Contrails

FOREWORD

This work was conducted primarily by the Aeronutronic Division of Ford Motor Company at Newport Beach, California under subcontract from the National Carbon Company, a Division of Union Carbide Corporation, under USAF Contract AF 33(616)-6915. This contract was initiated under Project No. 7350 "Refractory Inorganic Nonmetallic Materials", Task No. 735002 "Graphite Materials Development"; Project No. 7381 "Materials Application", Task No. 738102 "Materials Preproduction Process Development"; and Project No. 7-817 "Process Development for Graphite Materials". The work was administered under the direction of the Directorate of Materials and Processes, Deputy Commander/Technology, Aeronautical Systems Division, with Captain R. H. Wilson, L. J. Conlon and W. P. Conrardy acting as Project Engineers.

The work covered in this report was performed between November 1960, and September 1962. Materials and test specimens were supplied by the Research Laboratory of National Carbon Company, Parma 30, Ohio, which also performed some examination of specimens. This is the complete and final report on this Subcontract activity.

Other reports issued under USAF Contract AF 33(616)-6915 have included:

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 and R. N. Draper.
Volume VI	Creep of Carbons and Graphites in Flexure at High Temperatures, by E. J. Seldin.
Volume VII	High Density Recrystallized Graphite by Hot Forming, by E. A. Neel, A. A. Kellar, and K. J. Zeitsch.
Volume VIII	Electron Spin Resonance in Polycrystalline Graphite, by L. S. Singer and G. Wagoner.
Volume IX	Fabrication and Properties of Carbonized Cloth Composites, by W. C. Beasley and E. L. Piper.

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Contrails

Volume X	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Volume XI	Characterization of Binders Used in the Fabrication of Graphite Bodies, by E. de Ruiter, A. Halleux, 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.
Volume XIII	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and E. L. Piper.

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ABSTRACT

This report describes the short time tensile properties of ZTA grade artificial graphite. These tensile properties were measured under the conditions of (a) various annealings, (b) various load rates, (c) various atmospheres, and (d) argon pressure from 5 to 150 psig at temperatures ranging to 2760°C (5000°F). The tensile strength was found to be dependent upon grain orientation, load rate, prior degassing treatment, and on surrounding gas pressure.

This report has been reviewed and is approved.

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Chief, Ceramics and Graphite Branch
Metals and Ceramics Division
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Contrails

TABLE OF CONTENTS

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Contrails

LIST OF FIGURES

FIGURE PAGE

Approved for Public Release

Contrails

LIST OF FIGURES CONT'D.

Approved for Public Release

Contrails

LIST OF FIGURES CONT'D.

FIGURE PAGE

Approved for Public Release

Contrails

LIST OF TABLES

Approved for Public Release

Contrails

LIST OF TABLE CONT'D.

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Contrails

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Contrails

1. INTRODUCTION

A correct knowledge of the mechanical properties of manufactured graphite is of importance to its rational application as a structural material. Pertinent properties include the ultimate tensile, flexural and compressive strengths as well as the corresponding elastic moduli and plastic creep parameters. These have been studied extensively at elevated temperatures as well as at room temperature.(1, 2, 3) Well known are the increases in strength and elastic modulus with increasing temperature up to 2000-2500° C, above which plastic flow becomes significant. Diefendorf(4) has reported various atmospheric effects on the strength of graphite.

The recent application of "hot working" to the fabrication of graphite has provided a new material (the so-called ZT graphite) with unique properties which have been described.(5, 6) The high density and the high degree of anisotropy which set this material apart, made an examination of its strength behavior highly desirable. Accordingly, ZTA grade graphite has been studied in tension under varied conditions for approximately two years by Aeronutronic Division, Ford Motor Company. The results of this study are summarized in this report.

A brief history of the manufacture of the ZT-type graphites, with the attendant changes that transpire at several critical steps, is desirable to understand more fully the meaning of the test results.

Chronologically, sized calcined coke particles are combined with coal tar pitch and formed to a desirable shape. This mixture is then coked to convert the pitch to an infusible solid bond. At this point, a repeated pitch impregnation and coking may take place to fill voids left by volume changes that occur on coking. Following the final coking, the material is converted from carbon to graphite by an in situ transformation that occurs on heating to 2600 - 3000° C. Up to this point, manufactured graphites and ZT graphites follow essentially this process.

While manufactured graphites are finalized at this point, the ZT graphite undergoes an additional step.(5) The graphitized stock is heated to a temperature at which it becomes plastic and then compressed by applying mechanical pressure. This "hot working" increases the density of the stock and the order of the graphite crystallites in a controllable manner to yield ZT series graphites.

Specific members of the ZT series graphite use well-defined starting materials controlled carefully throughout the entire processing. ZTA in particular is produced by the hot working of a fine grain, prefabricated commercial graphite of carefully controlled manufacture.

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2. SUMMARY

The tensile testing program conducted on National Carbon Company grade ZTA "recrystallized" graphite has defined the following:

(1) The material is moderately anisotropic in strength properties; the normal to mold (with-grain) direction is about three times that of the mold (across-grain) direction strength.

(2) The strength of the graphite increases from about 4000 psi at room temperature to over 9000 psi near 2500°C (4500°F) for the with-grain oriented material.

(3) The corresponding across-grain material over the same temperature range increases in strength from about 1500 psi to 2000 psi.

(4) Above 2550°C (4600°F), the strength of ZTA grade graphite decreases in all cases.

(5) The rate of change of stress with strain increases from a relatively low value at low strain to higher values at higher stress.

(6) No difference was found in the strength of ZTA graphite which had been annealed at 3000°C prior to testing and its unannealed counterpart.

(7) The ultimate tensile strength of the ZTA graphite is load rate sensitive, with the slower rates resulting in the higher strength above 1650°C (3000°F).

(8) The ultimate tensile strength is insensitive to a test atmosphere of nitrogen or carbon monoxide at all temperatures.

(9) The strength of the across-grain oriented material decreases with increasing density within the density range examined.

(10) Over the same density range, the strength of the with-grain oriented material is not significantly affected.

(11) Between 1980°C (3600°F) and 2500°C (4500°F) the strength of the graphite is sensitive to external pressure, with an increase in pressure resulting in an increase in strength.

(12) The removal of contained gases by heating in a vacuum increases the strength of the material below the outgassing temperature.

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3. EXPERIMENTAL PROCEDURE

The tensile stress properties of a new grade of artificial graphite under development by National Carbon Company were measured using the test facilities of the Aeronutronic Division of Ford Motor Company. While the acquisition of the special testing equipment required to perform the work did not constitute a part of the contract, it is desirable that a description of this special equipment be included for completeness and an understanding of the means whereby the data were obtained.

3.1. Furnace

The furnace used for the tensile strength measurements of the ZTA grade graphite is diagrammed in schematic cross section in Figure 1. This furnace was designed and constructed to provide a short duration, high temperature tensile or compressional testing facility. The criteria of design considered necessary to achieve this goal may be summarized as follows:

(1) The specimen must be heated by radiation.

(2) Heating and cooling cycles must be as rapid as possible.

(3) The maximum operating temperature must be as high as possible.

(4) Internal operating pressures of controlled gas in the range 10^{-2} to 800 centimeters of mercury must be realizable during operations.

(5) The test temperature of the specimen must be constant over the gauge length and remain constant for periods up to one hour, if desired.

(6) View ports suitable for optical strain measurements must be present.

(7) Overall dimensions must be small enough to permit the furnace to operate in a wide frame Instron Testing Machine.

(8) Graphite heating elements must be replaceable with a minimum time consumption.

The furnace has proven to operate satisfactorily in keeping with an effective compromise of the design parameters itemized above. It is electrically heated, using the self-resistance of six hairpin shaped elements in a series-parallel circuit. The hairpin shape of these graphite elements provides the latitude necessary for matching the power supply and the compensation for thermal expansion by the expedience of slight variations in the cross sectional area while holding both legs uniform in area and length. Radiated heat from the elements over a length sufficient to traverse the specimen and gripper results in a uniform temperature test section. Insulation from this heat in

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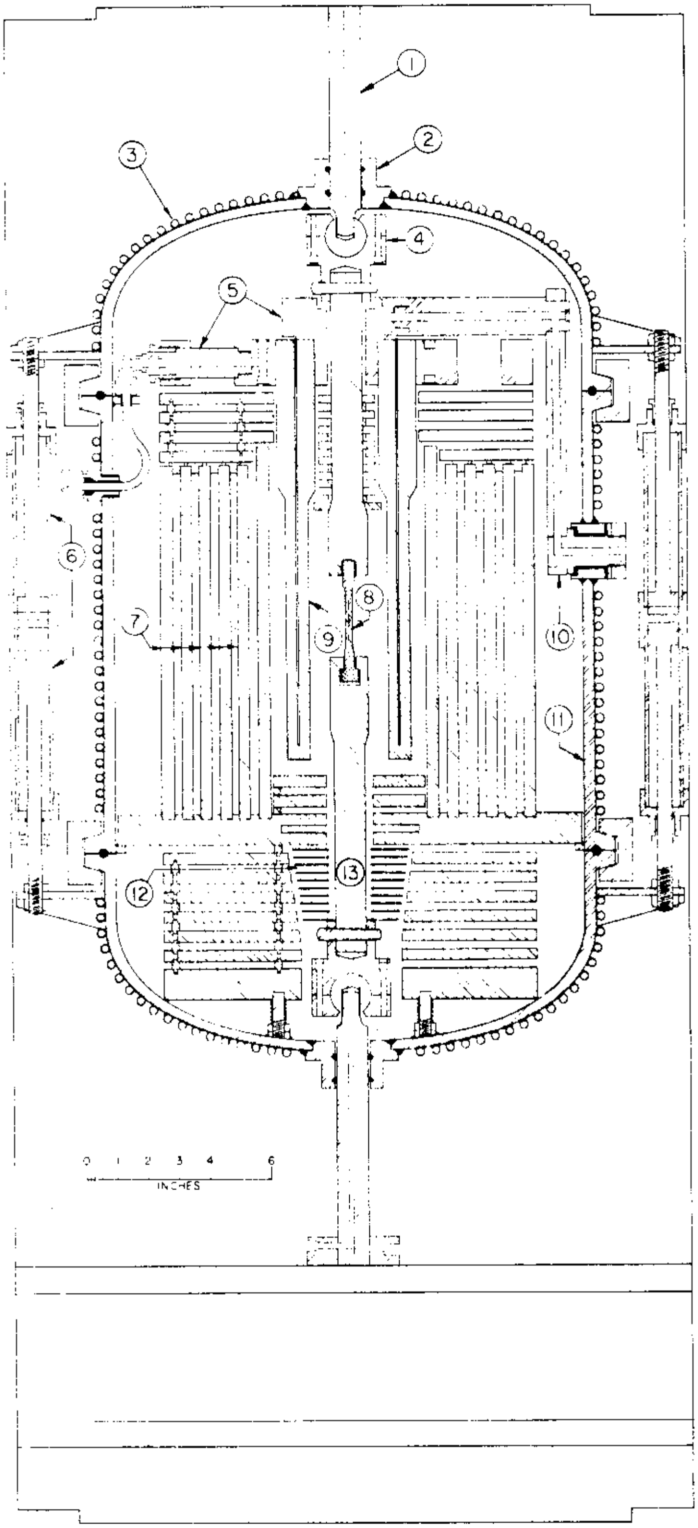

1. LOAD TRANSMISSION ROD
2. TRANSMISSION ROD HOUSING
3. HIGH-PRESSURE WATER COOLING COILS
4. WATER-COOLED BALL-AND-SOCKET COUPLING
5. WATER-COOLED COPPER-ELECTRODE CONNECTORS
6. HYDRAULIC END-CLOSURE LIFTS
7. CARBON RADIATION SHIELDS
8. TENSILE SPECIMEN
9. SPLIT-GRAPHITE HEATING ELEMENTS
10. WATER-COOLED TERMINALS AND BUS CONNECTORS
11. GOLD-PLATED STEEL SHELL
12. TANTALUM RADIATION SHIELDS
13. CARBON SPECIMEN GRIPS (TYPE OF CONNECTOR TO TENSILE SPECIMEN TO BEST SUIT REQUIREMENTS)

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FIGURE 1. TENSILE FURNACE SCHEMATIC CROSS SECTION

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directions away from the specimen is accomplished by a series of graphite or carbon radiation shields. The eight radial, five top, and eight bottom shields were selected to provide rapid transition temperature times while affording adequate insulation to avoid severely limiting the maximum operating temperature. In practice, it was found that the shields as such gave adequate insulation. However, the holes in the shields necessitated by the optical strain measuring system were responsible for some sacrifice in the ultimate temperature of operation.

The electrical power to heat the elements is brought into the furnace by a group of water cooled copper bus bars. Distribution and coupling of the power in the furnace is accomplished by a pair of concentric water cooled copper rings. The inner ring is split to gain the series-parallel circuit required to match the elements to the power supply. The hairpin elements, held apart at the terminal end by fired lava stone insulators, are clamped in position by controllable pressure exerted on the outer conducting ring by a water cooled copper hoop. The release of this pressure by loosening the compression nuts is all that is required to free the elements for replacement.

The ensemble of parts thus far discussed is housed in a water cooled, cylindrical pressure vessel with elliptic end sections. All ports of access to the interior of the vessel - electrical, optical or load train - are adequately sealed and insulated to withstand pressures in the range of 10^{-2} to 800 centimeters of mercury. The hydraulically removable end sections are clamped in position for increased pressure operations only. For ambient or reduced pressure operations, the action of the hydraulic cylinders (two per head) is adequate to effect a satisfactory "0" ring seal.

Access to the furnace is accomplished by swinging the center portion of the furnace about its mounting on one channel of the Instron Testing Machine. The elliptical heads are raised and lowered as applicable to free the center section for rotation. The removal of the heads also permits the access required to couple the specimen stress train to the load cell and movable platen of the Instron.

The specimen stress train consists of a series of ball and socket connected members, each member being a different material compatible with local temperature conditions and desired heat transfer. The train members that join directly to the testing machine are water cooled steel. These members pass through the pressure vessel inside "0" ring sealed glands. Once inside the furnace, each steel member is connnected to a column of ungraphitized carbon by the first of two ball and socket joints, using a taper pin to effect the carbon to steel grip. These taper pins are easily removable to permit the carbon-graphite-specimen portion of the stress column to swing free with the furnace. The carbon members, used to thermally insulate the specimen and grippers from the metal sections, are screwed into the graphite specimen gripping sections. The grippers also containing a ball and socket swivel, hold the test specimen in a threaded section. The swivels, two at each end, are incorporated in the design to insure pure tensile stress on the specimen even in the event of a slight misalignment of the terminal members of the column. Counterweighting of the upper portion of the stress column to

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relieve the load arising from pressurization of the furnace is used for pressure tests. The required counterweight is evaluated by comparing the room temperature strength with and without pressure.

3.2. Furnace Accessories

The furnace in position for testing is depicted in Figure 2. The power to operate this furnace is derived from two 800 amp d.c. constant potential welding units connected in parallel. Power variation from the welders necessary to control the test temperature of the specimen is obtained by manually controlling the regulated voltage output. This power source is ample to vary continuously the specimen temperature from 1000°C to the upper limit of the furnace (2760°C when optical projection of the specimen image is required) when the heating elements are properly matched. Continued testing, coupled with the rapid temperature changes and oxidation of elements during reloading, does cause considerable heating element deterioration. This results in a power supply mismatch and consequently in a reduction of the maximum temperature. The continuous flow of a nonreactive gas argon for neutral tests is used to prevent excess formation of gaseous carbon compounds.

The specimen temperature is measured by a disappearing filament optical pyrometer. Corrections for temperature deviation caused by viewing the specimen through a fused silica window were incorporated in the pyrometer calibration. The pyrometer was calibrated to achieve the overall correction to a N.B.S. standard lamp viewed with the window inserted between the pyrometer and black body cavity. Black body conditions are prevalent in the furnace, thus eliminating additional corrections.

A port directly opposed to the opening used to read the specimen temperature serves as an aperture for the projection of the specimen image from the furnace. The temperature port is used in duality to provide the dark background required to obtain the image from the self-illuminated specimen. Once removed from the furnace, the image is passed through a lens for magnification. A series of front-surfaced mirrors serve to separate the segments of the image containing the fiducial marks and reposition these close to one another for display purposes while continuing the magnification process. When a magnification factor of 50 has been reached, the image is projected on a ground glass screen. From the other side of the screen, this image is photographed simultaneously with a slave stress recorder and a clock by a time lapse camera. A variation of the distance between the fiducial marks at the accompanying stress level, as determined by reading the film, can then be accurately correlated to actual change in specimen length since the reference lengths and magnifications are known. Strain measurements accurate to 2×10^{-4} are attainable by the system.

3.3. Specimen

The specimen used throughout the grade ZTA graphite testing program is diagrammed in Figure 3. The gauge dimensions of this specimen were selected to be as large as possible to reduce size effects and at the same time small enough that the specimen could be failed within the 400-pound

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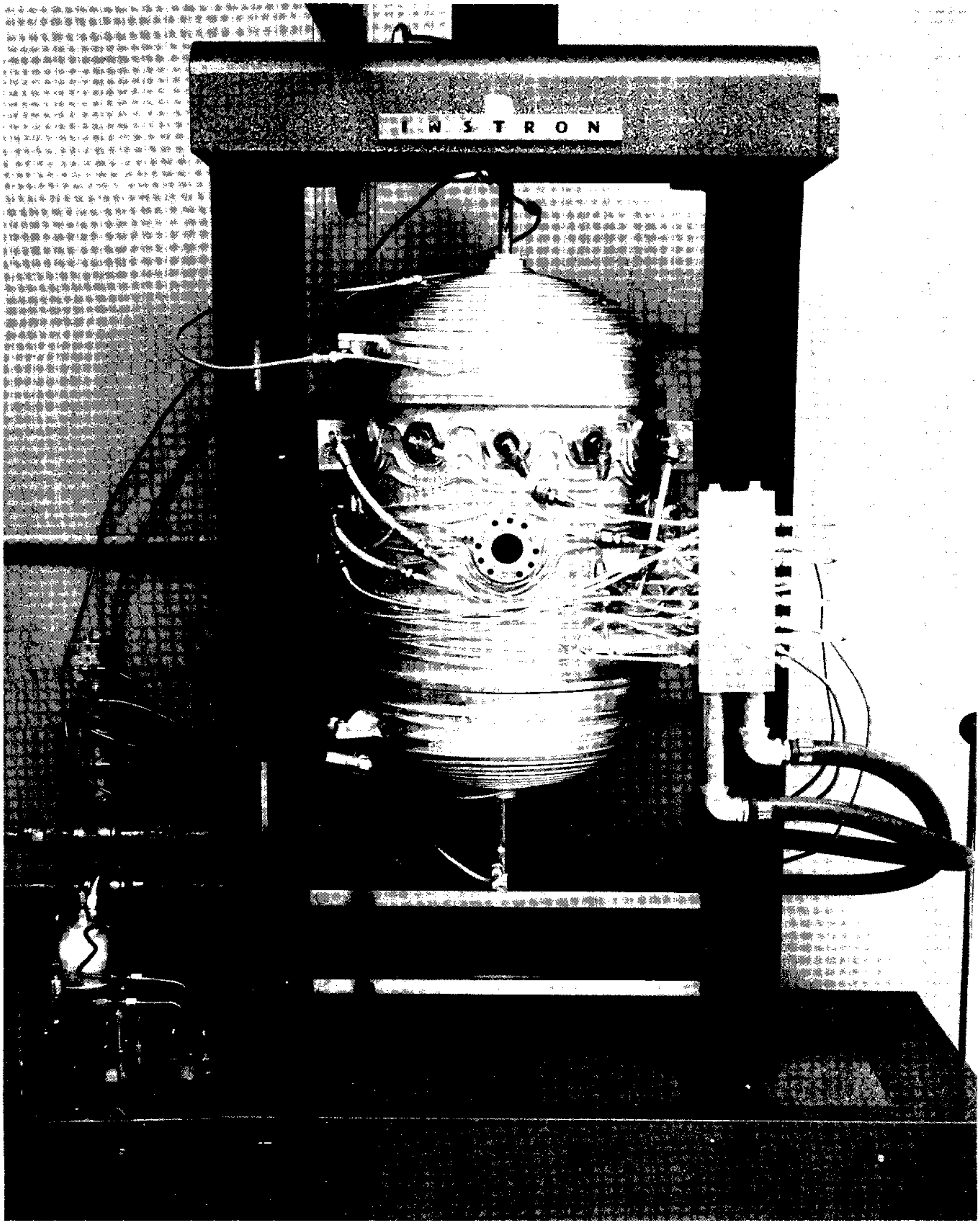

FIGURE 2. TENSILE FURNACE PHOTOGRAPHED AS OPERATED

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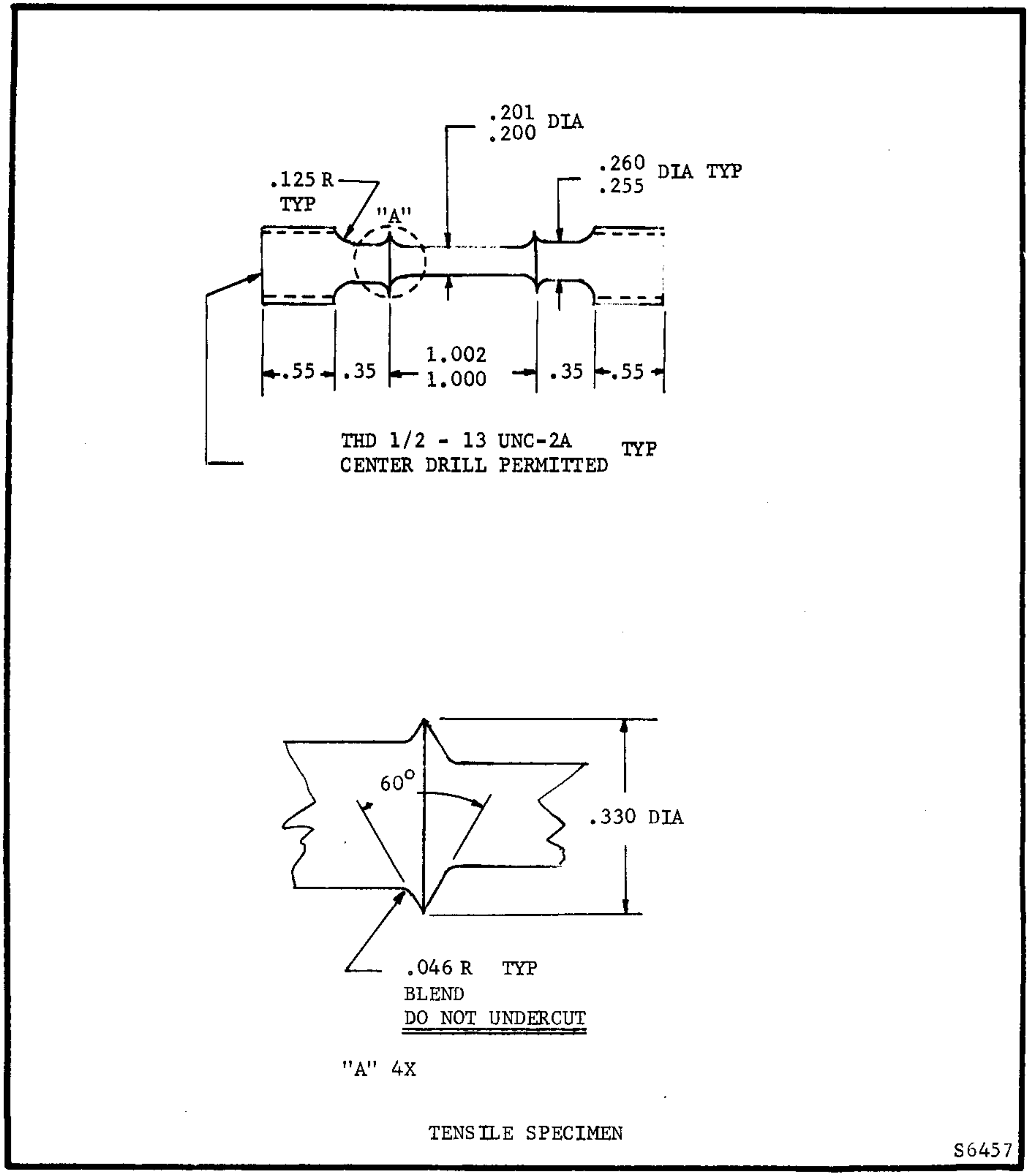

FIGURE 3. TENSILE SPECIMEN SCALED DRAWING

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load limit of the stress train. In the interest of comparing the data obtained with the varying factors of temperature, pressure, atmosphere, pretreatment and load rate, a single specimen size was extremely desirable. The specimen used for the calibration of the furnace using ATJ grade graphite was of similar size save for the increased gauge diameter (0.250 inch). This increase in size was based on the known properties of the material, and would serve as a basis to assure that the system gave results comparable to other testing facilities.

3.4. Calibration

Prior to the testing of the ZTA graphite, a series of tests was performed on National Carbon Company grade ATJ graphite. This preliminary testing served the dual purpose of verifying that the testing results of the new facility just assembled were compatible with the results of other facilities and of providing a shakedown period on the furnace for familiarization and correction of minor problems. The results of these preliminary tests are presented in Table I and Figures 4 and 5. Also included on Figures 4 and 5 are the range of results reported elsewhere (1) on ATJ graphite tensile strength variations with temperature. It will be noted that the results of the facility used are within the deviation range for ATJ graphite particularly where comparable size specimens are used. On the basis of this, it was assumed that the results measured on ZTA graphite could be reproduced elsewhere and constituted reliable data.

TABLE I

Temperature-Strength Data for ATJ Graphite
Tested in 1 Atmosphere of Argon
Load Rate: 0.020 in/min

Specimen Number	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
	With Grain		
12	70	21	2935
1	2000	1095	3130
2	2620	1440	3495
7	2900	1595	3740
11	2940	1620	4025
8	3120	1715	4125
10	3650	2010	3770
6	3990	2200	3380
4	4090	2260	4225
14	4300	2380	4565
	Across Grain		
2	70	21	2630
5	1530	830	3330
13	2250	1230	3380
4	2520	1380	3480
9	2920	1605	4395
10	3330	1830	3395
3	3350	1840	4065
17	3620	1995	4990
14	3660	2020	4025
1	4020	2220	4565

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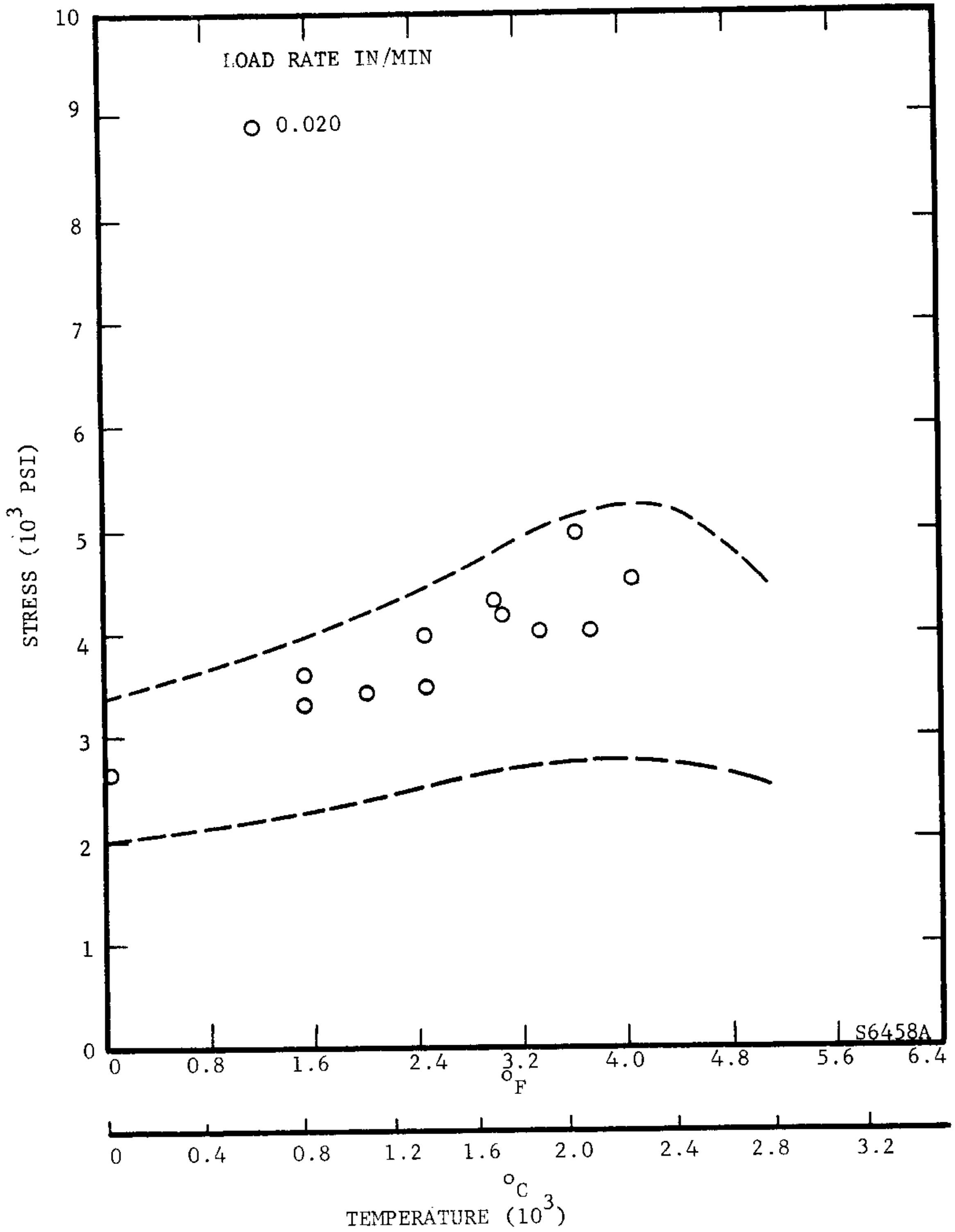

FIGURE 4. GRAPH ULTIMATE TENSILE STRESS VERSUS TEMPERATURE FOR ATJ GRAPHITE ORIENTED WITH GRAIN

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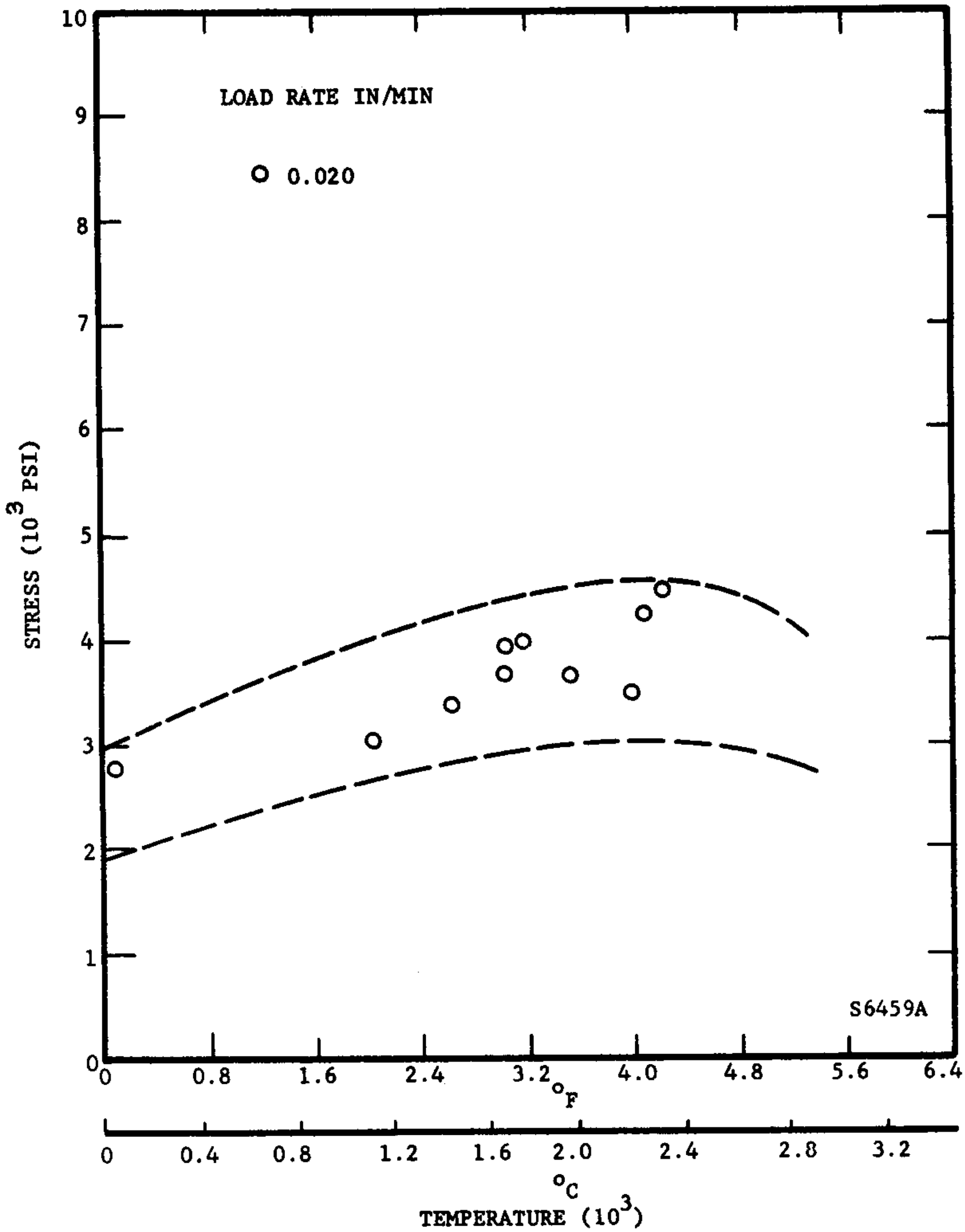

FIGURE 5. GRAPH FOR ATJ GRAPHITE ORIENTED ACROSS GRAIN

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4. TESTING RESULTS

Once an adequate number of specimens of National Carbon Company grade ATJ graphite had been tested to confidently use the equipment, National Carbon Company grade ZTA "recrystallized" graphite was used for all subsequent testing. The ZTA grade graphite tested is one of a group of artificial graphites produced by the hot working of a fine grain prefabricated commercial graphite.(5) Test specimens were cut from several production blocks fabricated while this material still was in a development stage and the process details were not yet standardized. The overall spread of data was therefore greater than might normally be expected. This variation is evidenced in the succeeding results by comparing the magnitude of the values between distinctive tests. Within each similar testing realm, this variation disappears, since all specimens for a particular set of tests were obtained from a single block. Variations in this subset must be characteristic of that single block from which the specimens were taken.

The experimental results will be described for each set of measurements performed on the ZTA graphite test coupon. The identities of the parent blocks from which the samples were cut are given in the Tables. A discussion of the various blocks is given in the Appendix. Bulk densities of individual test specimens listed in the Tables were, unless otherwise stated, measured on the rectangular blank before machining.

4.1. Exploratory Tests

The first series of tests conducted on the ZTA graphite were of an exploratory nature. Since the maximum strength that would be encountered constituted an unknown, these tests were conducted to obtain a value suitable for establishing a gauge diameter as discussed in subsection 2.3. The results of this exploratory testing are tabulated in Table II and presented graphically in Figure 6. From the data obtained, a gauge diameter of 0.200 inch was selected as the maximum safe size.

4.2. 3000°C Annealing

Concurrent with the exploratory testing, a series of specimens which had been annealed at 3000°C were also tested. As both groups of test coupons had been cut from the same block of ZTA grade graphite, a comparison of the test results should reveal any major changes that might occur. Since a comparison of Table III with Table II or Figure 7 with Figure 6 for material of like orientation reveals no consistent deviation of values between the 3000°C annealed material and its unannealed counterpart, the annealed specimen testing was not continued.

4.3. Load Rate Sensitivity

Upon completion of the exploratory tests, data were obtained to establish the effect of load rate on the tensile strength of the graphite. The variation in the ultimate tensile strength as a function of temperature was measured for

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TABLE II

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Argon Load Rate: 0.020 in/min

ZTA Block 1714

Specimen Number	Density(g/cc)***		Temperature		Ultimate Tensile Strength, psi
	Short	Long	°F	°C	
			With Grain		
2 - 10	1.978	1.972	70	21	4045
2 - 18	1.980	1.987	2400	1315	5410
2 - 22	1.954	1.968	2850	1565	5250
2 - 6			3055	1680	5095
2 - 2			3345	1840	5985
2 - 9			3605	1985	7510
2 - 1*			3875	2135	4900*
2 - 8			3975	2190	7350
2 - 15			4045	2230	5600
2 - 14**	1.958	1.974	4395	2425	8115**
2 - 21	1.944	1.970	4440	2450	7950
2 - 16	1.941	1.960	4500	2480	7630
2 - 12*			4520	2490	6630*
2 - 5			4755	2620	9295
			Across Grain		
1 - 2	1.970	1.954	70	21	1495
1 - 4	1.966	1.952	2410	1220	1210
1 - 18	1.975	1.947	2850	1565	1530
1 - 22			3055	1680	2390
1 - 17			3235	1780	2480
1 - 7			3515	1935	1690
1 - 8			3820	2100	2070
1 - 23*			3885	2140	1180*
1 - 3*			3885	2140	1050
1 - 11**			4155	2290	1780**
1 - 21*			4280	2350	1810*
1 - 13	1.967	1.937	4300	2375	2005
1 - 20	1.970	1.936	4440	2450	2160
1 - 16	1.982	1.960	4440	2450	1810
1 - 19			4650	2560	1780

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* Load rate: 0.200 in/min

** Load rate: 0.002 in/min

*** Measured by water immersion on broken fragments.

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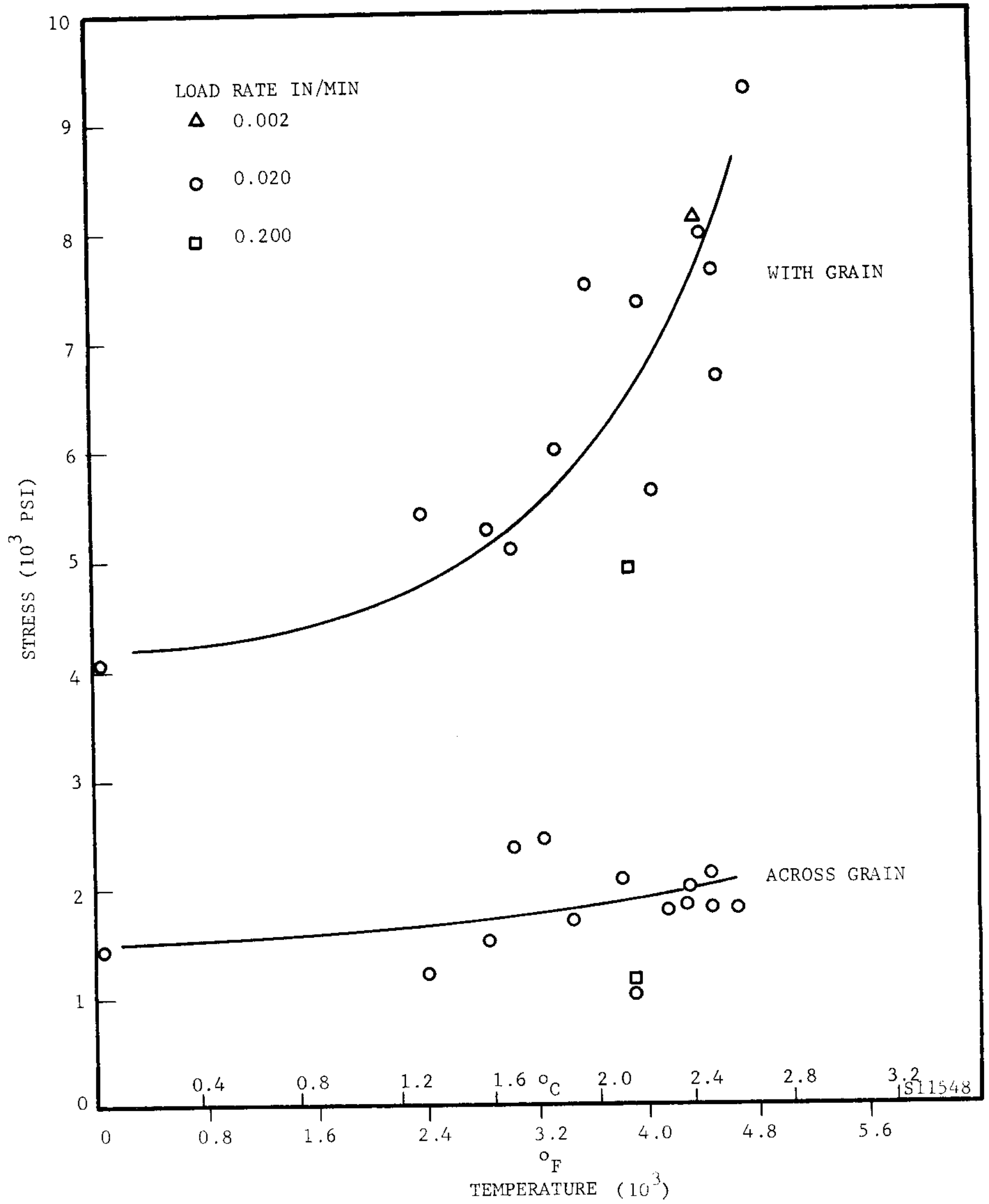

FIGURE 6. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE

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TABLE III

Temperature-Strength Data for 3000°C Annealed ZTA Graphite Tested in 1 Atmosphere of Argon Load Rate: 0.020 in/min

ZTA Block 1714

Specimen Number	Density(g/cc)*** Short	Density(g/cc)*** Long	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
4 - 7	1.967	1.966	70	21	3535
4 - 14	1.988	1.971	2445	1340	4650
4 - 10	1.975	1.981	2860	1570	3950
4 - 19			3055	1680	5985
4 - 16			3300	1815	6870
4 - 22			3515	1935	7350
4 - 8*			3955	2180	5635*
4 - 2**			4065	2240	7955**
4 - 9*			4065	2240	5890*
4 - 6			4155	2290	7990
4 - 3			4245	2340	8850
4 - 12			4425	2440	7195
4 - 18			4505	2465	6370
4 - 5			4540	2505	6420
4 - 15			4730	2610	8275
			Across Grain		
3 - 11	1.976	1.946	70	21	1180
3 - 7	1.950	1.944	2390	1310	1050
3 - 1	1.978	1.999	2850	1565	1240
3 - 12			3055	1680	1750
3 - 19			3315	1825	2035
3 - 6			3595	1980	1465
3 - 2			3955	2180	1750
3 - 14*			3975	2190	985*
3 - 15			3975	2190	2325
3 - 4			4205	2220	1335
3 - 21			4225	2325	1815
3 - 22*			4260	2350	1210*
3 - 20**			4295	2370	1400**
3 - 18			4435	2445	1845
3 - 13			4650	2565	1940

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* Load rate: 0.200 in/min

** Load rate: 0.002 in/min

*** Measured by water immersion on broken fragments.

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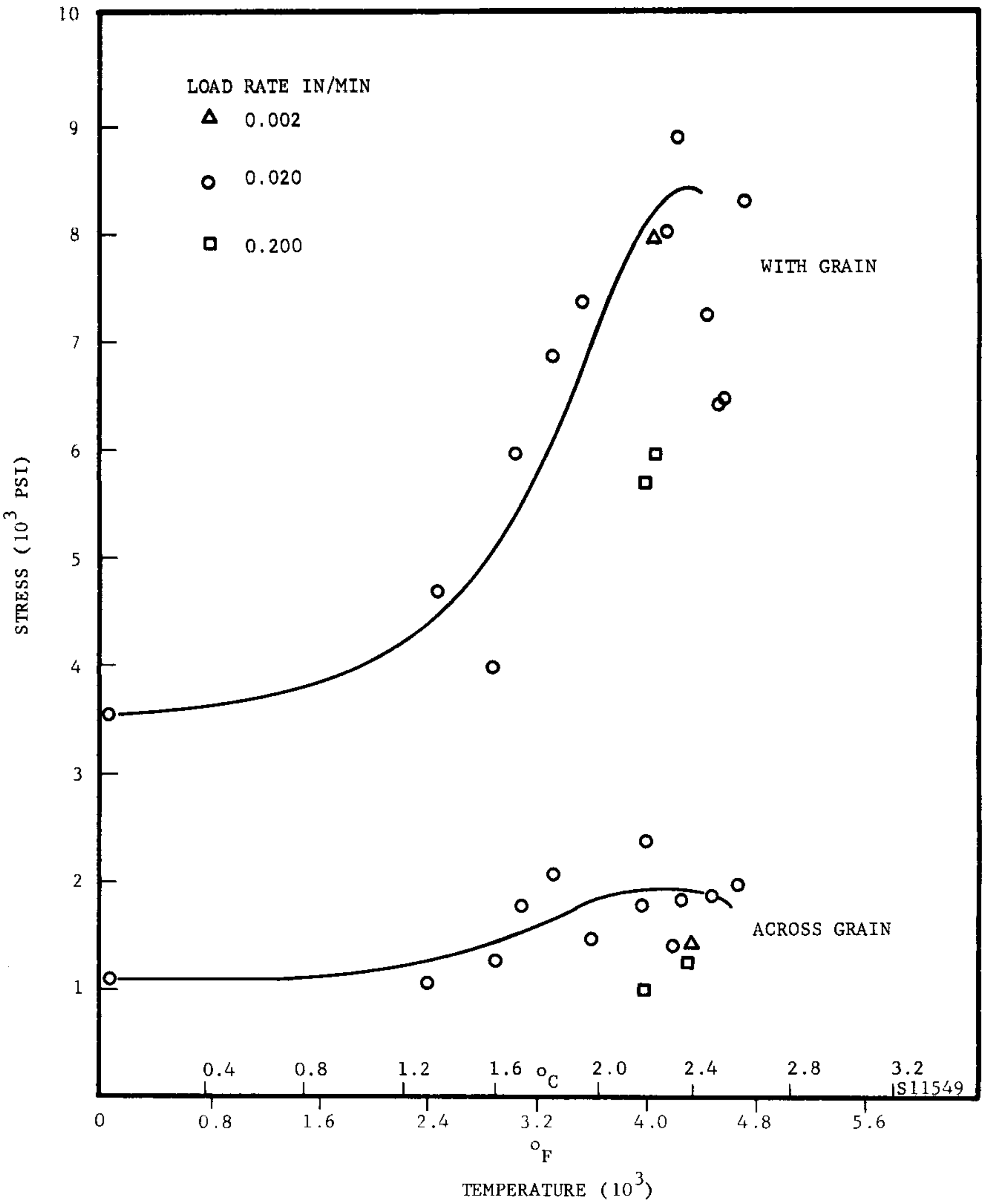

FIGURE 7. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE 3000°C ANNEALED

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three loading rates covering two decades (0.200, 0.020 and 0.002 inch per minute platen speed). These data are presented in Tables IV, V and VI and Figure 8. Data for the 0.002 inch per minute rate were not obtained above 2260°C (4100°F) as the time to fail the specimen was approximately 15 minutes. Tests of this duration can only be interpreted in terms of creep, and creep was not the property of interest. The tests conducted near 2650°C (4800°F) consumed approximately 5 minutes, again marginal to short time creep tests.

Some of the tensile properties of the ZTA grade artificial graphite may be delineated from these measured results. First, the ultimate tensile strength monotonically increases with temperature to a moderately well defined peak in the vicinity of 2500°C (4530°F). A similar phenomenon has been observed in most other artificial graphite.(1,2) Second, above 1650°C (3000°F), the ultimate tensile strength is load rate sensitive with the slower load application resulting in an increased strength. A discussion of this observation is deferred until Section 6. Third, the ZTA graphite strength reflects a considerable degree of anisotropy, a consequence of the increased ordered crystallinity compared to other artificial graphites.

4.4. 2000°C Vacuum Degas

Tests were conducted to evaluate the effect of removing contained gases on the ultimate tensile strength of the ZTA grade graphite. The specimens for this evaluation were tested in 1 atmosphere of argon after having been treated by evacuation to at least 10^{-5} mm Hg and heated to 2000°C at this pressure for a period in excess of 30 minutes. Upon cooling under vacuum and being returned to air pressure using clean argon, the specimens were tested for strength. The results of the degassing tests are given in Table VII and Figures 9 and 10.

An examination of these results indicate that at the lower test temperature the strength is increased by prior gas removal. However, above the degassing temperature there appears to be no measurable effect. It is not possible from the available data to delineate the cause of the observed phenomena. Two conceivable factors are: a) temperature activated gas removal by self-diffusion and b) high temperature redistribution of bond weakening gases adsorbed in the structure.

4.5. Density Variation

Upon examination of the broken tensile specimens, it was noted that the shorter fragment had the higher density for across-grain oriented specimens.* The density variation of the with-grain oriented specimens was insufficient to establish a preferred break location. As a consequence of this apparent variation in strength with density, a series of test specimens, selected to give the widest available density variation were broken at selected elevated temperatures. The results of these tests are given in Table VIII and Figure 11. The data substantiate the observation that the lower density graphite is stronger for the across-grain oriented material. For the with-grain oriented material the data are inconclusive. Prior evidence (5) indicates that the

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* See Tables II, III, and V.

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TABLE IV

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Argon Load Rate: 0.002 in/min

ZTA Block 1721

Specimen Number	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
	With Grain		
5 - 14	2770	1525	3020
5 - 25	2775	1530	2835
5 - 28	2775	1530	3215
5 - 2	3315	1825	4610
5 - 13	3325	1830	4490
5 - 23	3325	1830	4260
5 - 17	4065	2240	7390
5 - 19	4065	2240	6870
5 - 26	4065	2240	6380
	Across Grain		
6 - 1	2775	1530	1115
6 - 27	2775	1530	1335
6 - 30	2785	1535	1205
6 - 10	3310	1820	1870
6 - 12	3315	1825	1660
6 - 26	3325	1830	1660
6 - 5	4065	2240	2250
6 - 14	4065	2240	2380
6 - 15	4065	2240	1910

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TABLE V

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Argon Load Rate: 0.020 in/min

ZTA Block 1721

Specimen Number	Density(g/cc)* Short	Density(g/cc)* Long	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
5 - 6			72	22	3185
5 - 1	1.871	1.893	2775	1530	3120
5 - 11	1.883	1.896	2775	1530	2835
5 - 15	1.940	1.954	2775	1530	2985
5 - 4			3315	1825	4390
5 - 16			3315	1825	4230
5 - 29			3310	1820	3980
5 - 3			4055	2235	6840
5 - 5			4065	2240	6530
5 - 20			4065	2240	6680
7 - 9	1.994	1.982	4405	2430	8530
7 - 10	1.998	1.980	4415	2435	8180
7 - 13	1.973	1.983	4415	2435	8210
7 - 5			4710	2600	6370
7 - 25			4705	2595	6080
7 - 27			4710	2600	6620
7 - 11			4990	2755	6200
7 - 23			4980	2750	6370
7 - 29			4975	2745	6020
			Across Grain		
6 - 20			72	22	1180
6 - 2	2.004	1.963	2785	1530	1180
6 - 7	1.971	1.915	2775	1530	1205
6 - 19	1.936	1.885	2770	1525	1365
6 - 11			3310	1820	1840
6 - 21			3315	1825	1640
6 - 29			3315	1825	1960
6 - 4			4065	2240	2100
6 - 24			4055	2235	2000
6 - 25			4055	2235	1880
6 - 23	2.000	1.963	4415	2435	1910
8 - 4	1.997	1.917	4405	2430	1910
8 - 23	1.916	1.886	4400	2425	2670
8 - 24	1.984	1.943	4405	2430	2350
8 - 7			4710	2600	1430
8 - 9			4705	2595	1460
8 - 17			4705	2595	2130
8 - 10			4975	2745	1400
8 - 14			4965	2740	1910
8 - 22			4980	2750	1620

* Measured by water immersion on broken fragments.

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TABLE VI

Temperature-Strength Data for ZTA Graphite
Tested in 1 Atmosphere of Argon
Load Rate: 0.200 in/min

ZTA Block 1721

Specimen Number	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
	With Grain		
5 - 8	2775	1530	3405
5 - 21	2785	1535	2960
5 - 24	2775	1530	2780
5 - 10	3310	1820	4100
5 - 18	3325	1830	3930
5 - 22	3315	1825	3660
5 - 7	4055	2235	5190
5 - 12	4045	2230	5030
5 - 30	4055	2235	4780
5 - 9	4400	2425	5400
5 - 27	4400	2425	5350
7 - 8	4415	2435	6090
7 - 22	4695	2590	5100
7 - 26	4705	2595	4140
7 - 30	4710	2600	5700
7 - 3	4980	2750	4880
7 - 4	5000	2760	5170
7 - 14	4975	2745	5450
	Across Grain		
6 - 16	2775	1530	1205
6 - 17	2775	1530	1365
6 - 28	2775	1530	1460
6 - 8	3315	1825	1520
6 - 13	3315	1825	1650
6 - 18	3325	1830	1560
6 - 6	4055	2235	1720
6 - 9	4055	2235	1590
6 - 22	4055	2235	1650
8 - 11	4400	2425	2040
8-- 18	4405	2430	2450
8 - 19	4400	2425	2520
8 - 30	4415	2435	2450
8 - 5	4695	2590	1280
8 - 15	4695	2590	1080
8 - 29	4695	2590	1780
8 - 8	4975	2745	1080
8 - 20	4975	2745	1400
8 - 28	4965	2740	1620

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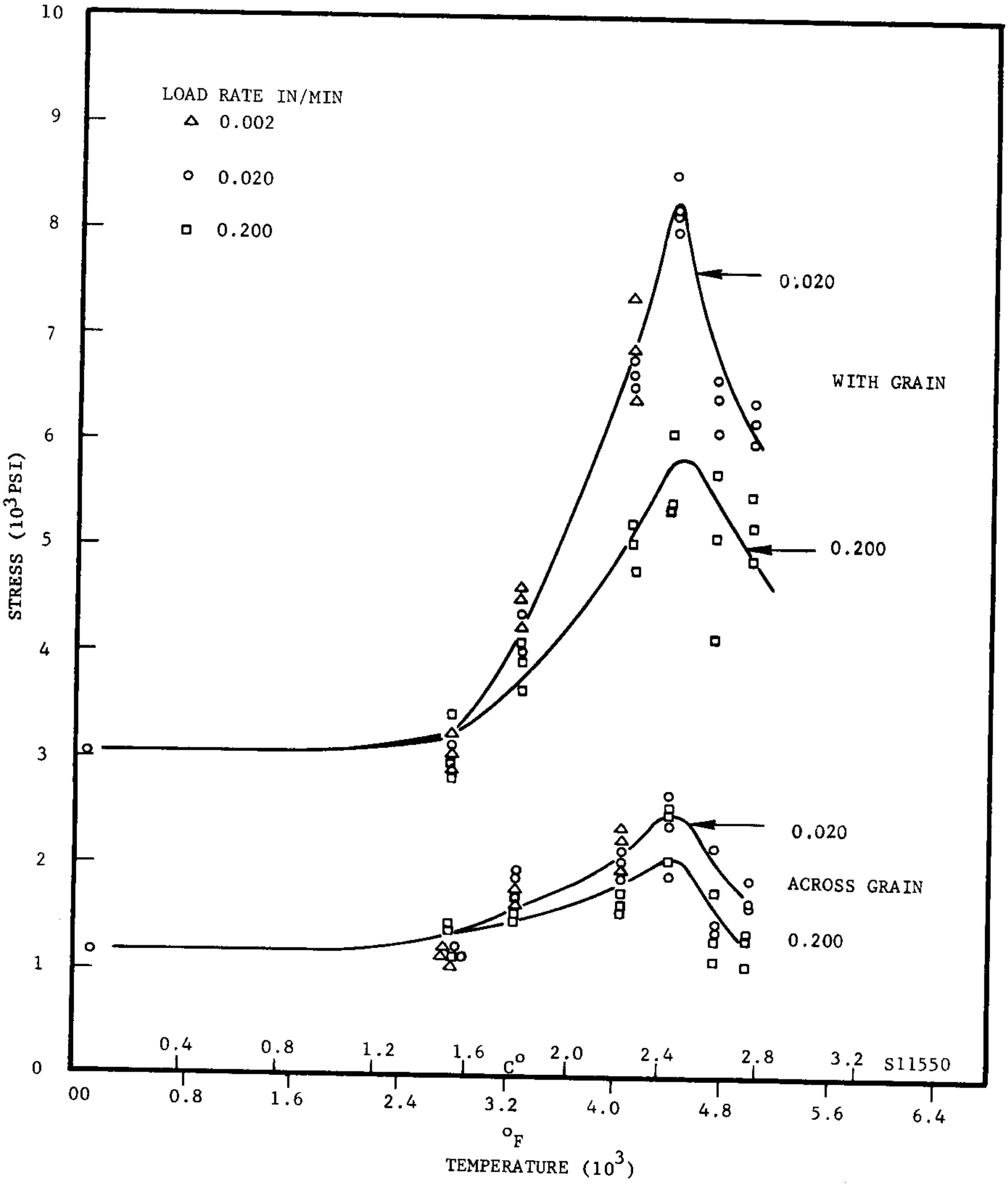

FIGURE 8. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE VARIABLE LOAD RATE

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TABLE VII

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Argon After Vacuum Degassing* Load Rate: 0.020 in/min

Specimen Number**	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
		With Grain		
7 - 16		3445	1895	6640
7 - 17		3450	1900	7050
7 - 18		3450	1900	7150
7 - 1		4705	2595	6130
7 - 7		4710	2600	6410
7 - 19		4710	2600	6020
33 - 9	1.968	3210	1765	5980
33 - 18	1.965	3235	1780	5890
33 - 11	1.973	3380	1860	5730
33 - 24	1.953	3975	2190	6330
33 - 30	1.974	4075	2245	6590
33 - 15	1.963	4090	2255	6900
		With Grain - No Degas		
33 - 5	1.971	3235	1780	5310
33 - 1	1.954	4035	2225	6690
33 - 25	1.961	4035	2225	6850
		Across Grain		
8 - 1		3445	1895	2160
8 - 2		3450	1900	970
8 - 3		3445	1895	2000
8 - 6		4710	2600	1655
8 - 12		4705	2595	1685
8 - 21		4705	2595	1560

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* 2000°C (3600°F) at less than 10^{-5} mm Hg pressure for in excess of 30 minutes.

** 7- and 8- specimens from ZTA Block 1721; 33- specimens from ZTA Block 1746.

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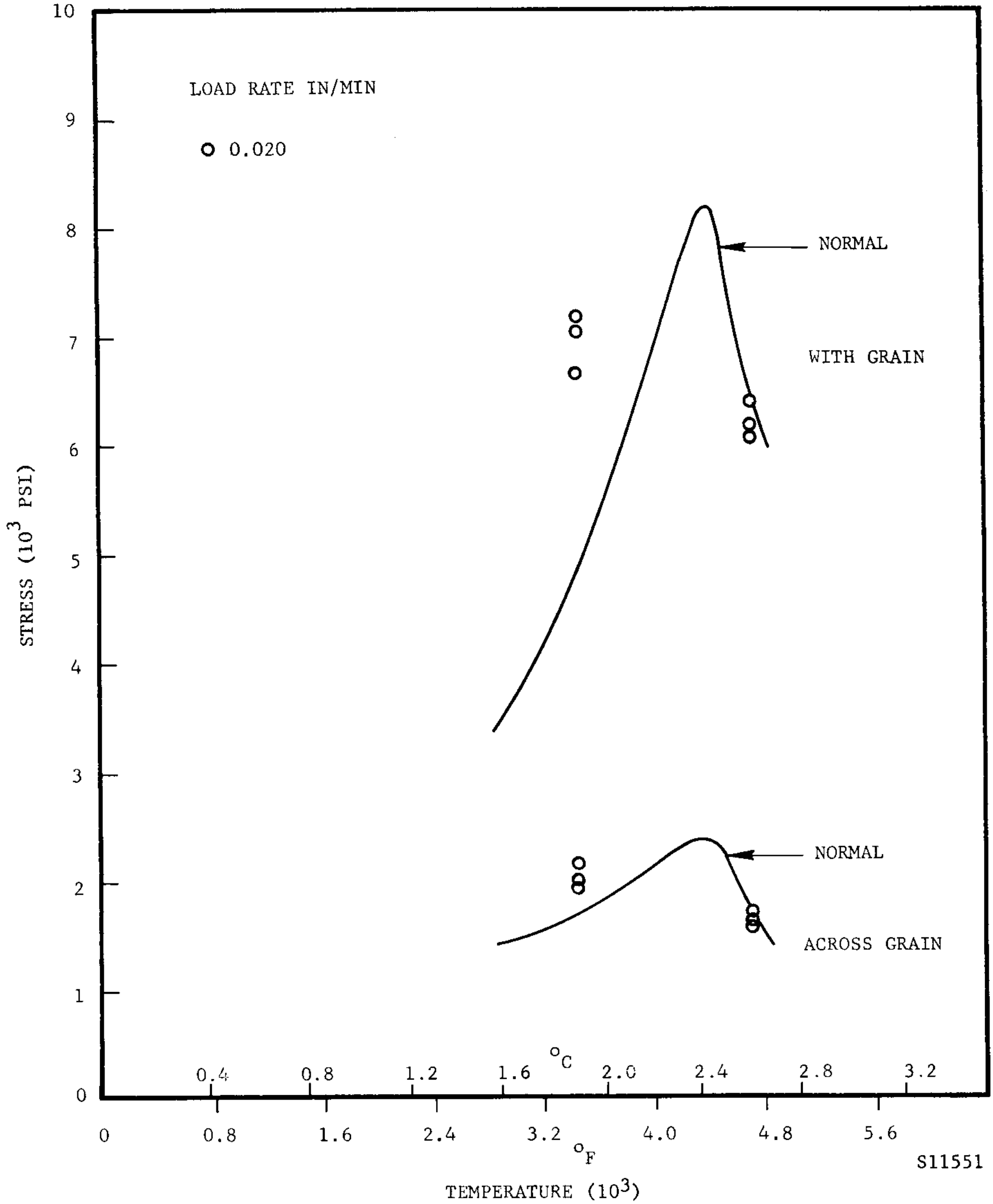

FIGURE 9. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE - 10^{-5} MM Hg VACUUM DEGAS AT 2000°C (3630°F)

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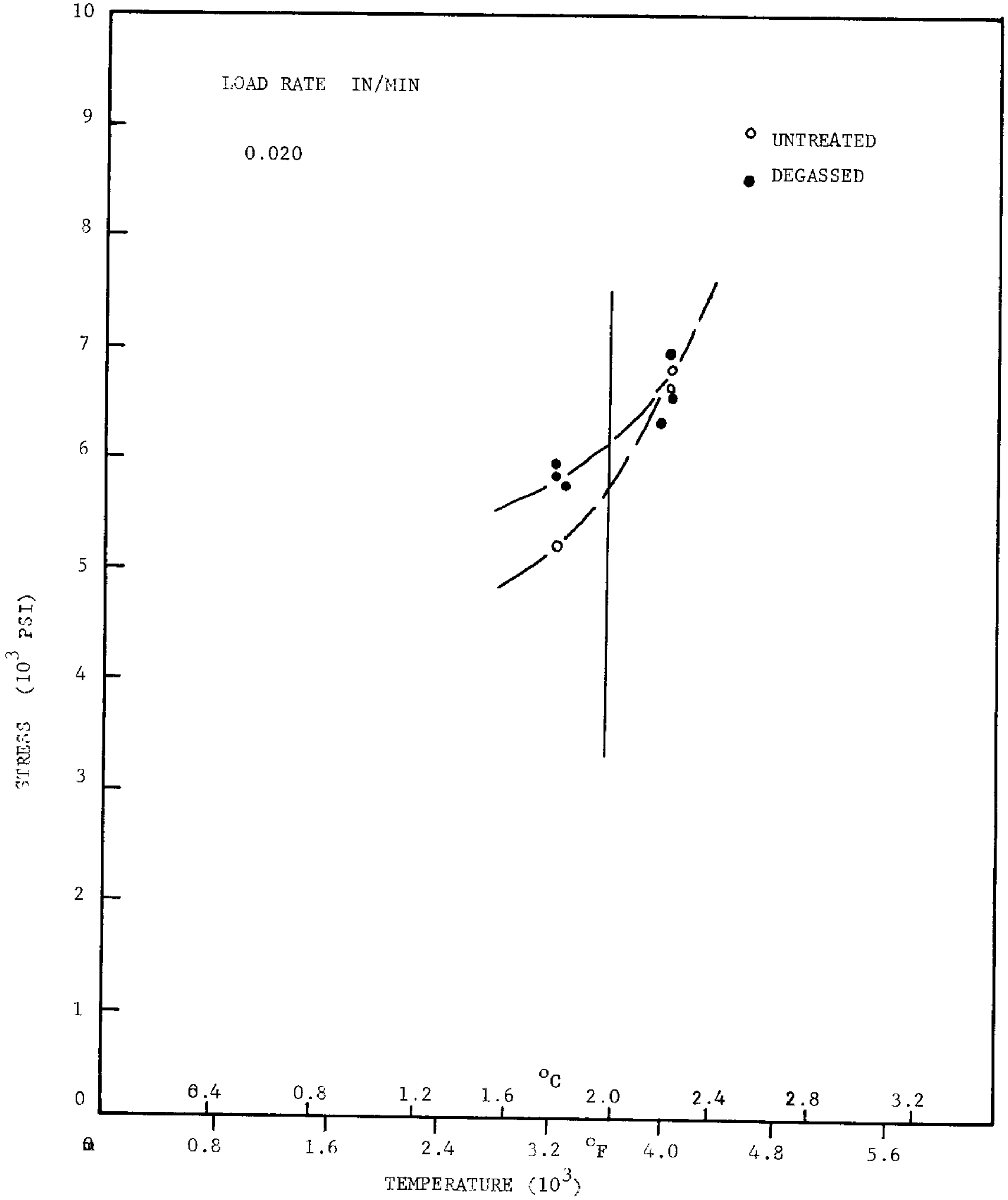

FIGURE 10. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE - 10^{-5} MM Hg VACUUM DEGAS AT 1950°C (3540°F)

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TABLE VIII

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Argon - Density Variation Load Rate: 0.020 in/min

ZTA Block 1745

Specimen Number	Density* g/cc	Temperature °F	°C	Ultimate Tensile Strength psi
		With Grain		
13 - 3	1.967	3795	2090	5900
13 - 4	1.970	3795	2090	5860
15 - 8	1.974	3780	2080	5850
21 - 5	1.989	3795	2090	6440
21 - 7	1.989	3780	2080	6350
21 - 1	1.988	3785	2085	5650
9 - 1	1.960	4270	2355	7350
9 - 6	1.958	4260	2350	7660
9 - 7	1.959	4260	2350	7590
23 - 1	1.993	4270	2355	6810
23 - 3	1.992	4260	2350	6630
23 - 5	1.991	4260	2350	6910
		Across Grain		
10 - 4	1.960	3780	2080	1500
14 - 3	1.967	3795	2090	1535
10 - 6	1.959	3795	2090	1485
16 - 1	1.972	3785	2085	1400
16 - 2	1.972	3780	2080	1435
16 - 5	1.972	3780	2080	1380
10 - 1	1.958	4260	2350	1910
10 - 3	1.956	4260	2350	1880
10 - 5	1.956	4270	2355	1910
16 - 3	1.974	4260	2350	1690
16 - 6	1.973	4260	2350	1720
16 - 7	1.973	4260	2350	1655

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* Measured on blanks before machining test specimens.

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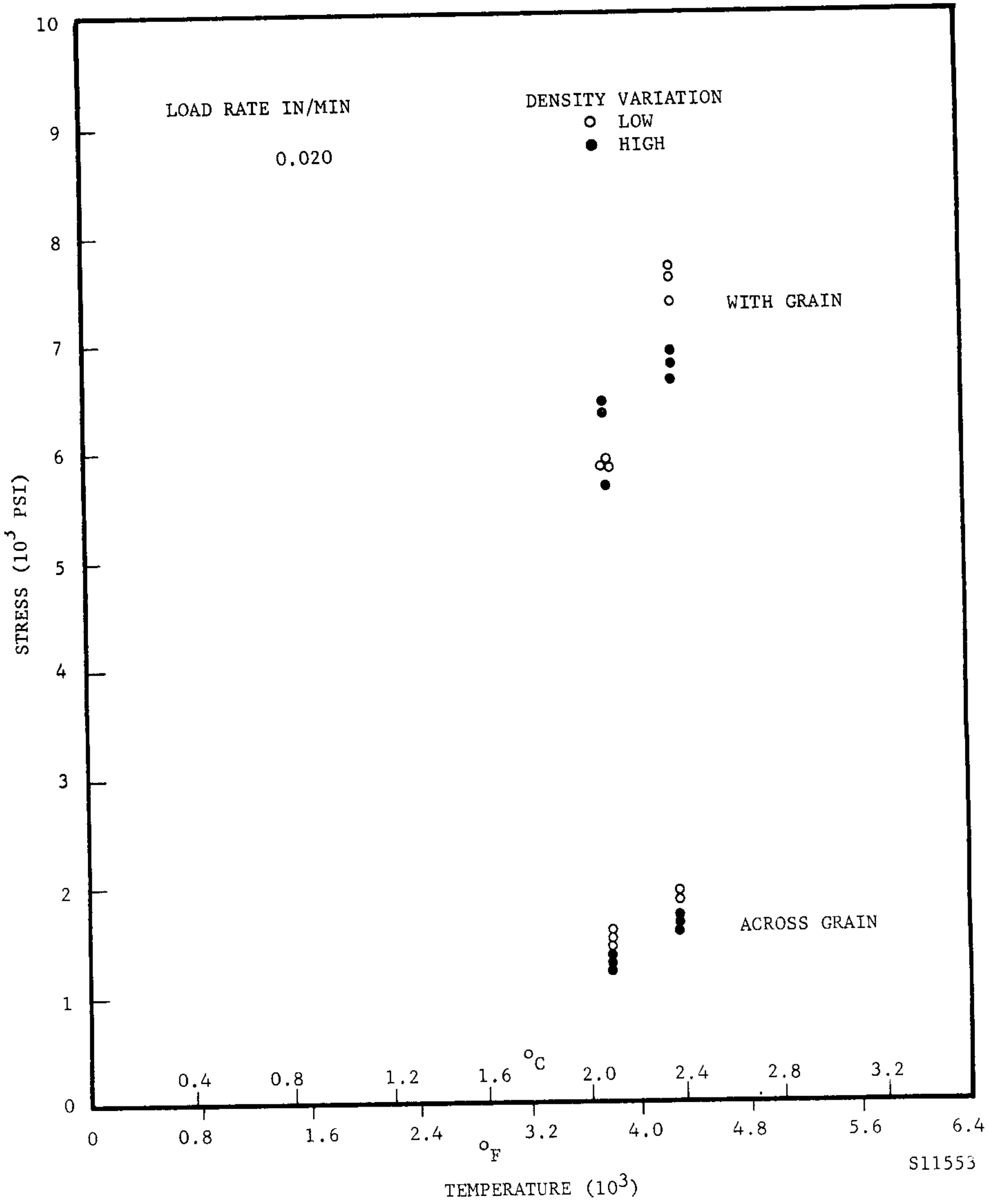

FIGURE 11. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE

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strength of ZTA graphite in the with-grain direction increases with the bulk density of the block.

4.6. Fracture Location

The measured and inferred variation of strength with density suggested further examination of the fracture location. Coupled with this was the observation that a majority of the specimens were failing near one end. To evaluate the fracture location, a series of specially prepared specimens were tested using several methods of gripping. The results of these tests are itemized in Table IX and Figure 12. From this series of tests, the following conclusions were made:

(1) The fiducial marks do not influence the break location.

(2) There was some evidence for an influence of variation of gauge section diameter on break location.

(3) Density variation over the specimen is a major contributory factor for the cross-grain direction with the specimen tending to break nearer the high density end.

From this study, it was also concluded that the gripping system in use did not affect the results within the limits of known error.

4.7. Test Atmosphere

The testing detailed thus far has been accomplished using one atmosphere of argon to surround the coupon and furnace components during elevated temperature operations. Supplemental information on the influence of atmosphere on the behavior was measured using nitrogen and carbon monoxide as the test atmospheres. These gases were selected because they react either slightly or not at all with the graphite and hence would reveal any external influence or changes in specimen strength as a function of temperature.

a. Nitrogen

The data resulting from testing ZTA grade graphite in one atmosphere of nitrogen at elevated temperatures is presented in Table X and Figure 13. These data reveal that the strength of the material is not altered by the nitrogen. However, this observation should be tempered by the realization that the test atmosphere probably has reached a stable composition at the test temperature by the time it is in contact with the specimen. This stabilization is a consequence of the gas having to pass over hot shields and heating elements prior to its impingement on the specimen.

No measurable change in section was noted on any test specimen at any temperature, even though cyanide was evolved in the exit gas stream. The major attack of the nitrogen on the graphite was taking place at the shields and elements, as evidenced by the increased erosion and shortened life of these components.

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TABLE IX

Test Data for Evaluating Fracture Location in ZTA Graphite Load Rate: 0.020 in/min

ZTA Block 1745 (except*)

Specimen Number	Density g/cc	Temperature		Ultimate Tensile Strength psi	Break Location***
		°F	°C		
			With Grain		
29-7- 2	1.988	3040	1670	5060	3 mm - remachine
29-7-15*	2.007	3030	1665	5480	1 mm - remachine
29-7-20*	1.978	3030	1665	5600	1 mm - remachine
29-7-21*	2.002	3030	1665	5510	1 mm - remachine
9-4**	1.956	3040	1670	6750	0 mm - no mark
9-5**	1.960	3040	1670	6750	1 mm - no mark
11-3**	1.964	3040	1670	6530	0 mm - no mark
23-2**	1.994	3030	1665	5860	1 mm - no mark
25-1	1.946	3040	1670	6750	3 mm - special mach
25-2	1.952	3030	1665	6750	2 mm - special mach
25-3	1.950	3030	1665	6530	3 mm - special mach
25-4	1.951	3040	1670	6010	2 mm - special mach
25-5	1.951	3030	1665	6620	2 mm - special mach
27-1	1.948	3030	1665	5820	special machine
27-2	1.944	3030	1665	5830	special machine
27-4	1.943	3030	1665	5920	special machine
19-1	1.985	3030	1665	6010	shear pin
19-3	1.984	3030	1665	5640	shear pin
19-5	1.981	3030	1665	in groove	sharp groove
19-7	1.982	3040	1670	5730	shear pin
19-8	1.982	3030	1665	6040	reduced section
11-1	1.964	3030	1665	5430 in grip	special grip
19-9	1.981	3030	1665	5380	loose thread
11-4	1.961	3030	1665	5440	loose thread
15-5	1.975	3030	1665	5630	loose thread
15-6	1.975	3030	1665	5820	loose thread
			Across Grain		
18-8-16*	1.989	3030	1665	1430	12 mm - remachine
18-8-27*	1.955	3030	1665	1490	3 mm - remachine
18-8-31*	1.960	3020	1660	1305	1 mm - remachine
10 - 10**	1.959	3040	1670	1460	5 mm - no mark
10 - 11**	1.959	3030	1665	1590	2 mm - no mark
10 - 12**	1.959	3040	1670	1490	3 mm - no mark
16 - 15**	1.974	3040	1670	1430	3 mm - no mark
16 - 16**	1.971	3030	1665	1460	3 mm - no mark
12 - 3	1.965	3030	1665	1520	7 mm - special mach
12 - 14	1.962	3030	1665	1460	2 mm - special mach
12 - 15	1.965	3030	1665	1400	3 mm - special mach
12 - 16	1.964	3040	1670	1490	3 mm - special mach
12 - 17	1.964	3040	1670	1430	10 mm - special mach
12 - 18	1.963	3040	1670	1460	4 mm - special mach

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* ZTA Block 1721 standard specimens remachined to remove fiducial ridges.

** Test specimens machined directly without fiducial ridges.

*** Distance of break location from end of gauge section.

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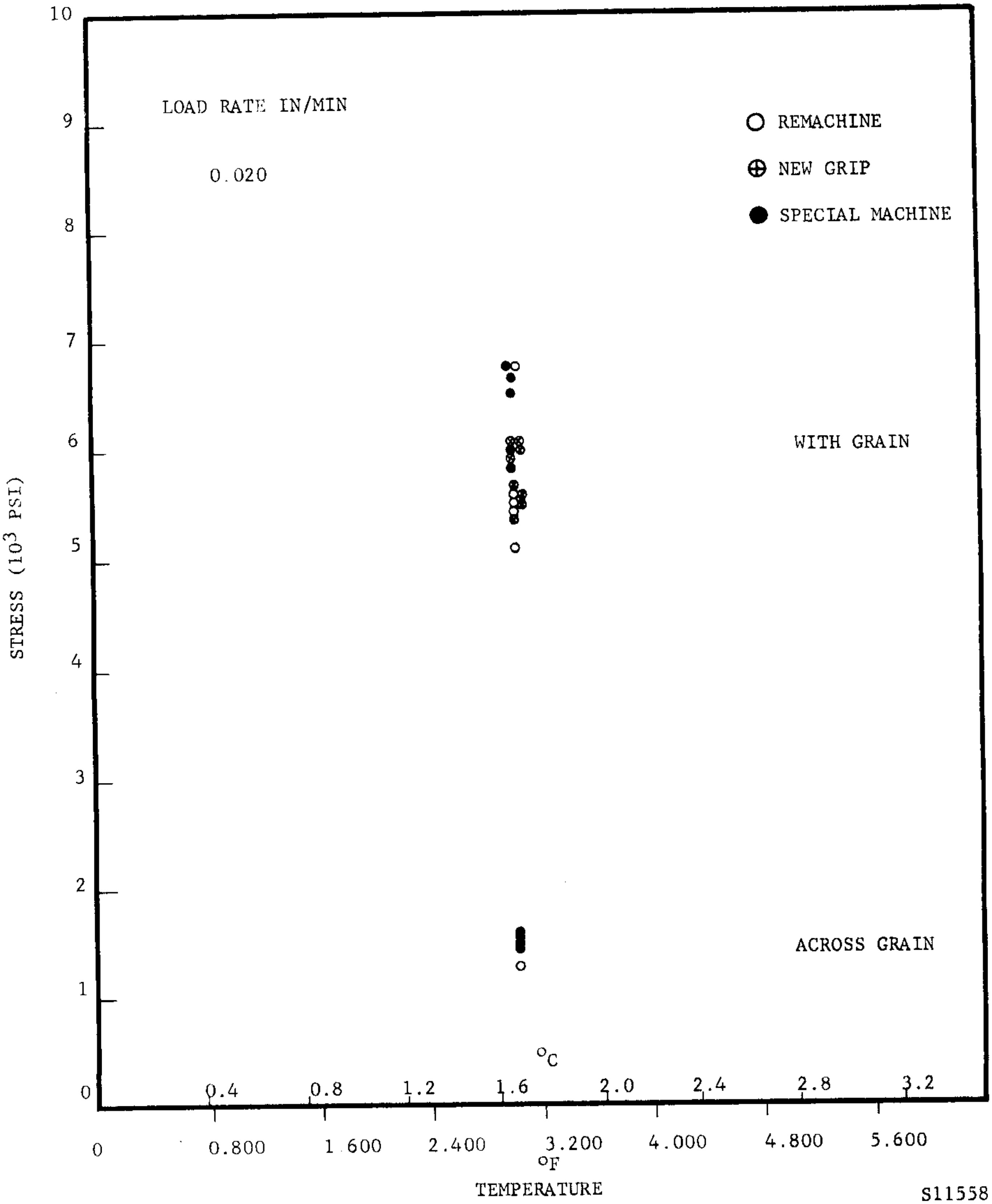

FIGURE 12 SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE FRACTURE LOCATION

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TABLE X

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Nitrogen Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature		Ultimate Tensile Strength psi
			°F	°C	
			With Grain		
1744	31 - 2	1.977	2820	1550	4700
1745	11 - 2	1.964	3385	1865	5000
"	13 - 1	1.969	3380	1860	4870
"	15 - 3	1.974	3415	1880	4010
"	17 - 5	1.976	3410	1875	4960
"	21 - 3	1.986	3425	1885	4960
"	21 - 6	1.987	3410	1875	5090
"	21 - 4	1.989	3795	2090	5970
1744	31 - 12	1.982	3795	2090	5750
"	17 - 3	1.979	3810	2100	5820
"	17 - 7	1.978	3795	2090	5430
"	21 - 9	1.989	3790	2085	5620
1744	31 - 24	1.970	4415	2435	7800
"	31 - 18	1.990	4415	2435	7830
"	31 - 9	1.972	4500	2485	8710
"	31 - 1	1.987	4550	2510	9030
"	31 - 19	1.987	4590	2535	8800
			Across Grain		
1745	10 - 9	1.959	2830	1555	1150
"	10 - 8	1.957	3370	1855	1350
"	14 - 3	1.967	3360	1850	1270
"	10 - 5	1.956	3425	1885	1110
"	12 - 2	1.964	3425	1885	1180
"	14 - 1	1.970	3415	1880	1150
"	14 - 2	1.968	3415	1880	1100
"	14 - 7	1.967	3410	1875	1210
1744	30 - 26	1.970	3795	2090	1620
"	30 - 27	1.972	3810	2100	1530
1745	12 - 4	1.961	3795	2090	1340
"	12 - 5	1.961	3795	2090	1280
"	12 - 7	1.962	3800	2095	1370
"	14 - 4	1.970	3810	2100	1240
1744	30 - 12	1.986	4420	2435	1550
"	30 - 4	1.979	4425	2440	1580
"	30 - 23	1.972	4435	2445	1685
"	30 - 3	1.981	4605	2540	1750
"	30 - 25	1.970	4610	2545	1560

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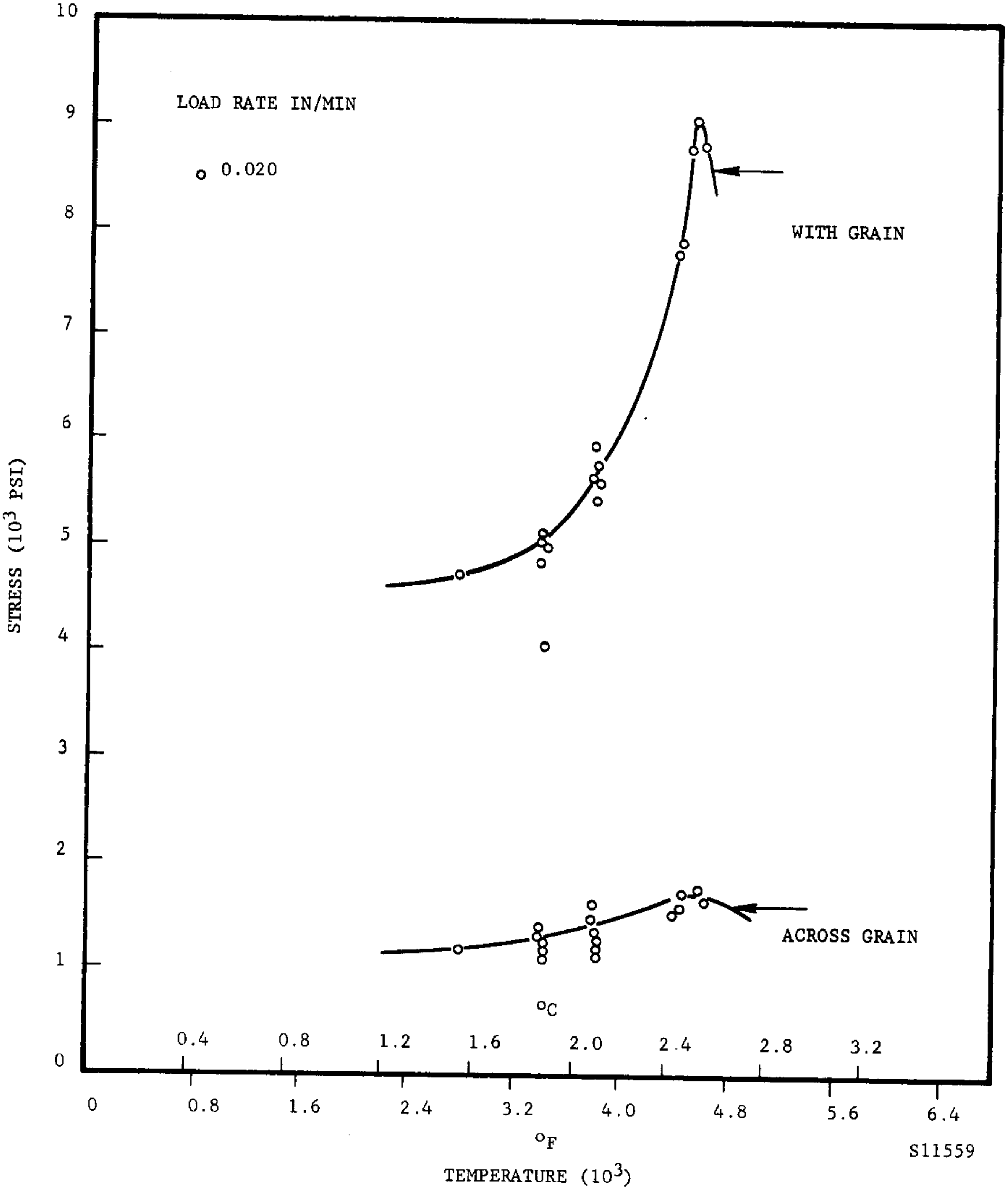

FIGURE 13. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN NITROGEN

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b. Carbon Monoxide

The data obtained on testing ZTA grade graphite in carbon monoxide, as detailed in Table XI and Figure 14, show that the strength of the material is unaffected by this atmosphere. The lack of any noticeable attack on the shields, elements, or specimen is indicative that suboxide formation was at a minimum. Other than cost considerations and toxic gas disposal, the carbon monoxide proved to be the most desirable atmosphere for testing. The principle impurity, free oxygen, is reduced at a low temperature to carbon monoxide to alleviate any extraneous atmosphere problems. Also, the heat transfer appeared to be less, as indicated by the exit cooling water temperature.

4.8. Pressure

In the previous series of tests, it was found that a change in atmosphere does not affect the strength when chemical attack is not available. In this series of tests, the influence of pressurized argon as the surrounding atmosphere will be enumerated. For this sequence of measurements, five pressures (5, 15, 30, 75 and 150 psig) were selected to give a coverage adequate to follow any changes noted. The data from the measurements are given in Tables XII through XVI and presented graphically in Figures 15 through 19. From this information, it is observed that below approximately 1980°C (3600°F) there is no change in strength with pressure independent of the orientation of the specimen. Above this temperature, for the across-grain oriented materials the strength appears to be either unchanged or decreased slightly. The deviation of the data from that obtained with one atmosphere (0 psig) pressure of carbon monoxide or nitrogen is within the measured variation in strength of the material. For the with-grain oriented graphite, the strength appears to increase with pressure above 1980°C. This increased strength prevails to about 2500°C. Above this temperature, there is no measurable change in the strength. A condition very similar to this was also found to occur with outgassing pretreatment within the limits measured.

A comparison of Figures 15 through 19 shows that as the pressure is increased the strength is also increased. This increase, further, seems to be proportional to the logarithm of the pressure. This form of mathematical dependence is suggestive of extended hot working of the specimen during the stressing. The magnitude of the maximum atmospheric stress (150 psig) is inappreciable compared to the 5000 - 10,000 psi tensile stress present in the temperature range involved; thus direct mechanical effects seem to be ruled out. Indirect mechanical effects of pressure on the distribution of dislocations and associated stresses which result in fracture are conceivable. Alternatively, it has been suggested that pressure may suppress vaporization at locations where fracture originates. More data will be required to arrive at the correct interpretation of the effect of atmospheric pressure on the tensile strength of ZTA graphite and to determine whether similar effects are observed with other graphites.

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TABLE XI

Temperature-Strength Data for ZTA Graphite Tested in 1 Atmosphere of Carbon Monoxide Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
1744	31 - 4	1.980	2875	1580	4830
"	31 - 31	1.983	2840	1560	5110
1745	17 - 6	1.978	3360	1850	5220
1744	31 - 30	1.975	3345	1840	5280
"	31 - 34	1.986	3360	1850	5360
1745	19 - 4	1.985	3800	2090	6170
1744	31 - 8	1.985	3805	2095	6000
"	31 - 15	1.985	3820	2105	6260
"	31 - 29	1.990	4520	2495	8400
"	31 - 25	1.979	4530	2500	8970
"	31 - 6	1.983	4560	2515	8695
"	31 - 21	1.978	4610	2545	8210
"	31 - 14	1.972	4730	2610	7800
			Across Grain		
1745	12 - 13	1.962	2860	1570	1430
1744	30 - 7	1.972	2820	1550	1440
1745	16 - 14	1.971	3320	1830	1570
1744	30 - 1	1.979	3345	1840	1560
"	30 - 19	1.978	3330	1835	1560
"	30 - 6	1.973	3780	2080	1710
"	30 - 22	1.972	3795	2090	1680
"	30 - 29	1.983	3795	2090	1750
"	30 - 14	1.981	4470	2465	1590
"	30 - 2	1.981	4485	2475	1740
"	30 - 20	1.976	4555	2510	1780
"	30 - 17	1.981	4570	2520	1780
"	30 - 16	1.981	4670	2575	1590

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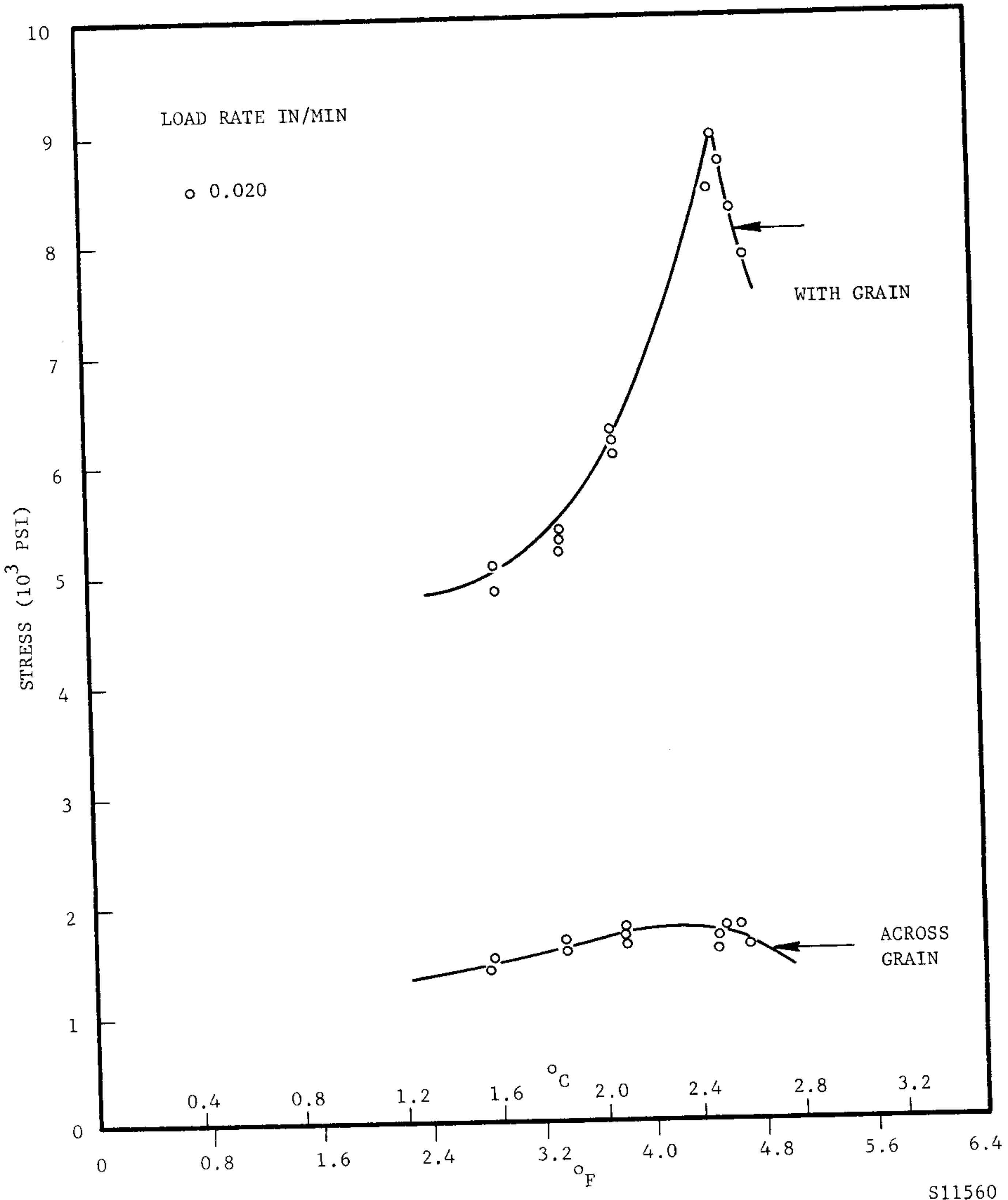

FIGURE 14. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN CARBON MONOXIDE

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TABLE XII

Temperature-Strength Data for ZTA Graphite
Tested in 5 PSIG of Argon
Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature		Ultimate Tensile Strength psi
			°F	°C	
With Grain					
1744	31 - 33	1.984	72	22	4155
"	31 - 27	1.982	2065	1130	4460
"	31 - 22	1.983	2560	1405	4495
"	31 - 16	1.983	2770	1520	4710
1745	21 - 11	1.986	3515	1880	5410
"	19 - 2	1.985	3525	1885	5030*
"	15 - 7	1.972	3525	1885	5480
1744	31 - 10	1.978	3535	1890	4945
1745	15 - 1	1.975	3535	1890	5410
"	13 - 2	1.969	3550	1900	4870*
1744	31 - 13	1.967	3640	2005	5405
"	31 - 28	1.972	3730	2055	5905
1745	19 - 6	1.983	3950	2180	7210
1744	31 - 3	1.980	3975	2190	7300
1745	21 - 2	1.989	3975	2190	6110*
1721	7 - 12	1.983	3990	2200	7420
1744	31 - 5	1.974	4010	2210	7400
1745	9 - 2	1.960	4025	2220	6295*
"	17 - 1	1.976	4060	2240	7600
1744	31 - 17	1.983	4060	2240	7490
"	31 - 11	1.983	4155	2290	7810
"	31 - 7	1.978	4270	2355	8020
"	31 - 43	1.990	4425	2440	8770
"	31 - 57	1.984	4435	2445	8960
"	31 - 45	1.984	4710	2600	7800
"	31 - 50	1.986	4810	2655	7320

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* Load rate: 0.200 in/min

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TABLE XII (Cont'd.)

Temperature-Strength Data for ZTA Graphite
Tested in 5 PSIG of Argon
Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
		Across Grain			
1744	30 - 5	1.976	72	22	1175
"	30 - 30	1.979	72	22	1335
"	30 - 30	1.977	2130	1165	1335
"	30 - 21	1.973	2570	1410	1370
"	30 - 24	1.972	2785	1530	1430
"	30 - 13	1.988	3370	1855	1560
1745	10 - 7	1.960	3515	1935	1465
"	14 - 5	1.970	3515	1935	1240*
"	12 - 6	1.964	3535	1945	1240*
"	12 - 8	1.962	3535	1945	1430
"	14 - 8	1.970	3590	1980	1370
1744	30 - 8	1.976	3900	2150	1560
1745	12 - 10	1.965	3900	2150	1525
"	14 - 11	1.970	3975	2190	1495
1744	30 - 11	1.982	3975	2190	1590
"	30 - 10	1.981	3985	2195	1655
1745	14 - 9	1.968	3985	2195	1370*
"	16 - 13	1.973	3985	2195	1465*
"	12 - 1	1.965	3990	2200	1560
1744	30 - 31	1.977	4030	2220	1715
"	30 - 40	1.979	4425	2440	1655
"	30 - 55	1.978	4425	2440	1815
"	30 - 52	1.978	4755	2625	1785
"	30 - 66	1.979	4800	2650	1620

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* Load rate: 0.200 in/min

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TABLE XIII

Temperature-Strength Data for ZTA Graphite
Tested in 15 PSIG of Argon
Load Rate: 0.020 in/min

ZTA Block 1744

Specimen Number	Density g/cc	Temperature °F	°C	Ultimate Tensile Strength psi
		With Grain		
31 - 23	1.980	72	22	4210
31 - 35	1.984	72	22	4170
31 - 37	1.990	2650	1455	4710
31 - 20	1.980	2740	1505	4930
31 - 59	1.970	3460	1905	5130
31 - 63	1.977	3610	1990	5405
31 - 36	1.990	3930	2165	7640
31 - 71	2.000	4080	2250	7740
31 - 52	2.000	4450	2455	9380
31 - 58	1.980	4560	2515	9600
31 - 69	1.995	4800	2650	7570
31 - 54	1.991	4810	2655	7160
		Across Grain		
30 - 50	1.978	72	22	1400
30 - 68	1.981	72	22	1335
30 - 54	1.978	2715	1490	1365
30 - 57	1.982	2850	1565	1400
30 - 60	1.975	3550	1955	1495
30 - 53	1.979	3590	1980	1620
30 - 64	1.978	4030	2220	1655
30 - 42	1.981	4065	2240	1780
30 - 59	1.978	4360	2405	1620
30 - 56	1.981	4415	2435	1790
30 - 65	1.978	4740	2615	1845
30 - 62	1.976	4785	2640	1790

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TABLE XIV

Temperature-Strength Data for ZTA Graphite
Tested in 30 PSIG of Argon
Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
1744	31 - 48	1.980	72	22	4155
"	31 - 68	1.998	72	22	4270
"	31 - 53	1.990	2680	1470	4930
"	31 - 46	1.985	2785	1530	5320
"	31 - 49	1.988	3460	1905	5680
"	31 - 66	1.993	3560	1960	5680
"	31 - 64	1.990	3900	2150	7300
"	31 - 74	1.979	4100	2260	8380
1746	33 - 22	1.964	4370	2410	9320
"	33 - 28	1.963	4425	2440	9640
"	33 - 27	1.963	4730	2610	7860
1744	31 - 62	1.992	4820	2660	7250
			Across Grain		
1744	30 - 43	1.979	72	22	1335
"	30 - 49	1.976	72	22	1305
"	30 - 34	1.976	2725	1495	1440
"	30 - 32	1.978	2820	1550	1465
"	30 - 44	1.988	3590	1975	1715
"	30 - 51	1.979	3590	1975	1685
"	30 - 58	1.983	3850	2180	1810
"	30 - 38	1.979	3990	2200	1750
"	30 - 47	1.976	4390	2420	1620
"	30 - 67	1.986	4425	2440	1790
"	30 - 37	1.977	4795	2645	1720
"	30 - 61	1.977	4800	2650	1685

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TABLE XV

Temperature-Strength Data for ZTA Graphite Tested in 75 PSIG of Argon Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
1744	31 - 72	2.000	72	22	4260
"	31 - 73	1.998	72	22	4150
"	31 - 40	1.975	2595	1425	4840
"	31 - 51	1.975	2615	1435	4750
"	31 - 65	1.991	3405	1875	5510
"	31 - 39	1.990	3570	1965	5760
"	31 - 47	1.972	3570	1965	5570
"	31 - 56	1.982	3885	2140	7000
"	31 - 55	1.978	3995	2200	7420
1746	33 - 8	1.977	4045	2230	8180
"	33 - 10	1.970	4090	2255	8500
"	33 - 21	1.972	4245	2340	8970
"	33 - 26	1.974	4360	2405	9410
"	33 - 23	1.951	4395	2425	9680
"	33 - 12	1.969	4470	2465	10040
"	33 - 31	1.948	4470	2465	10180
			Across Grain		
1744	30 - 35	1.977	72	22	1495
"	30 - 41	1.981	72	22	1340
"	30 - 46	1.973	2610	1435	1590
"	30 - 63	1.978	2685	1475	1720
"	30 - 39	1.976	3535	1890	1815
"	30 - 33	1.978	3550	1955	1845
"	30 - 48	1.976	3850	2120	1875
"	30 - 36	1.982	3920	2160	1940
"	30 -104	1.980	4010	2210	1980
"	30 - 97	1.979	4030	2220	1920
"	30 - 85	1.966	4190	2310	2020
"	30 - 90	1.965	4450	2455	2020
"	30 - 89	1.968	4475	2475	2130

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TABLE XVI

Temperature-Strength Data for ZTA Graphite
Tested in 150 PSIG of Argon
Load Rate: 0.020 in/min

Block No.	Specimen Number	Density g/cc	Temperature °F	Temperature °C	Ultimate Tensile Strength psi
			With Grain		
1744	31 - 61	1.996	72	22	4260
"	31 - 67	1.991	72	22	4260
"	31 - 38	1.975	2625	1440	4870
"	31 - 42	1.990	2660	1460	4990
"	31 - 60	1.985	3345	1840	5520
"	31 - 41	1.985	3440	1895	5780
1746	33 - 3	1.954	3705	2040	7010
"	33 - 7	1.970	3705	2040	6940
"	33 - 6	1.959	3940	2170	8430
"	33 - 19	1.970	4345	2395	9850
"	33 - 29	1.970	4345	2395	9990
"	33 - 2	1.973	4380	2415	10240

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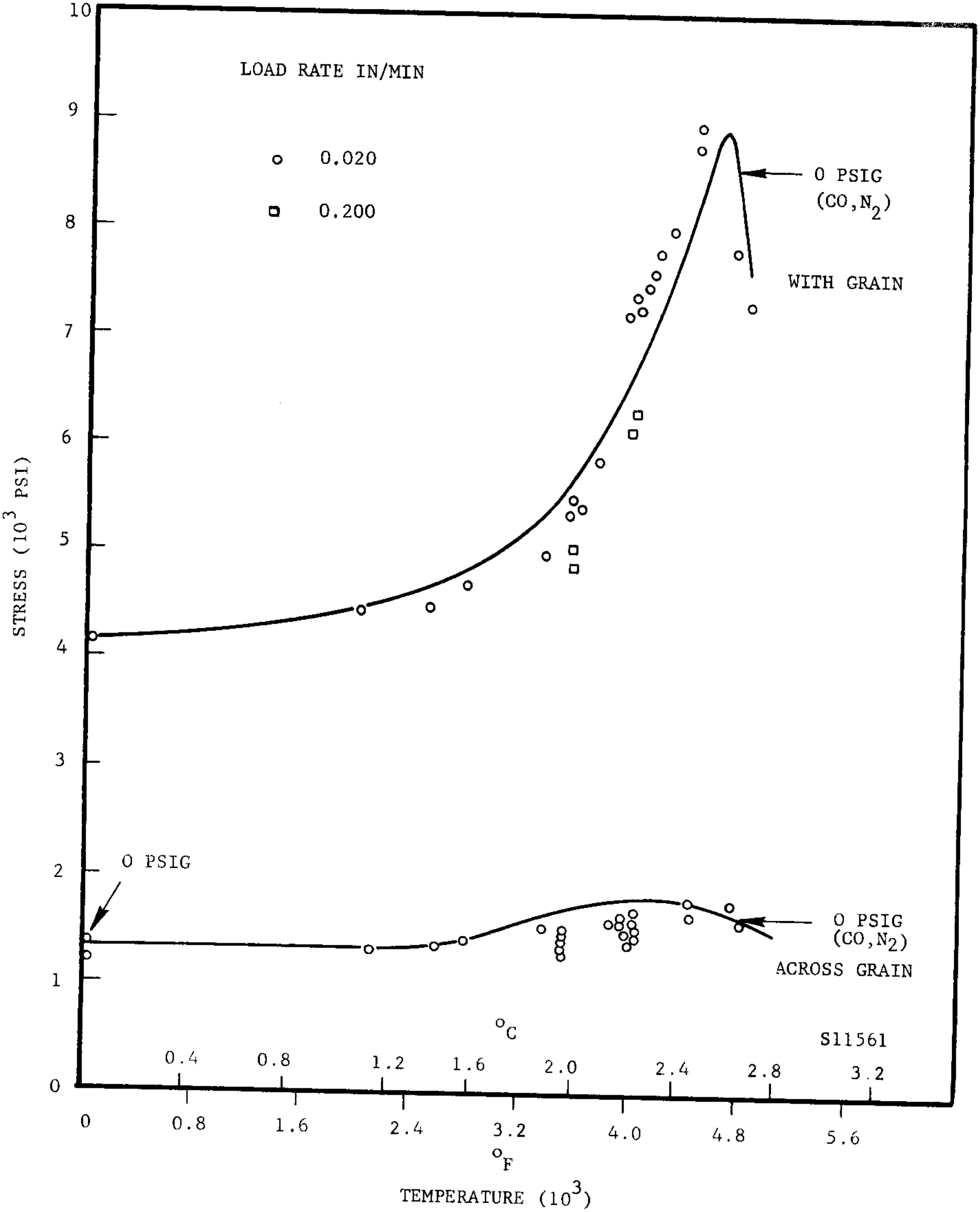

FIGURE 15. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN 5 PSIG ARGON

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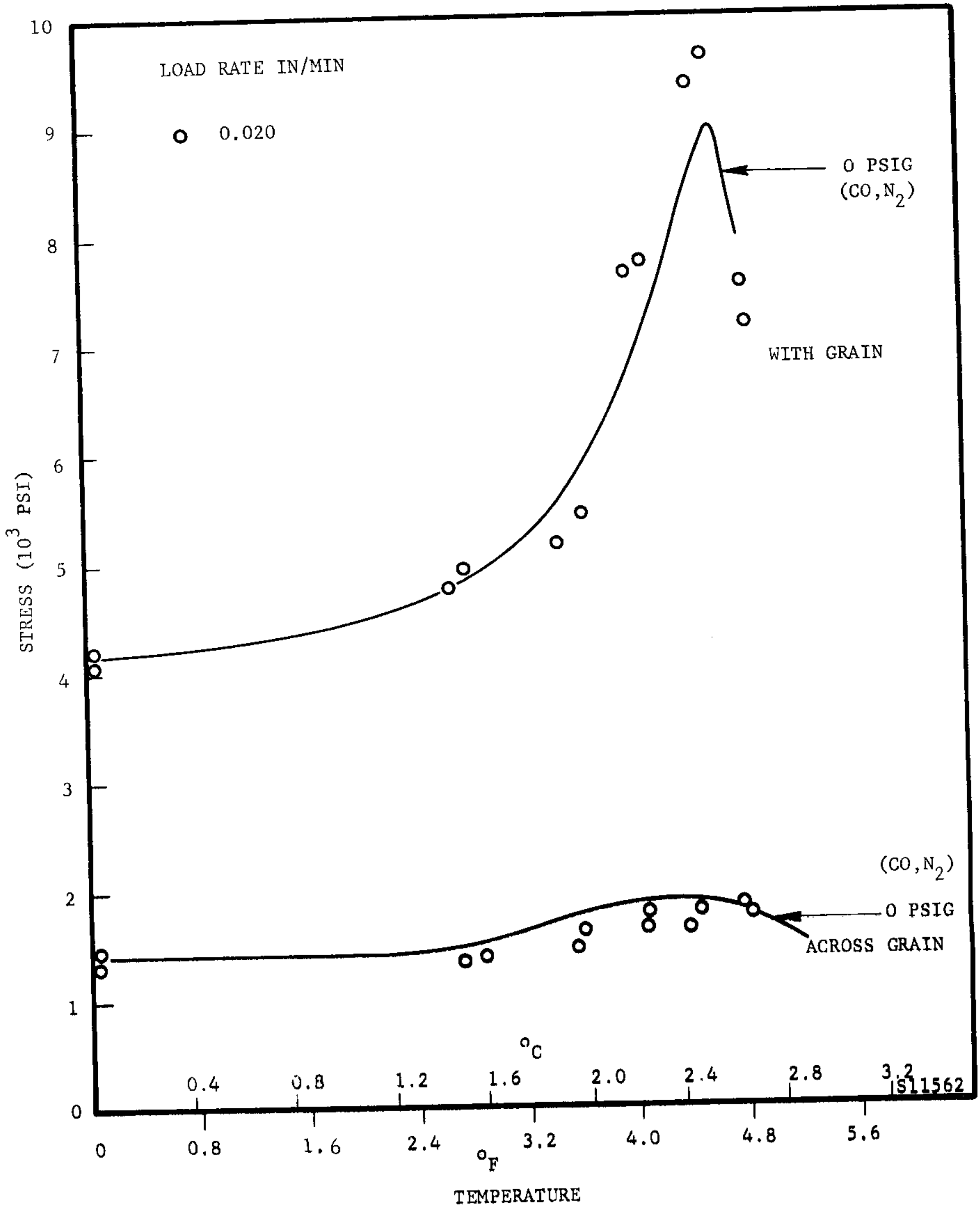

FIGURE 16. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN 15 PSIG ARGON

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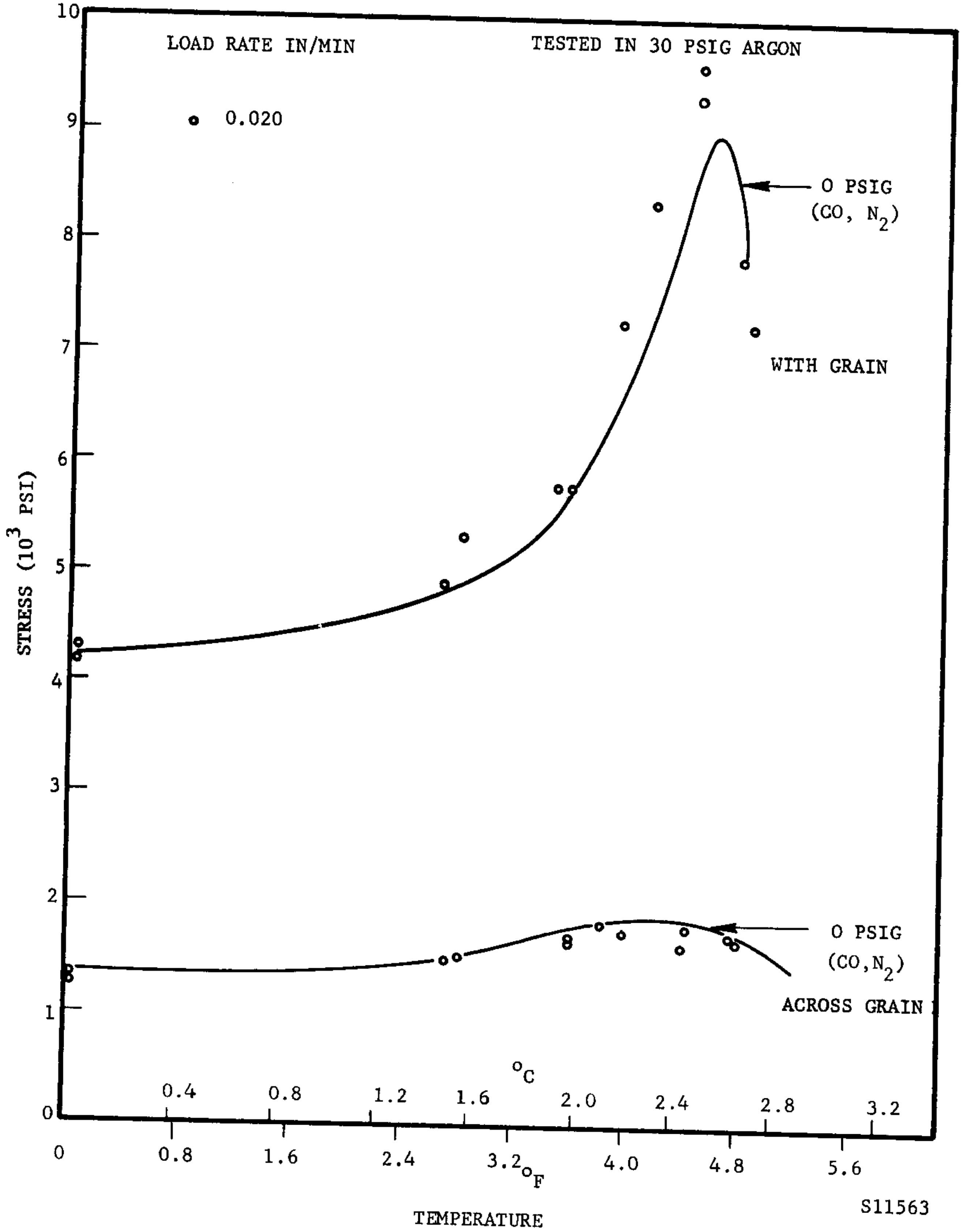

FIGURE 17. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN 30 PSIG ARGON

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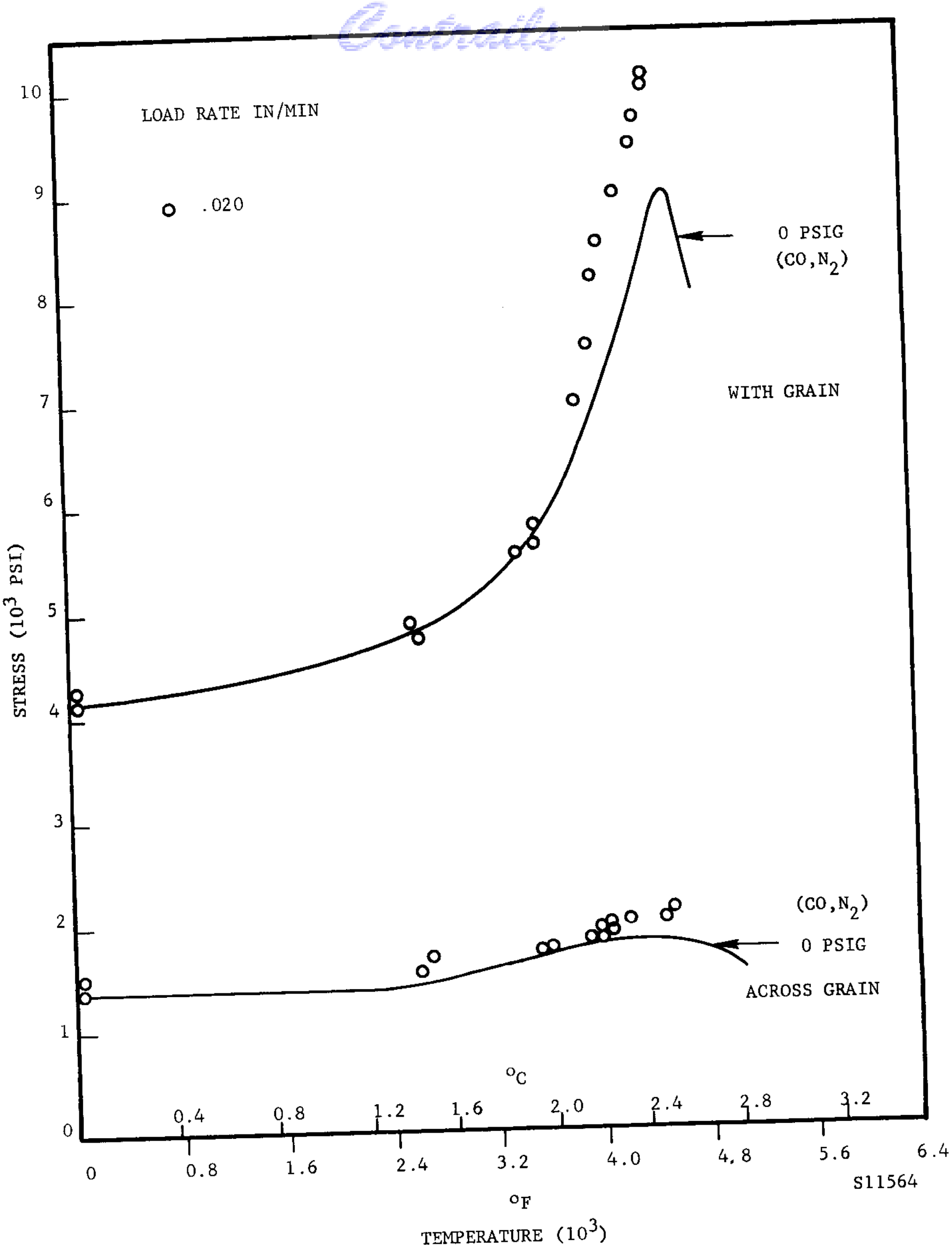

FIGURE 18. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN 75 PSIG ARGON

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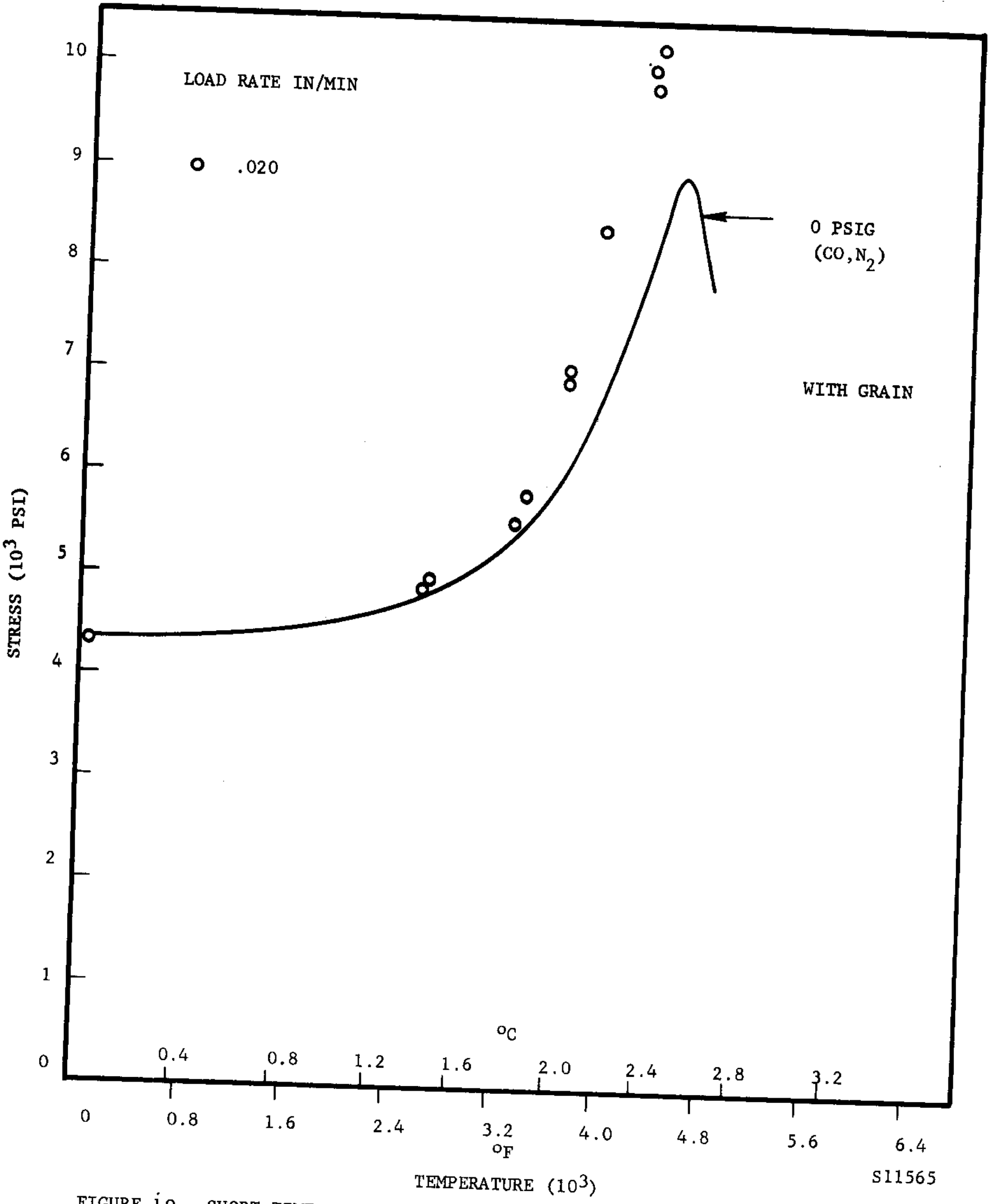

FIGURE 19. SHORT TIME TENSILE STRENGTH VERSUS TEMPERATURE FOR ZTA GRAPHITE TESTED IN 150 PSIG ARGON

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5. SUPPLEMENTAL MEASUREMENTS

In the preceding section, the ultimate strength of ZTA graphite as a function of temperature was detailed for various test conditions. Supplementing these data are the stress-strain curves of individual specimens and electron micrographs of the fracture surface. Included in this report is a typical sampling of voluminous amounts of results from these measurements, along with a critical examination of these results.

5.1. Stress - Strain Curves

Corresponding to each datum value on the ultimate tensile strength diagrams, the stress-strain data of that specimen being tested were obtained. The strain data were limited to those specimens tested at temperatures generally in excess of 1800°C (3300°F), since below this temperature the contrast between the specimen and its background was insufficient to obtain a clear image. This situation was particularly true when the heat shields were rotated to give a minimum opening in order to gain the maximum temperature. A second restriction on strain measurements was imposed by the nitrogen atmosphere and by pressurized argon. In these instances, the gas, because of turbulence, acted as a constantly, nonuniformly varying lens. The net result was an unpredictable variation of the displacement of the fiducial mark images relative to each other.

Typical stress-strain data obtained are presented in Figures 20 through 26. The strain rate was 0.020/min except where noted otherwise. The slope of the stress vs. strain curves gives an indication of the variation in elastic modulus with strain. The general shape of the curves shows an increase in slope (and elastic modulus) as the strain increases. Some of the specimens show a third stage region showing a markedly decreased slope over an extended strain interval before fracture. The observation from Figures 24, 25 and 26 that this third stage effect is greater at the low load rates suggests that it is associated with creep phenomena.(7)

Figure 27 shows the strain at fracture data for Block 1714 samples plotted vs. the temperature. Insufficient data are available to clearly separate the effects of temperature and tensile strength variation. However, the following observations seem valid:

1. The fracture strain (as well as the tensile strength) for the with-grain orientation increases markedly with increasing temperature; the cross-grain trend is less marked but suggests that the weaker specimens show greater strain at fracture.
2. The fracture strain for the with-grain orientation appears to be decreased by annealing, but the reverse is indicated for the cross-grain orientation.
3. The fracture strains at elevated temperatures are markedly greater for the with-grain than for the across-grain orientation.

The larger fracture strain observed in the with-grain direction is opposite to the high temperature creep behavior of ZTA graphite where the cross-grain creep has been found to be much the greater.(7, 8)

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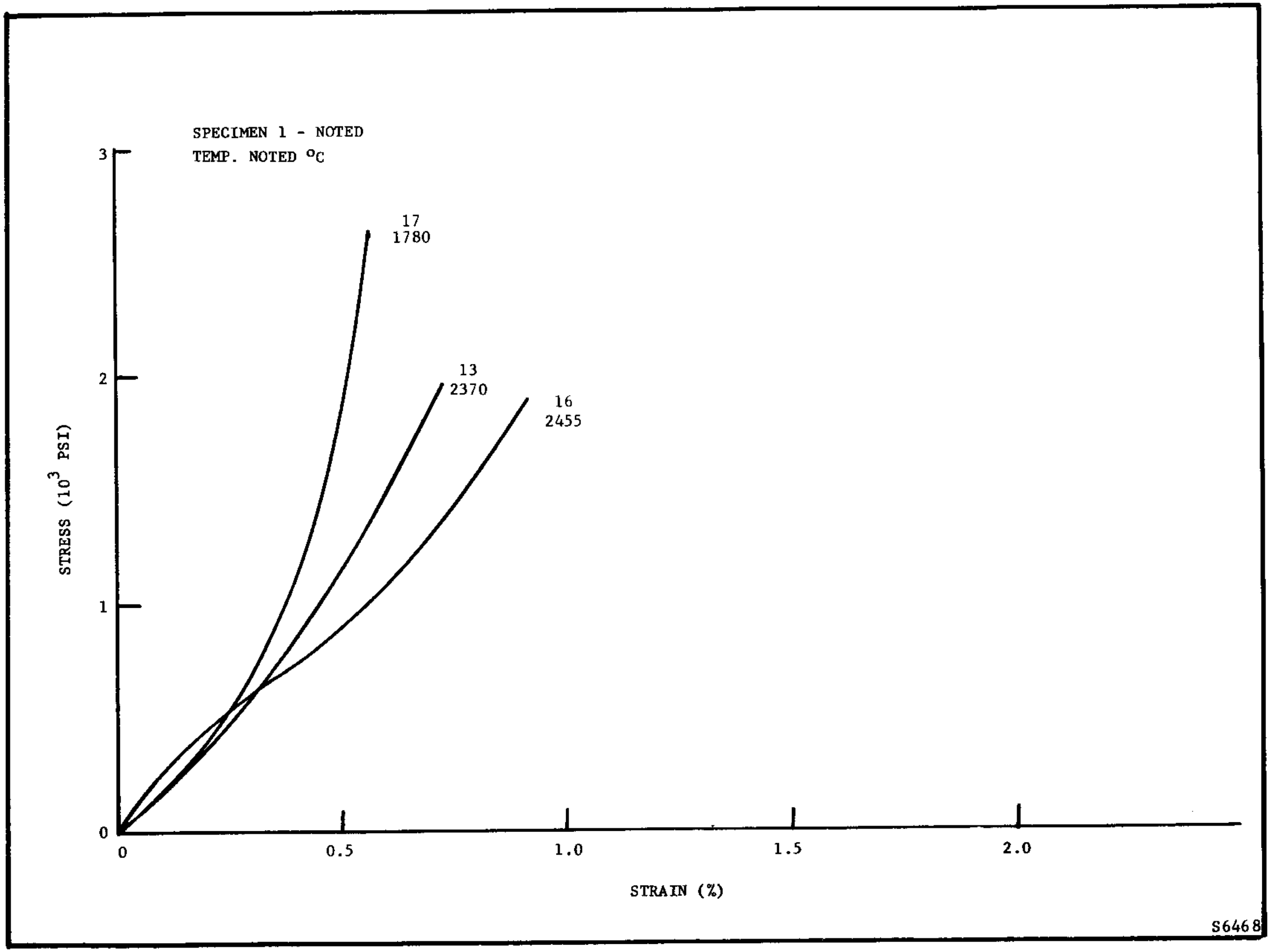

FIGURE 20. STRESS-STRAIN CURVES FOR ZTA GRAPHITE ORIENTED CROSS GRAIN

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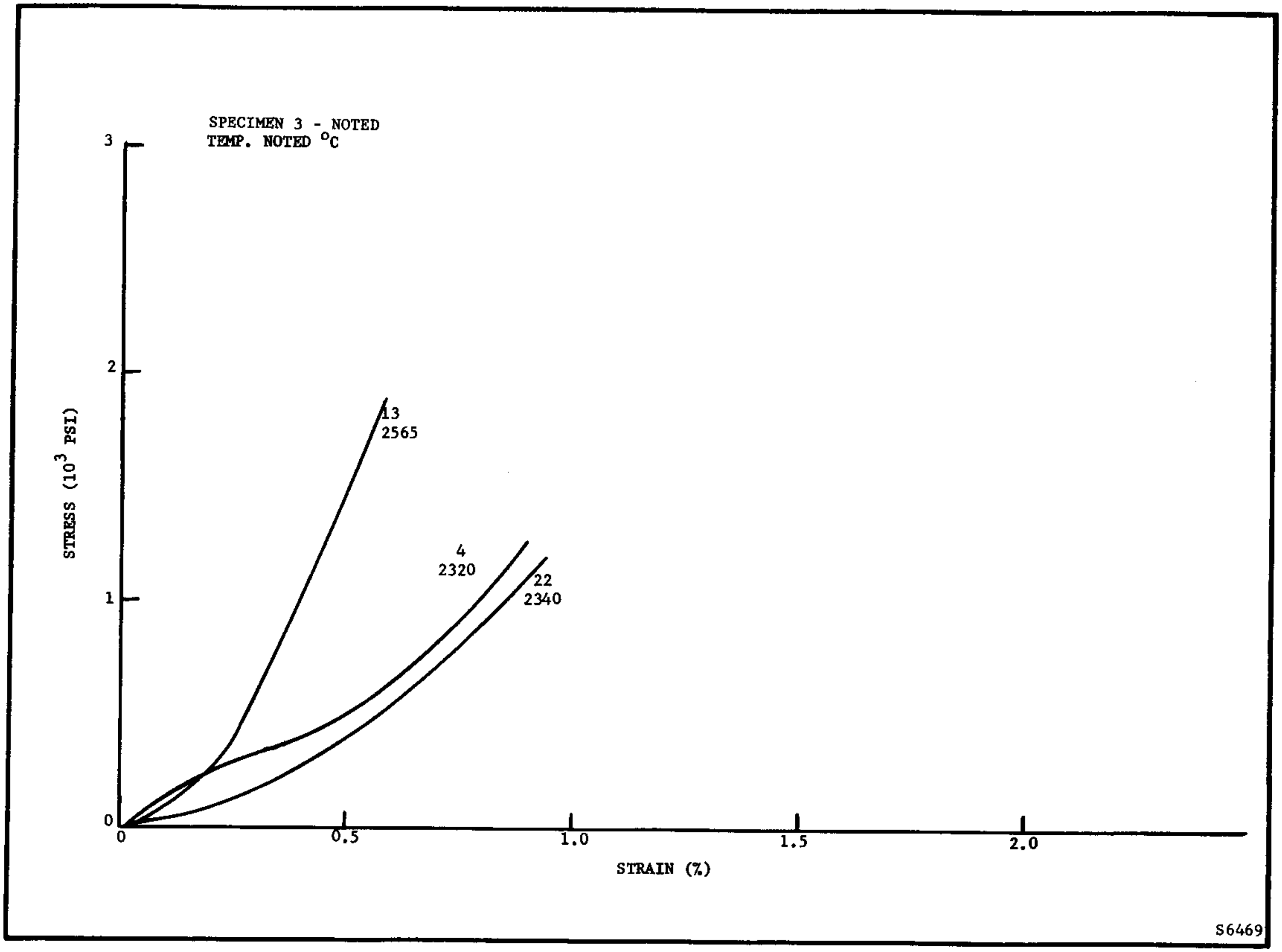

FIGURE 21. STRESS-STRAIN CURVES FOR 3000°C ANNEALED ZTA GRAPHITE ORIENTED CROSS GRAIN

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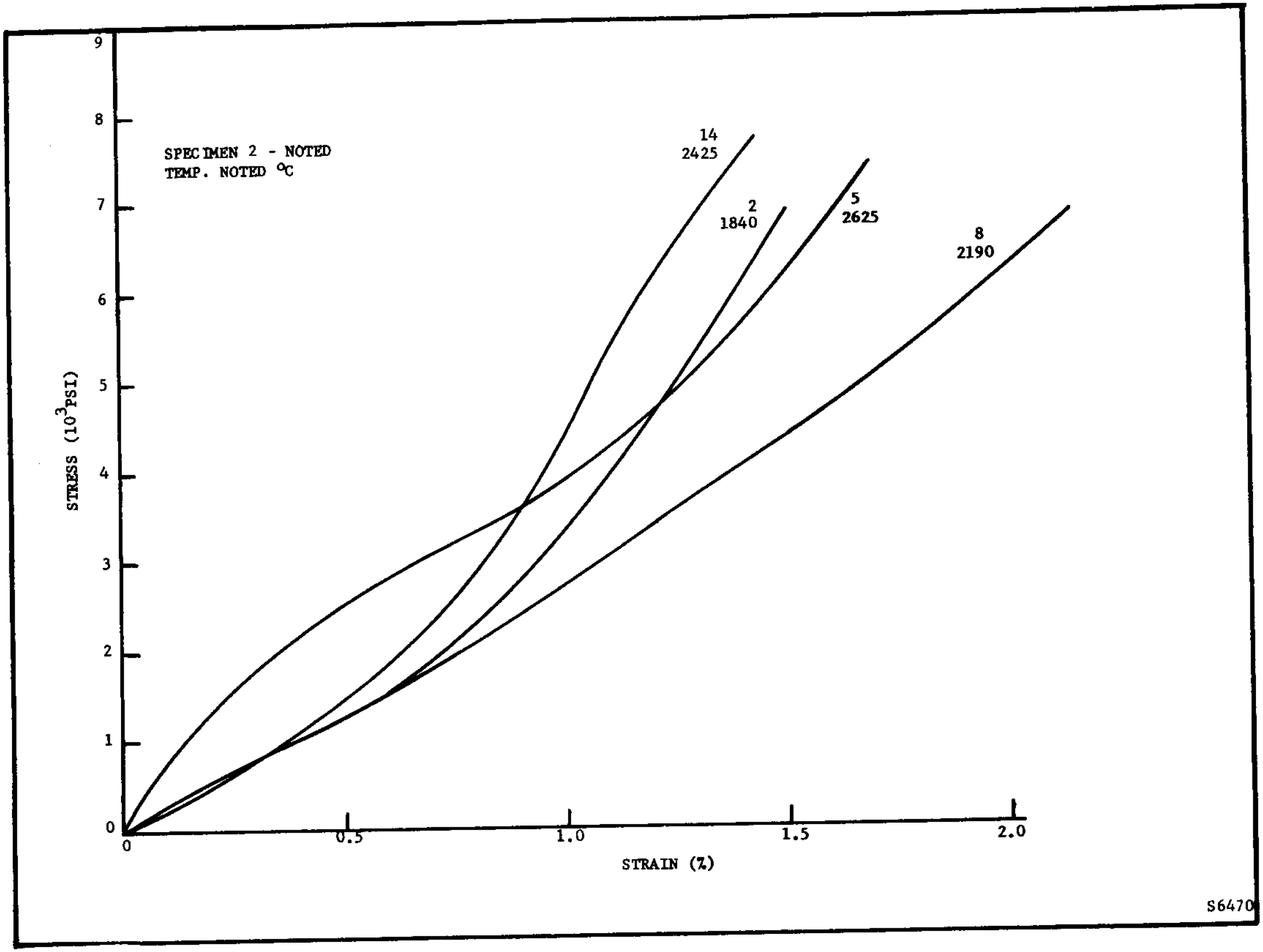

FIGURE 22. STRESS-STRAIN CURVES FOR ZTA GRAPHITE ORIENTED WITH GRAIN

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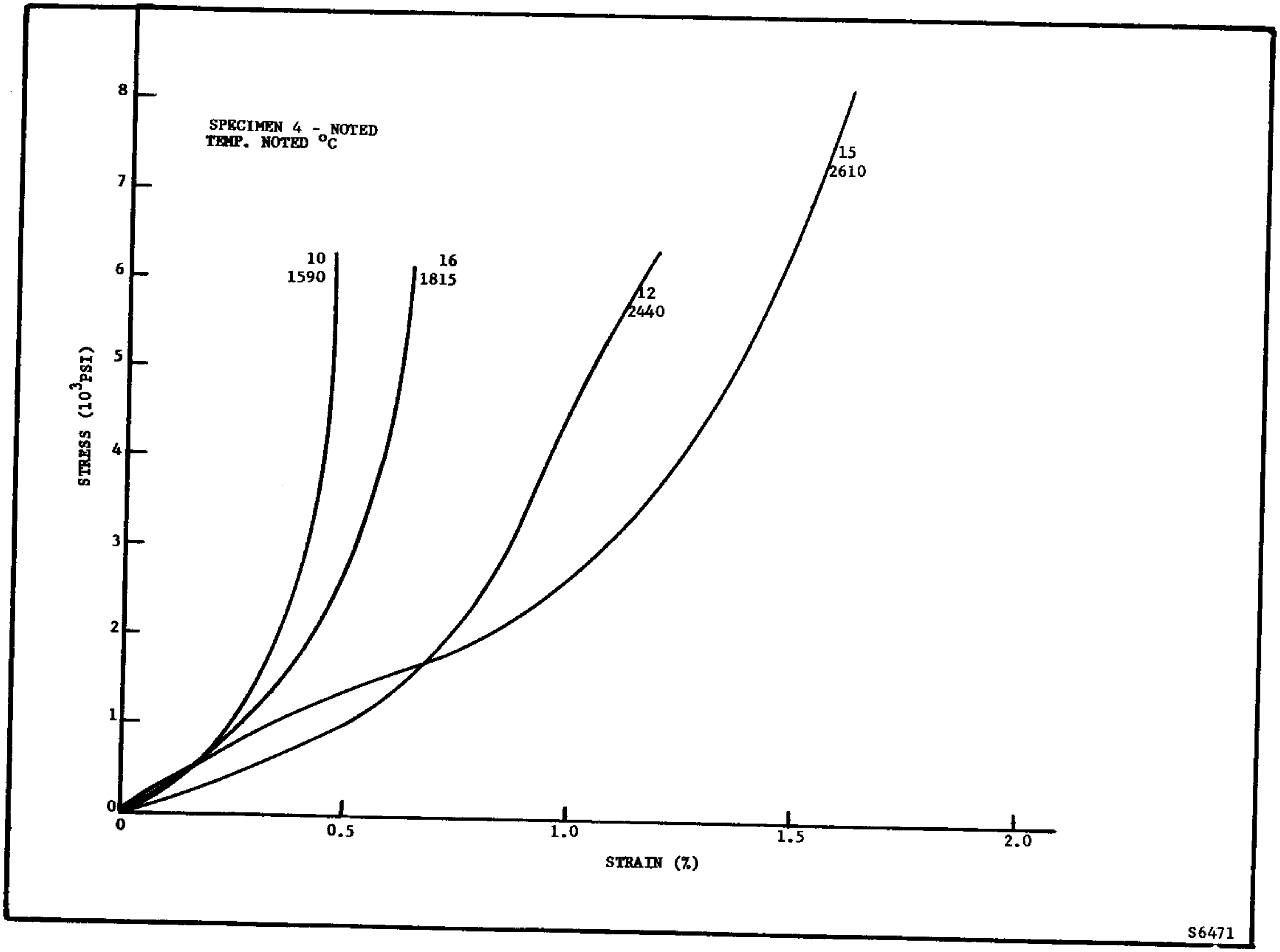

FIGURE 23. STRESS-STRAIN CURVES FOR 3000°C ANNEALED ZTA GRAPHITE ORIENTED WITH GRAIN

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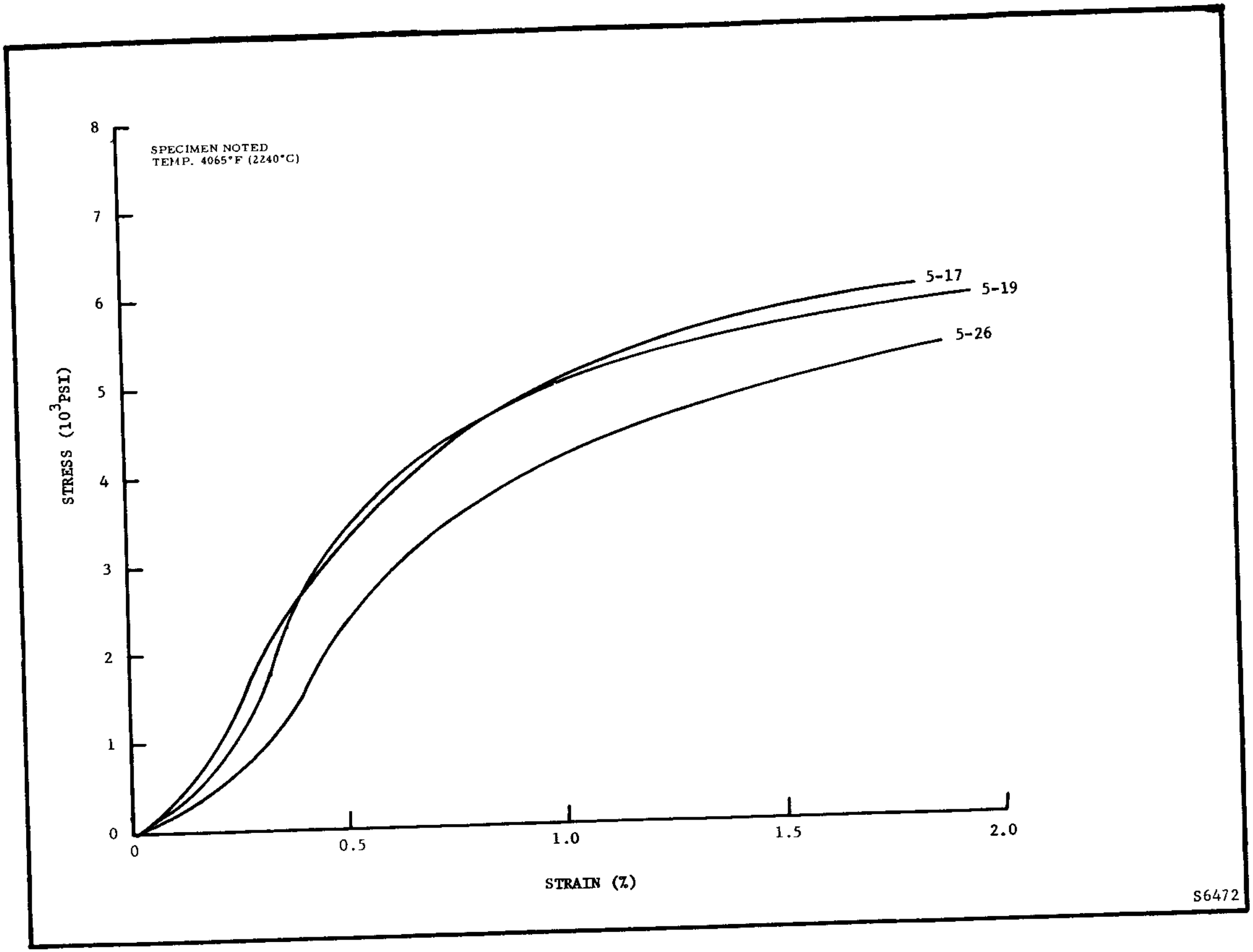

FIGURE 24. STRESS-STRAIN CURVES FOR ZTA GRAPHITE ORIENTED WITH GRAIN .002 INCH/MIN LOAD RATE

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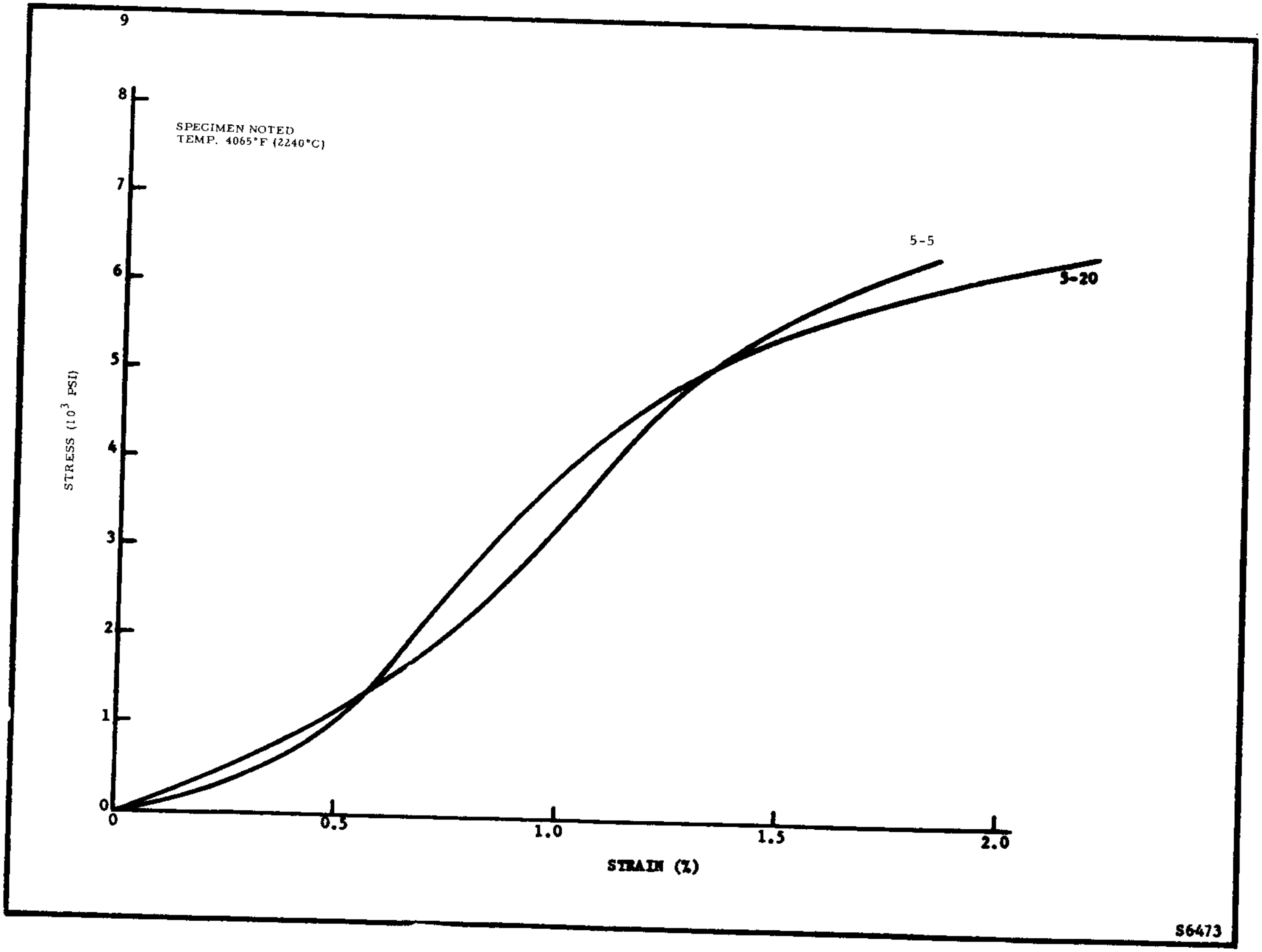

FIGURE 25 STRESS-STRAIN CURVES FOR ZTA GRAPHITE ORIENTED WITH GRAIN 0.020 INCH/MIN LOAD RATE

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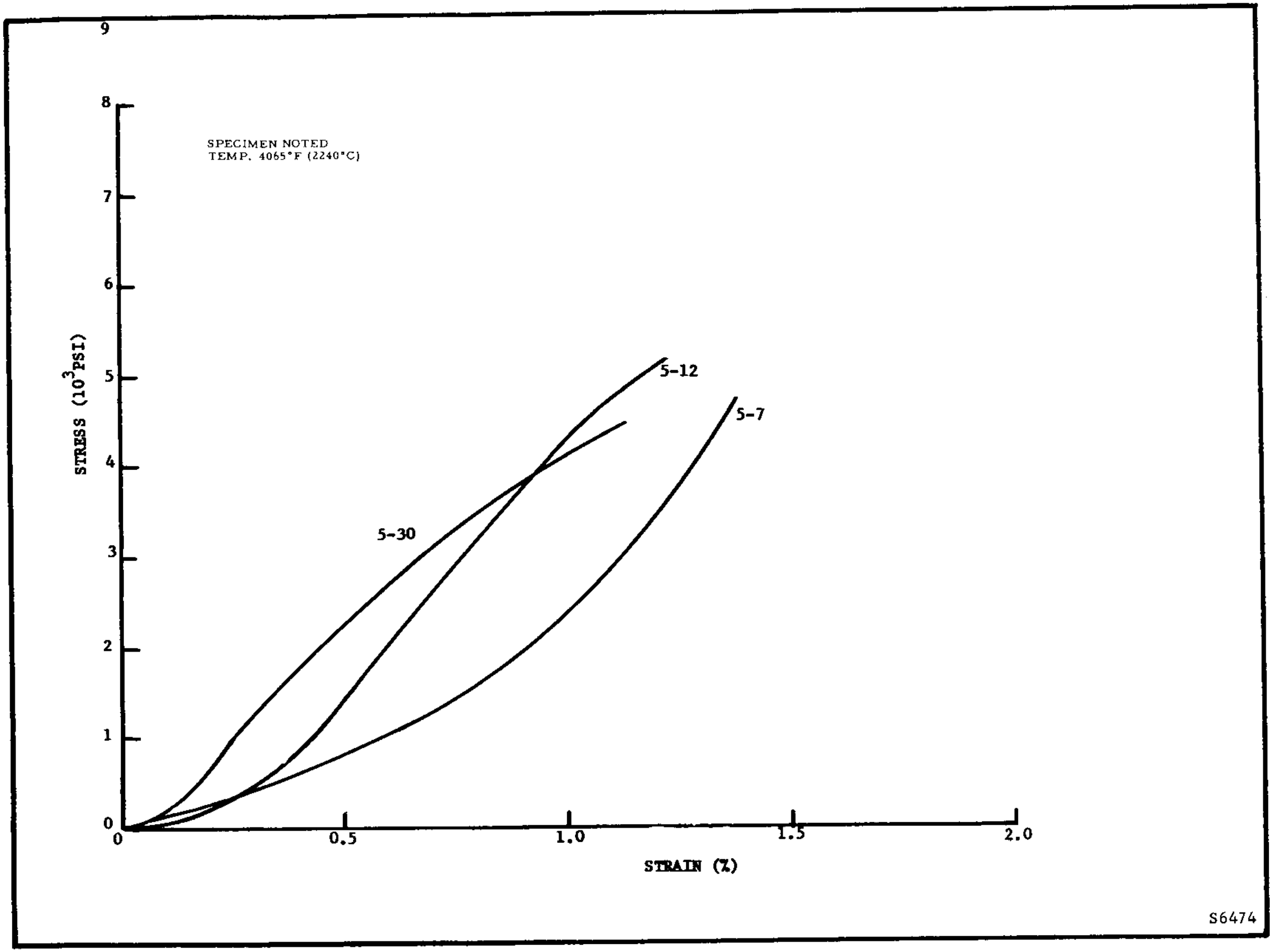

FIGURE 26. STRESS-STRAIN CURVES FOR ZTA GRAPHITE ORIENTED 0.200 INCH/MIN LOAD RATE

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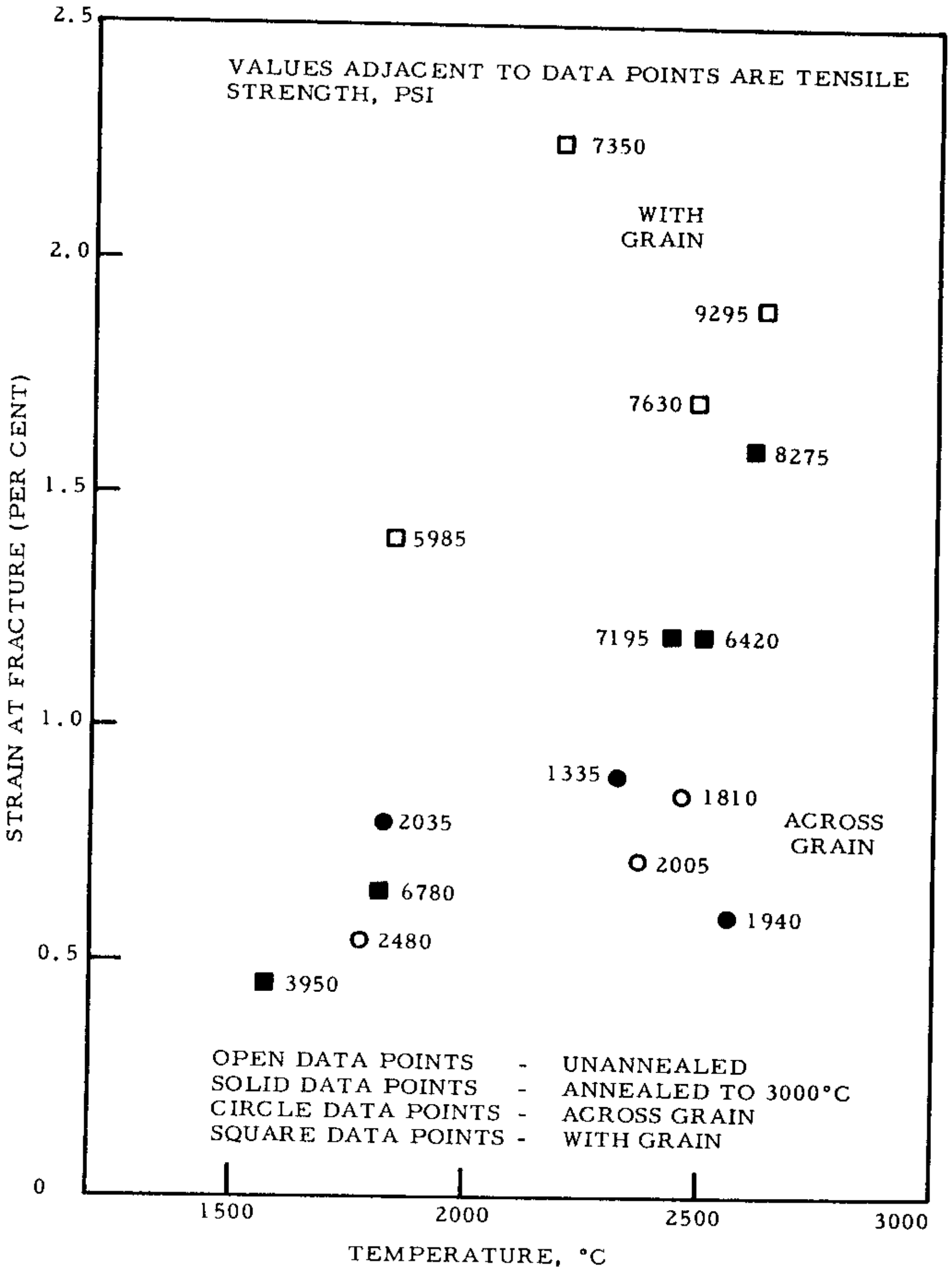

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FIGURE 27. STRAIN AT FRACTURE FOR ZTA GRAPHITE BLOCK 1714 VERSUS TEMPERATURE

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5.2. Electron Micrographs

During the early portion of work reported in this document, electron microscope studies of the fracture surfaces were conducted. Figures 28 through 35 show some typical micrographs obtained from replicas prepared as follows:

(1) Dry a solution of 10 per cent nitrocellulose in amyl acetate on the fracture surface.

(2) Peel the coating from the surface and coat at 45 degrees with chromium.

(3) Recoat at 90 degrees with carbon.

(4) Place the replica on a copper microscope screen and dissolve out the nitrocellulose with amyl acetate.

It should be recognized that some of the details of the micrographs may reflect the method whereby the replicas were obtained, and may not be truly representative of the fracture surface.

All breaks that have been observed can be classified in two general areas:

(1) Moderately smooth and oriented near normal to the stress axis.

(2) Rough and irregular and oriented 60 to 80 degrees from the stress axis.

The first category is typical of all across-grain oriented specimens. This break seems to be principally grain boundary separation with little if any grain fracture. The latter category is representative of many with-grain oriented specimens. Here, as noted in the micrographs (see Figure 33b) the break does sometimes occur in the grain as indicated by the layered fracture. Since the areas adjacent to this layered type break show extreme irregularity (see Figure 33a), what appears to happen is a general grain fracture that propagates to a boundary. At the boundary the fracture surface moves up or down seeking the following surface of least resistance. The trend in this movement is grossly in one preferred direction. This direction might be partially explained by the conchoidal nature of many of the fractures. Unfortunately, the lower magnifications of the electron microscope tend to lose the detail needed to identify the conchoidal nature, while the magnification used limited the field of vision to the extent that the region of interest cannot be photographed.

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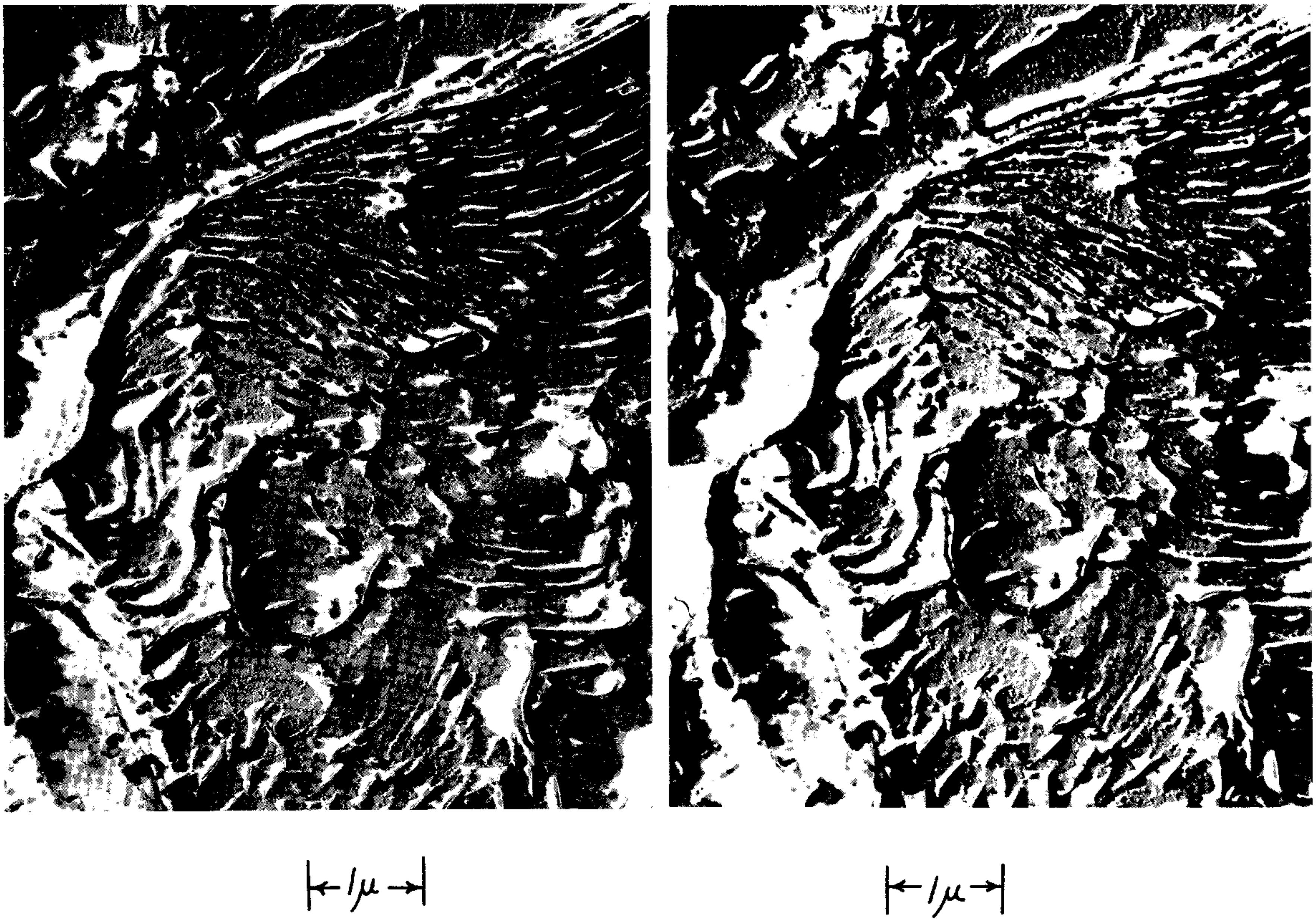

FIGURE 28. FRACTURE SURFACE - SPECIMEN 3-13, 3000°C ANNEALED AND ORIENTED ACROSS GRAIN. STEREO PAIR OF BREAK AT 2565°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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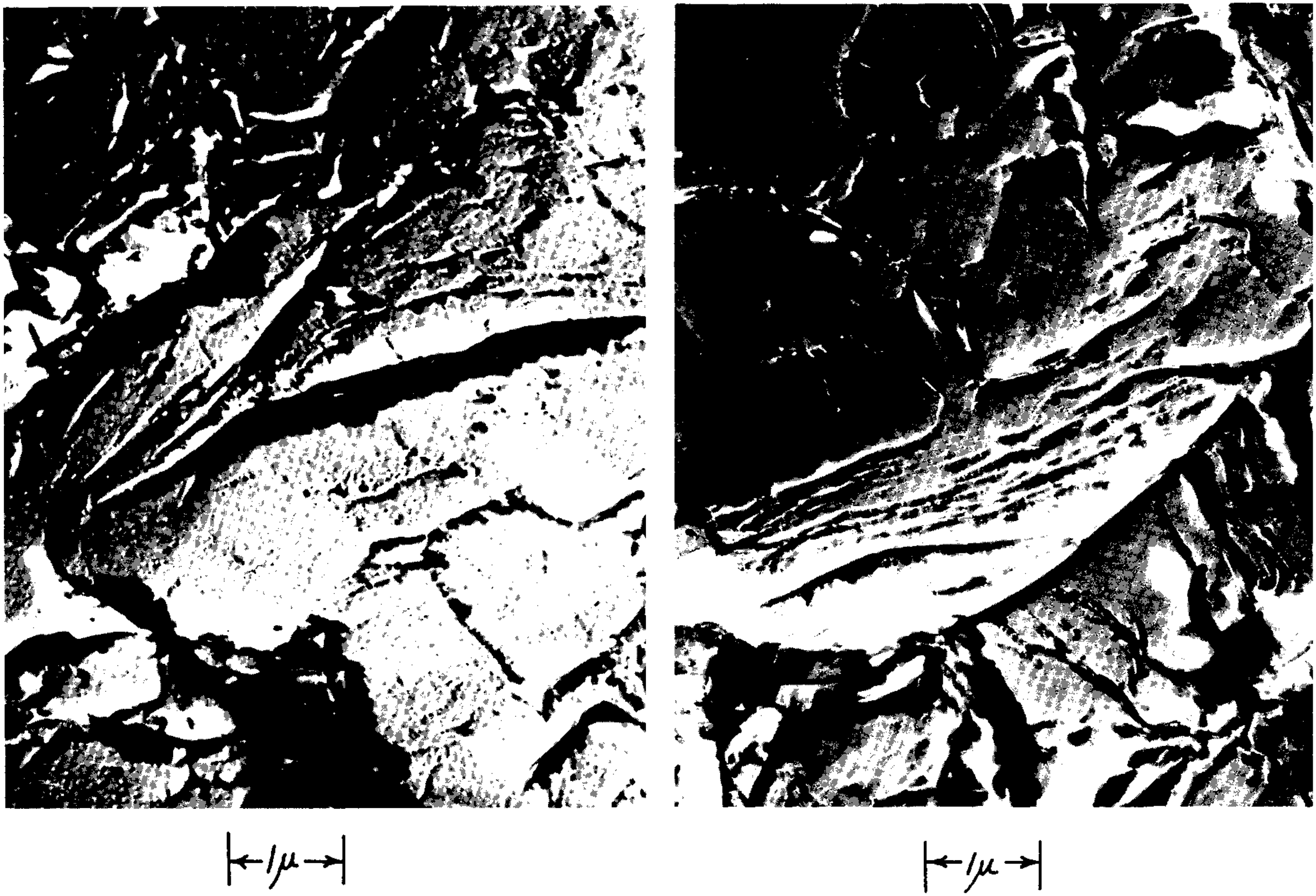

(a) (b)

FIGURE 29. FRACTURE SURFACES - SPECIMEN 3-13, 3000°C ANNEALED AND ORIENTED ACROSS GRAIN. BROKEN AT 2565°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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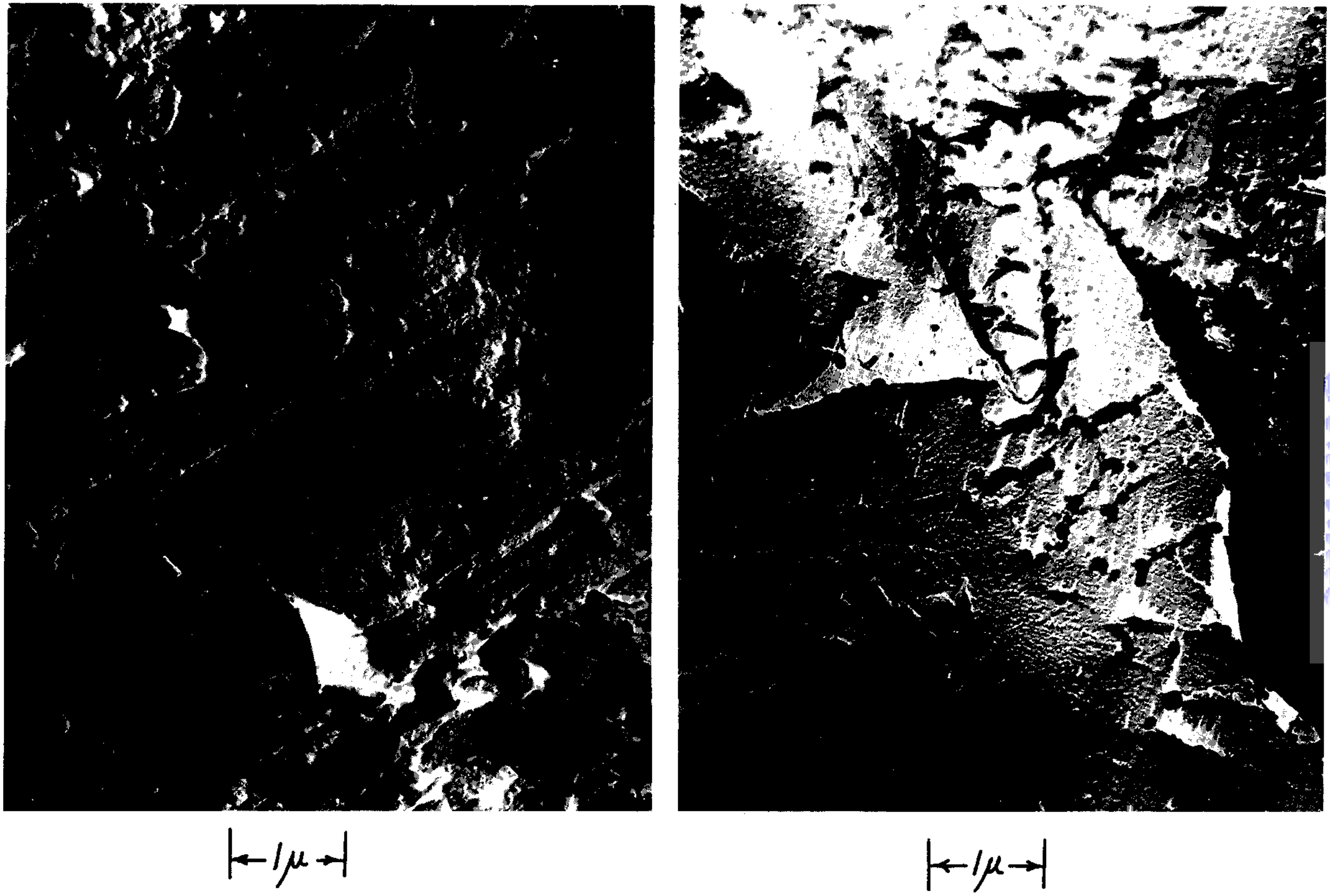

(a)

(b)

FIGURE 30. FRACTURE SURFACES - ORIENTED ACROSS GRAIN. (a) SPECIMEN 3-13, 3000°C ANNEAL BROKEN AT 2565°C. (b) SPECIMEN 1-20 BROKEN AT 2455°C - BOTH IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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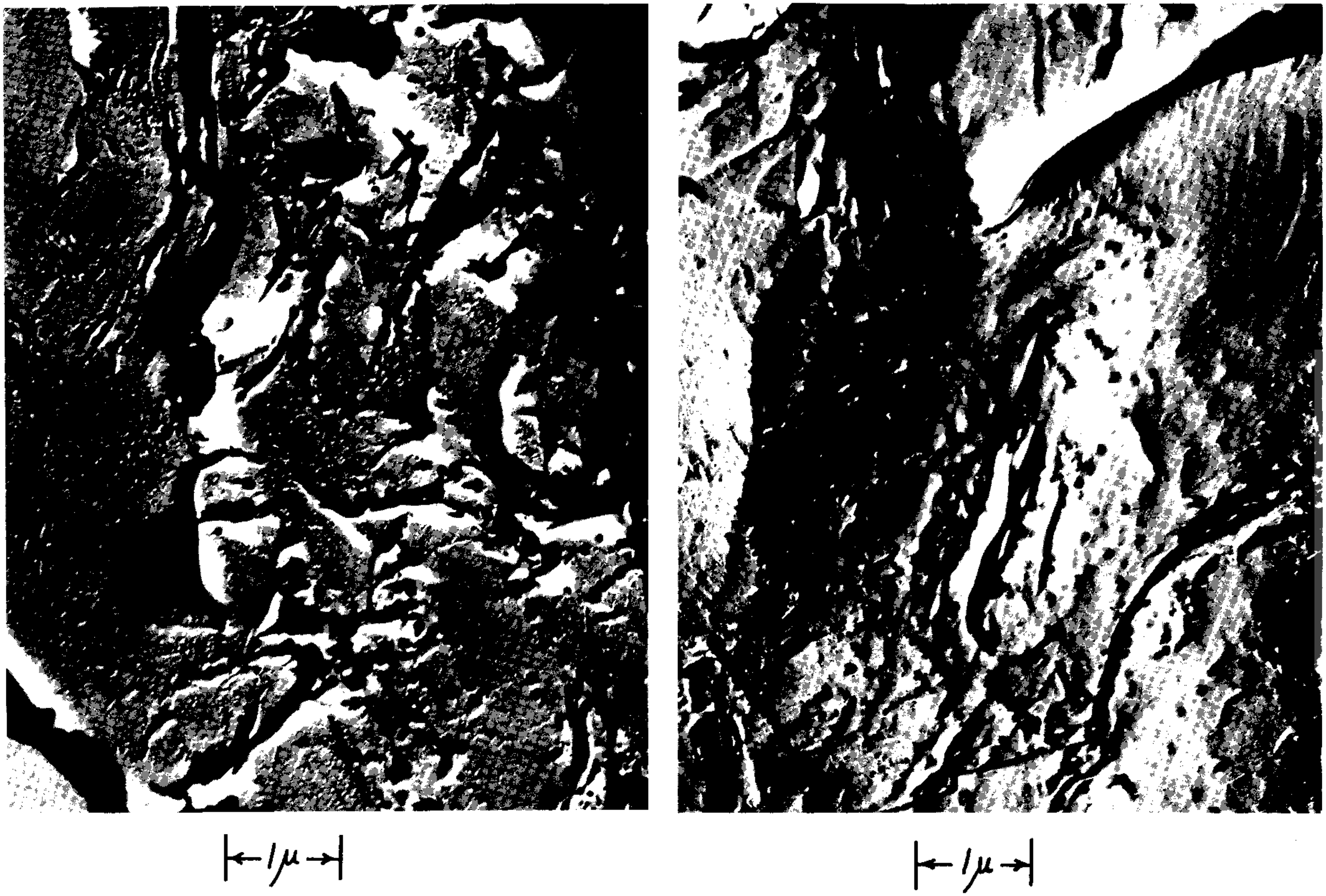

(a)

(b)

FIGURE 31. FRACTURE SURFACES - SPECIMEN 1-20 ORIENTED ACROSS GRAIN. BROKEN AT 2455°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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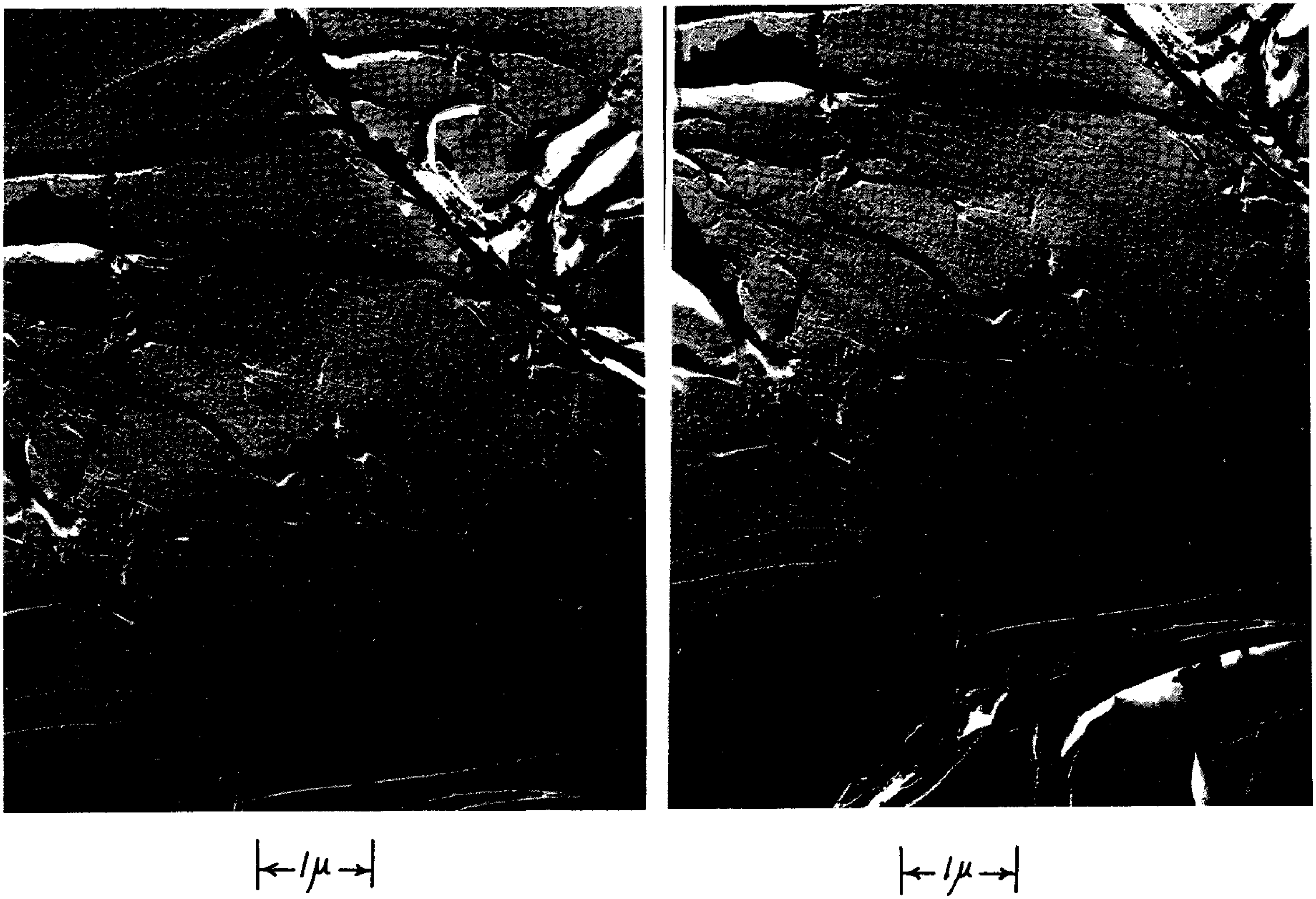

FIGURE 32. FRACTURE SURFACES - SPECIMEN 4-15, 3000°C ANNEALED AND ORIENTED WITH GRAIN. STEREO PAIR OF BREAK AT 2610°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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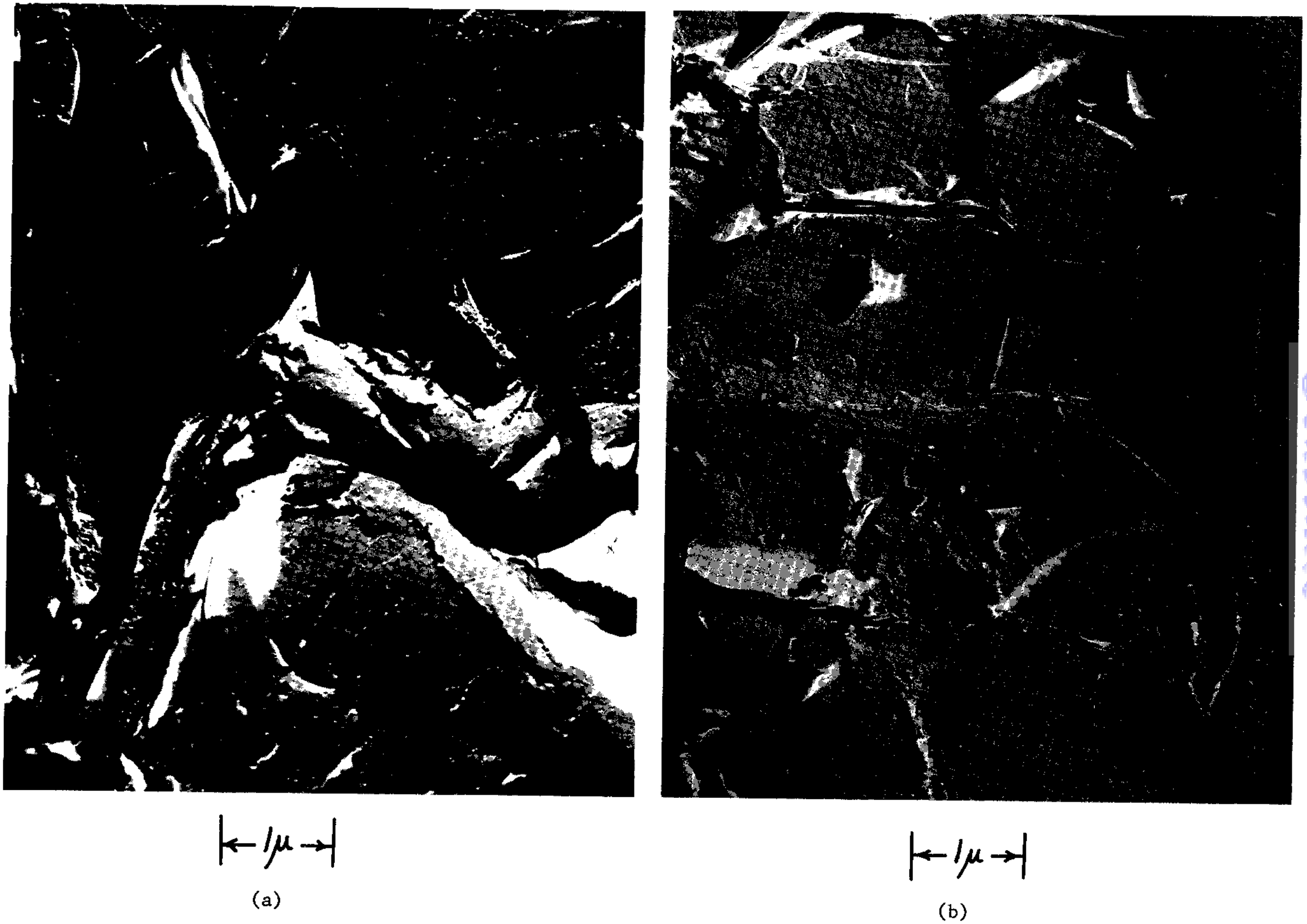

(a)

(b)

FIGURE 33. FRACTURE SURFACES - SPECIMEN 4-15, 3000°C ANNEALED AND ORIENTED WITH GRAIN. BROKEN AT 2610°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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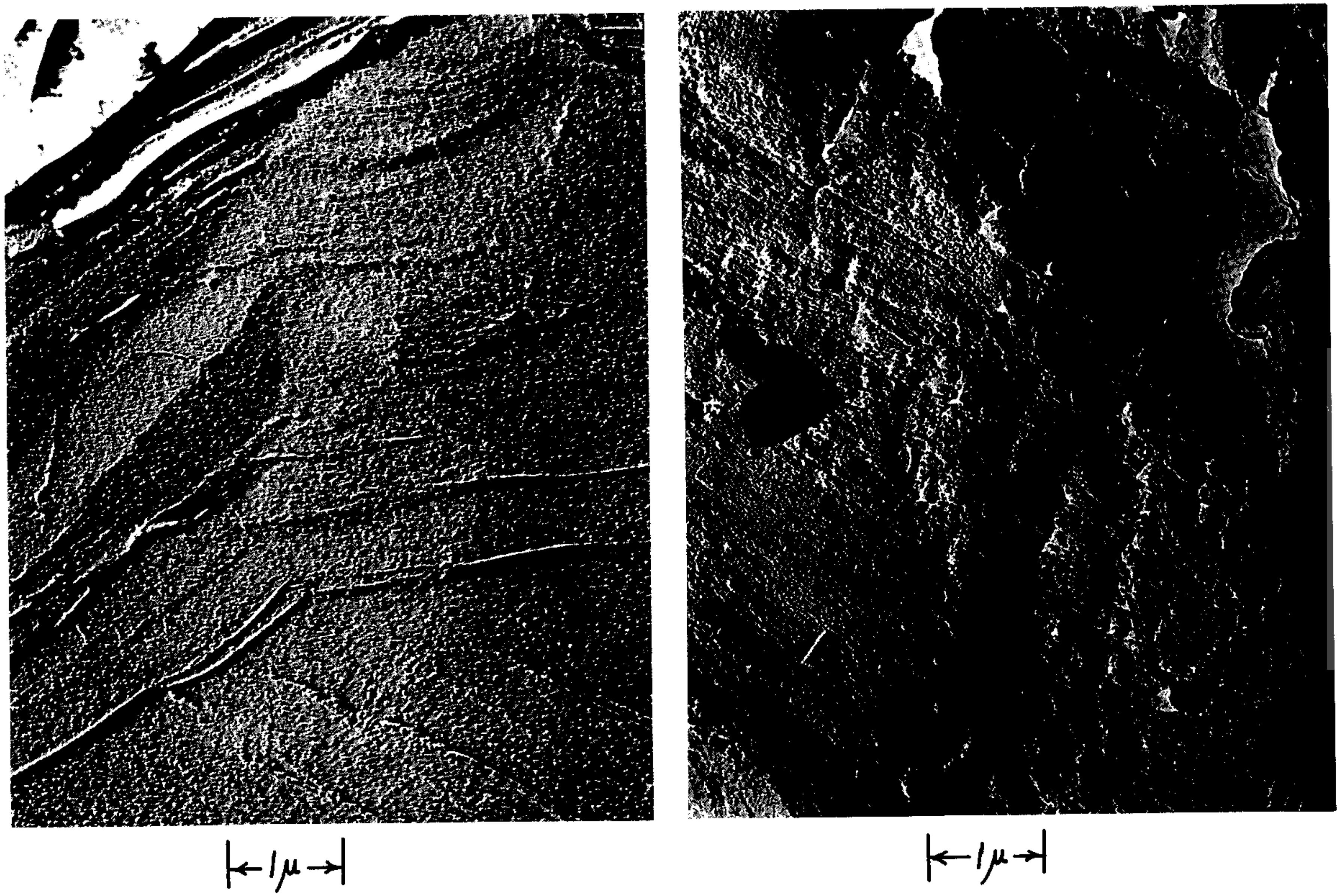

(a)

(b)

FIGURE 34. FRACTURE SURFACES - ORIENTED WITH GRAIN. (a) SPECIMEN 4-15, 3000°C ANNEALED AND BROKEN AT 2610°C. (b) SPECIMEN 2-21 BROKEN AT 2455°C - BOTH IN 1 ATMOSPHERE OF ARGON - 0.2"/MIN. LOAD RATE.

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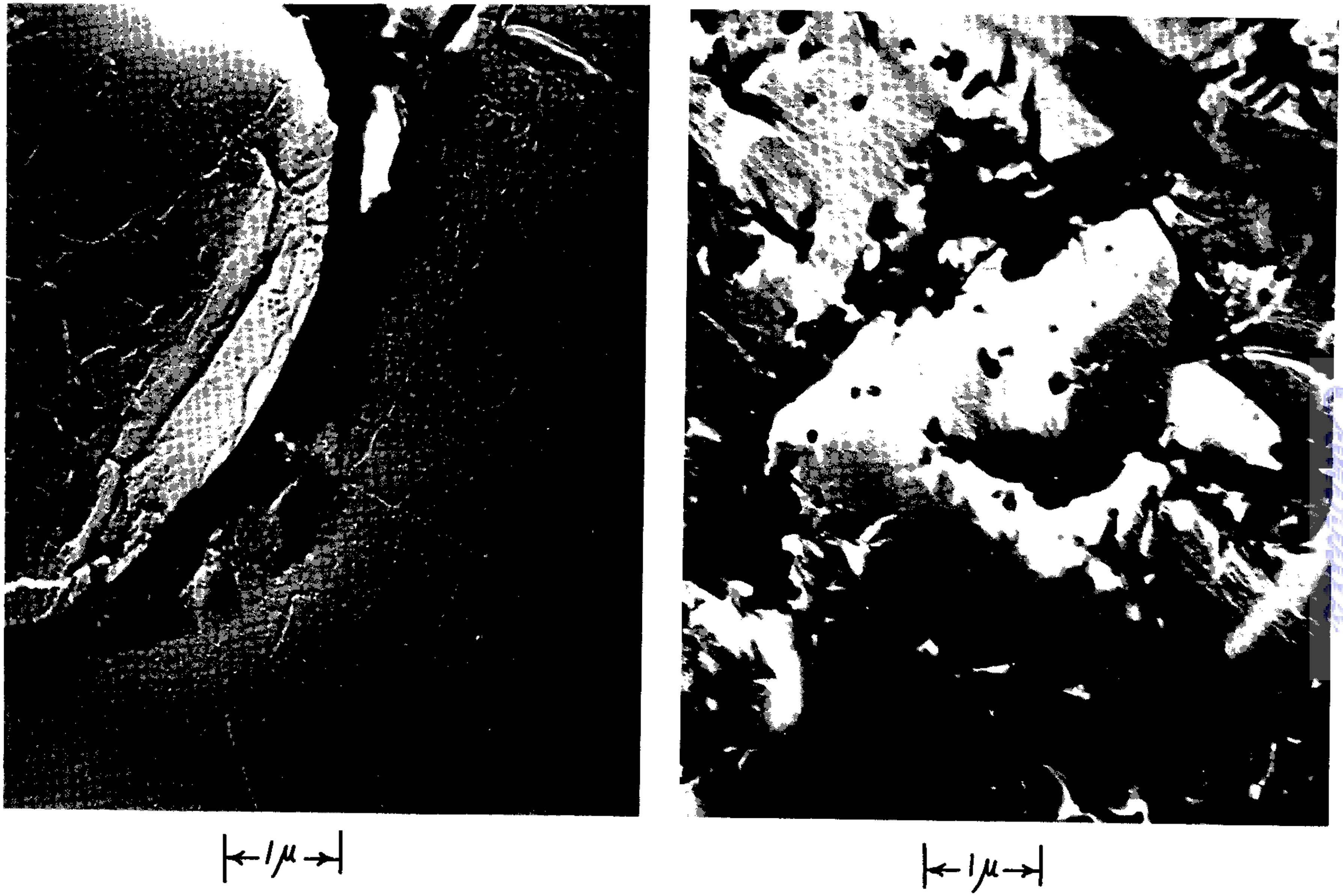

(a)

(b)

FIGURE 35. FRACTURE SURFACES - SPECIMEN 2-21, ORIENTED WITH GRAIN. BROKEN AT 2455°C IN 1 ATMOSPHERE OF ARGON - 0.02"/MIN. LOAD RATE.

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6. DISCUSSION

Thus far, each group of tests has been evaluated separately in terms of the particular variable of the test. Additions and/or further substantiation to this set of conclusions may be gained by looking at the tests as a group.

The first obvious result is that, independent of the variation in testing, the graphite strength decreases above 2550°C (4600°F). It is also found that for the across-grain oriented material the strength is very insensitive to variations except density.

The increase in strength with a decrease in load rate, coupled with the increase in strength with increased pressure for the with-grain oriented material, suggests that the ZTA grade graphite is undergoing further "hot work" during the time of applied stress. This is not unreasonable, since the ZT grade graphites are achieved using a "hot work" process which undoubtedly does not go to completion. The testing increases the orientation in the same manner as the "hot working" during manufacture.

The molding direction of the ZTA grade graphite can be very easily ascertained from a piece of the slightly oxidized material. The oxidized surface of the material in the plane normal to the molding direction (with-grain) has a silvery sheen. The plane containing the molding direction (across-grain) looks black and unreflecting when viewed under the same light.

Another interesting phenomenon occasionally observed, which does not lend itself to electron microscopy, is the occurrence of spires on the fracture surface (the mating surface shows a corresponding pit) for the with-grain orientation. These spires vary in size to approximately 0.05 inch on a side and 0.05 inch high and are rectangular in shape. The vertical orientation is always parallel to the stress axis. This fracture system resembles the local laminar sheets noted on pyrolytic graphite failures.

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3. Leon Green, Jr., Proc. Fourth Carbon Conf., Pergamon Press (1960), p. 497.
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6. WADD Technical Note 61-18 Pt II "Research and Development on Advanced Graphite Materials."
7. E. J. Seldin, WADD Technical Report 61-72, Volume VI "Creep of Carbons and Graphites in Flexure at High Temperatures."
8. E. J. Seldin, Unpublished results to be contained in Volume XVIII of WADD Technical Report 61-72, "High Temperature Tensile Creep of Graphite."

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APPENDIX

DISCUSSION* OF GRAPHITE MATERIAL USED FOR STUDY

The tensile specimens of ZTA graphite used in this study were machined from five different fabricated blocks; 1714, 1721, 1744, 1745, and 1746. Bulk density was measured and controlled on the specimens taken from the last three blocks but not the first two. However, limited measurements of bulk density were made after the tensile test by immersion in water of the broken fragments of the tensile specimens from blocks 1714 and 1721. From these examinations, it can be stated that the bulk density of the specimens from all except block 1721 generally fell in the range 1.92 - 1.98 g/cc and are therefore representative of typical ZTA graphite. Specimens from block 1721 covered the density range from 1.87 - 2.00 g/cc and therefore resulted in wider spread of strength values than those from the other four blocks. Figure 36 shows tensile strength values obtained from blocks 1714, 1721, and 1745 in the temperature range 2775 - 3030°F under comparable atmosphere and strain rate. The effect of the wider spread in density for block 1721 specimens is clearly evident. The opposite variation of strength with density for the two grain orientations is in agreement with other experience.(5)

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* Supplied by National Carbon Company

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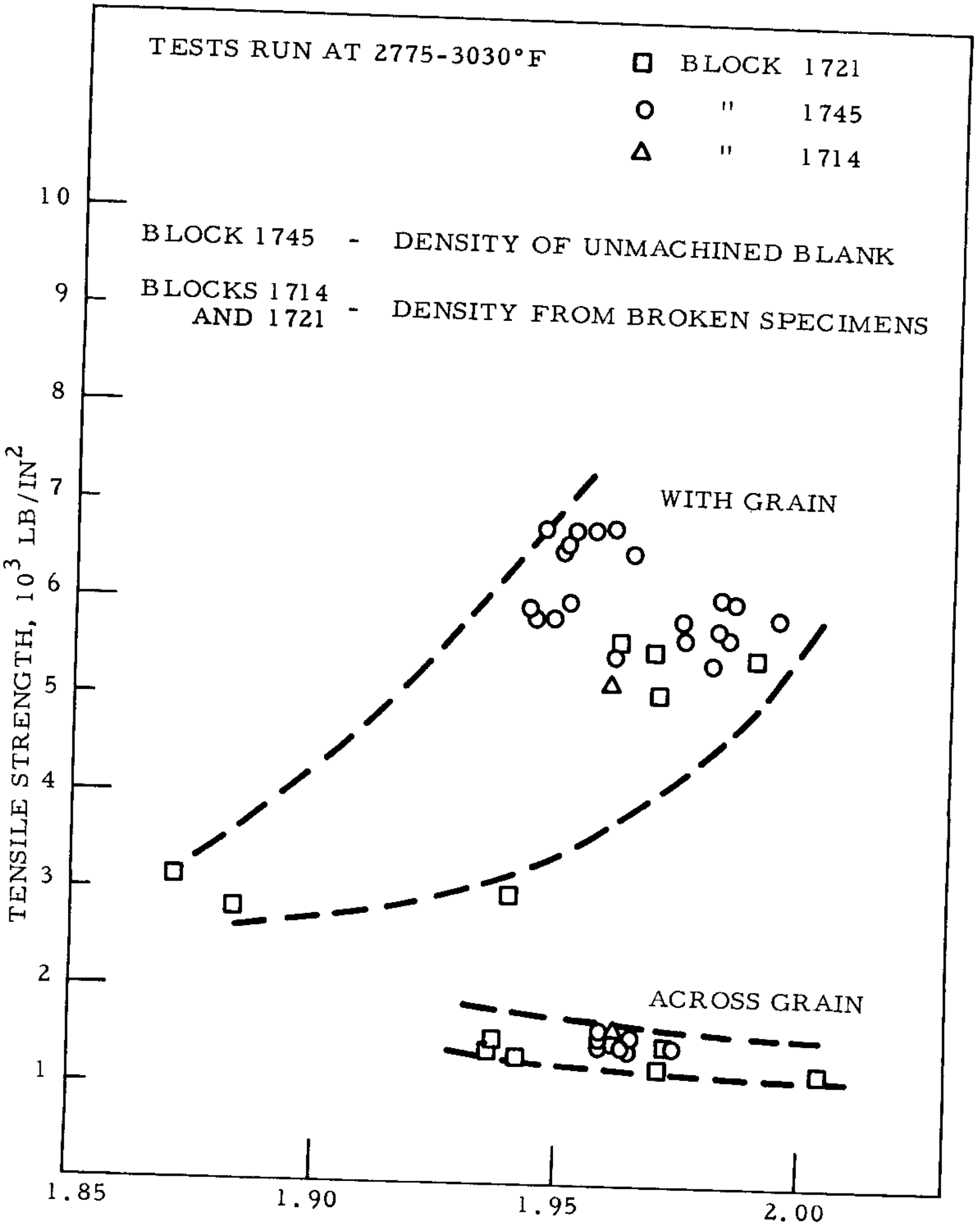

FIGURE 36. TENSILE STRENGTH OF ZTA GRAPHITE VERSUS DENSITY

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