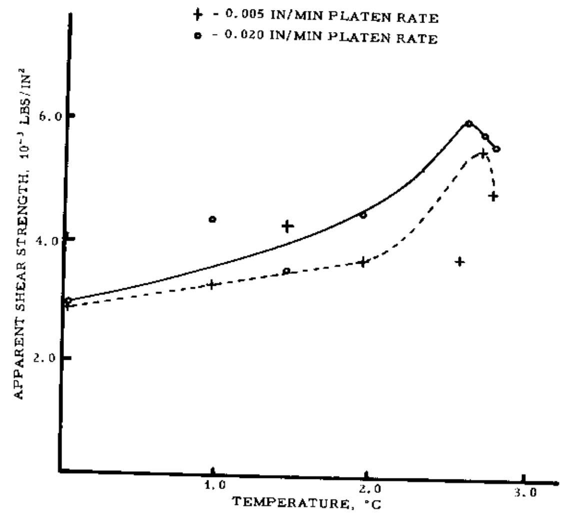

Figure 34. Apparent Shear Strength Versus Temperature, With Grain, Large-Grain Recrystallized Graphite

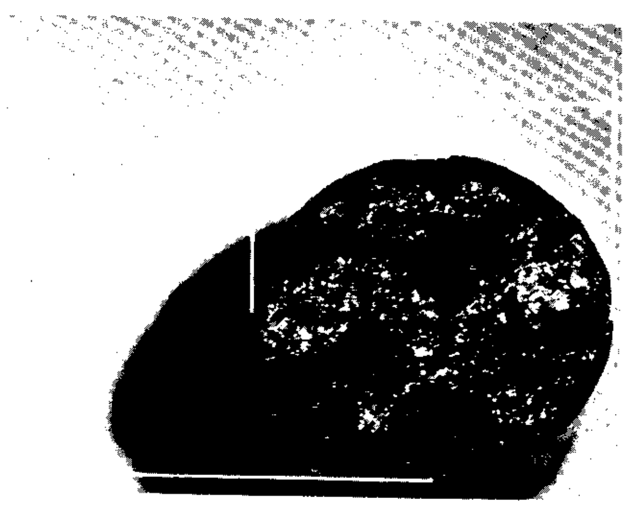

Figure 35. Photograph of Cross Section of Shear Specimen Plastically Deformed at 2500°C

4.3.2. Stress-Strain Curves

Figures 36 and 37 are stress-strain curves for graphite in shear obtained by the clevis method, at platen rates of 0.005 and 0.020 inch/minute, respectively. The points at which the bending fractures occur in these tests are indicated by the horizontal lines in Figures 36 and 37. The curves are rather peculiar, particularly at the higher temperatures and higher load rate. More work on stress-strain is required to determine whether or not strain measurement can be made with a reasonable degree of certainty by the clevis method. The extensometer rods cannot be attached to the clevis at points near enough to the sample to prevent measuring some of the strain in the fixture blade or yoke. This problem is not present in the room temperature measurements because the strain in the steel apparatus should be negligible at the very small loads involved. This may not be true, however, for graphite under high loads at high temperatures. Strain-gauge measurements will have to be made on the graphite fixture at room temperature in order to deter- the magnitude of the strain in the holders and load train.

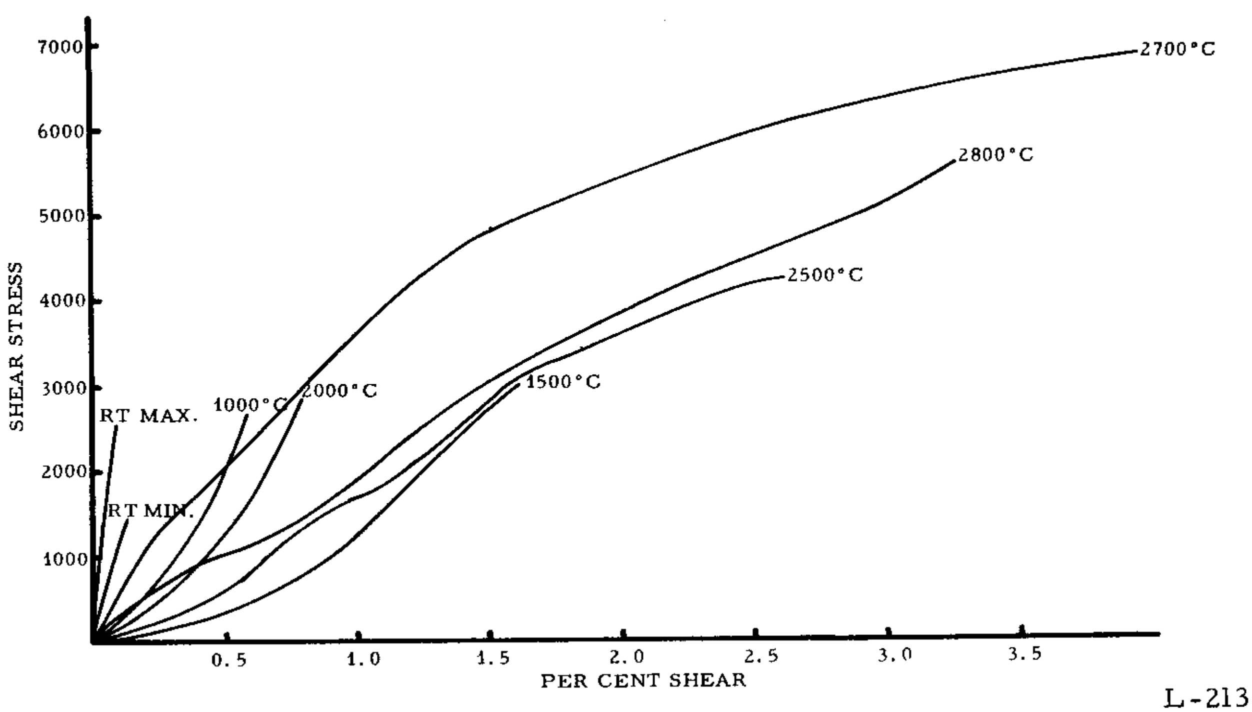

Figure 36. Apparent Shear Stress-Strain Curves Made at 0.005 In/Min Platen Rate

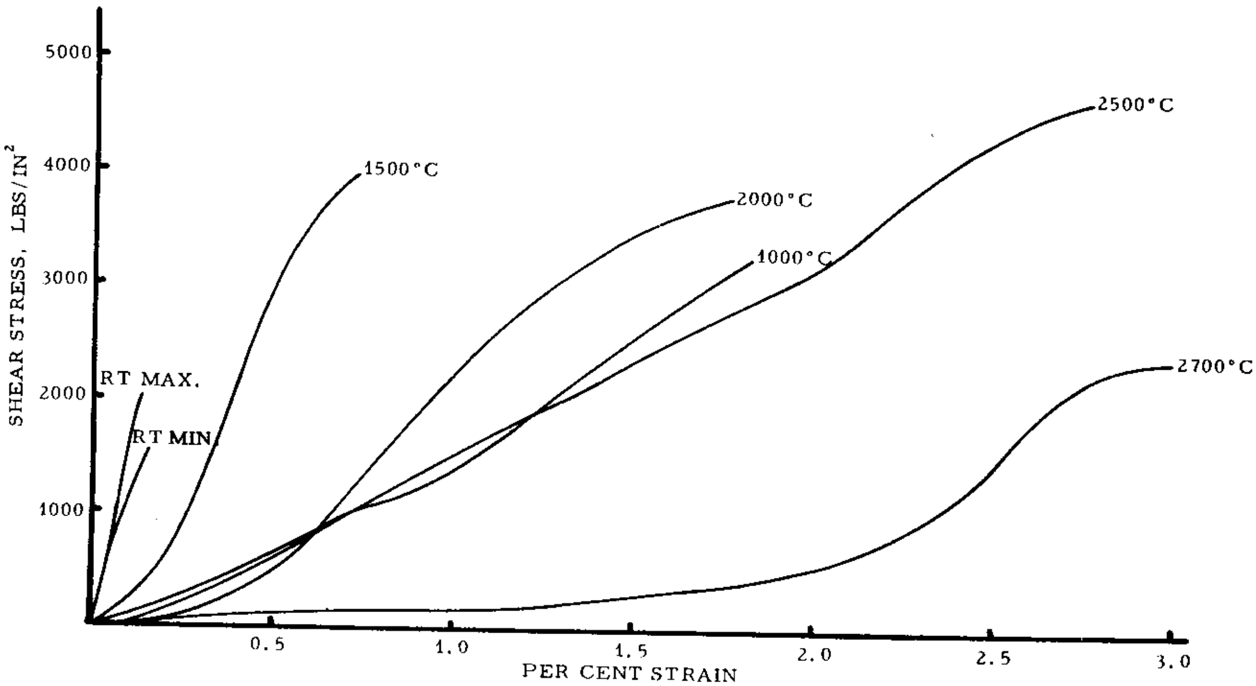

Figure 37. Apparent Shear Stress-Strain Curves Made at 0.020 In/Min Platen Rate L-214

Table 6. Comparison of High-Temperature Apparent Shear Strengths of Two Different Strain Rates

Temperature, °C	Platen Rate, in/minute	Ultimate Strength in Shear, lbs/in², w.g.	a.g.
R.T.	0.005	2775	1760
1000	0.005	3190	2330
1500	0.005	4155	3050
2000	0.005	4545	3940
2500	0.005	5890	4320
2700	0.005	8045	6980
2800	0.005	4515	4600
R.T.	0.020	2880	2160
1000	0.020	4265	1625
1500	0.020	3425	2205
2000	0.020	4400	3450
2500	0.020	5980	3495
2700	0.020	6025	5480
2800	0.020	5780	4600

5. FLEXURAL STRENGTH MEASUREMENTS

Flexural strength (modulus of rupture) is the maximum fiber stress which a material will withstand before rupture in bending.

5.1. Specimens

Two sizes of specimens are used in routine room-temperature tests; both are 5-inch-long prismatic bars but one has a $^1/_2$-inch and the other a $1^1/_4$-inch-square cross section. The specimens are easily formed to precise dimension by finish grinding. In order to obtain enough samples to produce a more significant strength profile, the $^1/_2$- by $^1/_2$- by 5-inch specimen is used when the stock to be tested is 14 inches in diameter or smaller, and the $1^1/_4$- by $1^1/_4$- by 5-inch specimen is used for all sizes of stock over 14 inches in diameter. The use of specimens smaller than those mentioned above results in unduly higher flexural strength values.

5.2. Grain Orientation

The fiber strength measurements have been made routinely for quality control of brittle materials for many years. The peculiar crystalline structure of graphite presents a difficulty to the measurement of its flexural strength. Graphite has an ordered structure of hexagonal crystals which are layered to produce a grain in the formed mass. When specimens are cut from the graphite stock, care must be taken to note the orientation of the grain in the specimen.

Flexural specimens cut from molded graphite have three grain orientations – two with grain (Figure 38A and B) and one across grain (Figure 38C). With-grain measurements are made with the grain orientations shown in Figure 38A.

5.3. Equipment

5.3.1. Room Temperature

Figure 39 is a photograph of the apparatus used for room-temperature flexural strength measurement. The method employed routinely is the "third-point-loading" technique adopted by ASTM as a standard for concrete.[11] The method is so named because the distance between the two points of the upper span is one-third the distance between the lower supports. In addition, the upper span is located such that the horizontal distance between each pressure point and the lower support nearest it is one-third the lower span length. This third-point-loading technique is illustrated in Figure 38A. The advantage of this method is that the uniform loading prevents the specimen from breaking anywhere but at the point of maximum stress.[4]

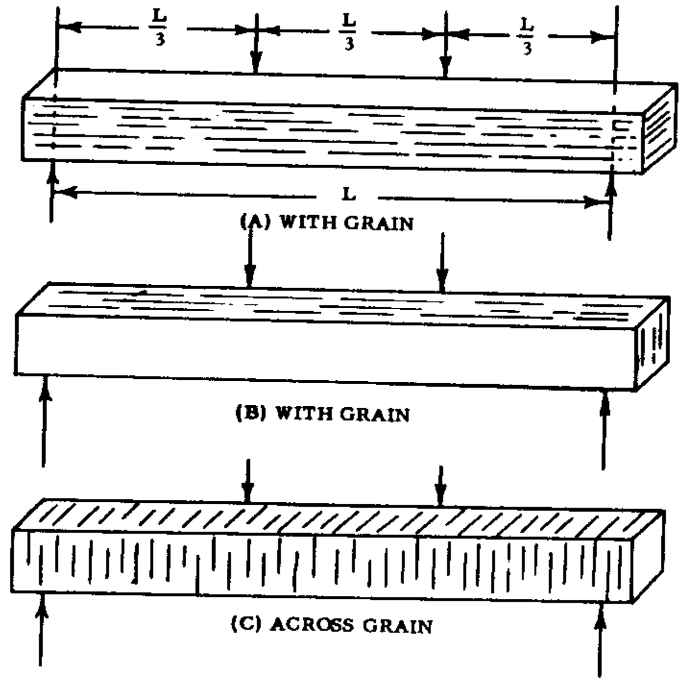

Figure 38. Illustration of Three Grain Orientation and Load Points of Flexural Strength Specimen

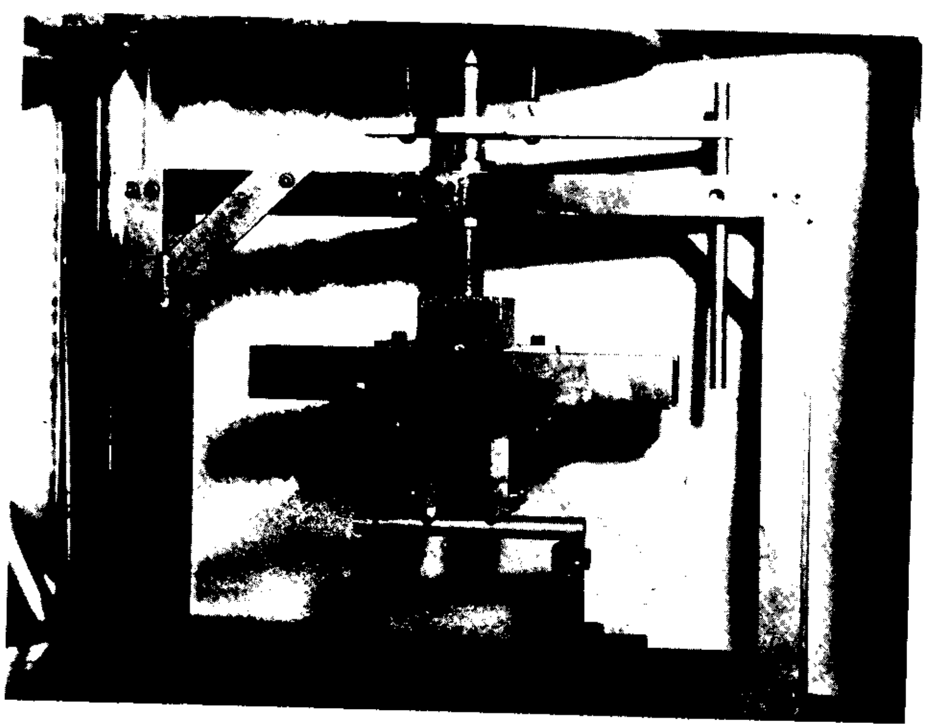

Figure 39. Photograph of Room-Temperature Flexural Strength Apparatus

Flexural strength (modulus of rupture, MR) in lbs/in^2 is calculated by means of the following equation:

$$MR = \frac{3W(L_1 - L_2)}{2BD^2} \quad (1)$$

where

W = Load in pounds,
L_1 = Length of lower span in inches,
L_2 = Length of upper span in inches,
D = Thickness of sample in inches,
B = Width of sample in inches.

5.3.2. High Temperature

Figure 40 is a photograph of the equipment for measuring high temperature flexural strengths, including the furnace, mounted in the test machine. The apparatus, with the exception of the furnace, is identical to the room-temperature apparatus. All parts, except those of the load train which extend into the furnace, are made of lampblack-base carbon stock. Those parts which extend into the furnace are made of ZTA graphite. Figure 41 is a schematic drawing of the high-temperature equipment for measuring modulus of rupture.

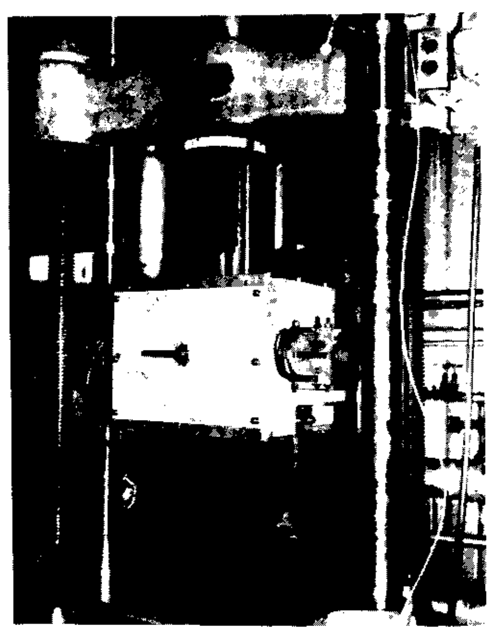

Figure 40. Photograph of High-Temperature Flexural Strength Apparatus Mounted in Testing Machine

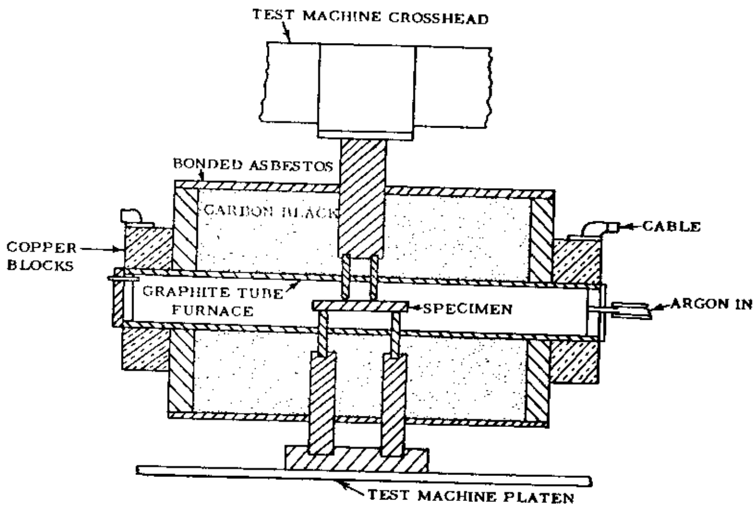

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Figure 41. Schematic of Apparatus for Measuring Flexural Strength at Elevated Temperatures

5.4. Stress-Strain Measurements

Because the location of the neutral axis in the beam is subject to question, the measurement of beam deflection (strain) in flexure is of doubtful value since the elastic theory is based upon the supposition that the neutral axis is in the horizontal center plane of the specimen. No flexural strain measurements are contemplated, therefore, other than those made at room temperature with strain gauges.

5.5. Performance of Equipment

The room-temperature apparatus shown in Figure 39 is a type which has been used by National Carbon Company for many years to make flexural measurements for quality control.

The high-temperature apparatus has been proved in room-temperature tests but as yet it has not been operated at elevated temperatures. Room-temperature data obtained with this high-temperature apparatus are in good agreement with data obtained from the room-temperature apparatus shown in Figure 38.

6. CONCLUSION

Equipment has been developed for measuring ultimate tensile, compressive and shear strengths, and for obtaining stress-strain curves, in the 20° to 2700°C temperature range. The development of high temperature flexural strength equipment is at the stage where the load train is ready for prove-in trials.

The load train in high temperature tensile strength equipment must be constructed from a high tensile strength graphite, such as ZTA, to minimize distortion of the train at temperatures above 2000°C. It is advisable to construct the entire load train from a single block of graphite in order to lessen the degree of mismatch in thermal expansion.

The elimination of all but tensile stresses in the specimen is a primary objective in designing a load train and specimen. The surface condition of the specimen is of equal importance as it also has an effect upon the magnitude of the ultimate tensile strength. A special carbide tool has been developed for machining specimens without tool marks and machining pits. All specimens break within the gauge length when the cutting edge of the tool is kept in good condition.

The rams for the load train of the high temperature compressive strength apparatus may be constructed of any medium or high compressive strength graphite. If a graphite of medium strength is used, RVA for example, the ends should be capped with a high compressive strength graphite (ZTA) to increase the life of the ram by preserving the surfaces which contact the specimen.

Compressive specimens may be in the form of cylinders, cubes or rectangular prisms. Stress concentrations at the corners of cubes and rectangular prisms facilitate local distortion of both the specimen and the ram surfaces at temperatures exceeding 2000°C, consequently cylinderical specimens are recommended for high temperature measurements.

A high strength graphite (ZTA) clevis apparatus, with removable blades to facilitate repair when distortion occurs, has been developed for measurement of ultimate shear strength at elevated temperatures. Shear and tensile stresses resulting from a bending moment cause one or more fractures in the central portion of the specimen before it shears at the ends of the gauge length. The fractures in the central portion of the gauge length relieve the stresses induced by the bending moment, consequently the ultimate stress is a shearing stress.

Strain has been measured in tension and compression by means of linear variable differential transformers, with values comparable to strain gauge measurements at room temperature. Serrated stress-strain curves at 2000°C and higher temperatures were observed in tension, but not in compression. No attempt has been made to explain the serrations. Stress-strain curves in shear are of little engineering value because of the fractures resulting from the bending moment. They are helpful, however in interpreting what has occurred during a shear test by the clevis method.

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