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SOME MECHANICAL PROPERTIES OF GRAPHITE AT ELEVATED TEMPERATURES

ATOMIC ENERGY RESEARCH DEPARTMENT

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ABSTRACT

The short-time tensile breaking strength of various grades of graphite was measured as a function of temperature from room temperature to the sublimation point. A characteristic, common to all the strength versus temperature curves, is that the strength approximately doubles in going from room temperature to about 2500° C and then decreases rapidly to zero near the sublimation point. The dependence of strength on room temperature, bulk density and the density distribution within an original block is given for grade EBP graphite. The creep characteristics of grade ECA graphite under tensile stress at elevated temperatures were investigated. The testing temperature ranged from 2200° C to 2900° C while the applied stress varied from 500 psi to 5,500 psi. The range of "steady" creep rates measured extended from 3×10^{-8} to 2×10^{-4} inches per inch per second. The activation energy and change in entropy defined by rate theory were calculated for the above conditions. Low frequency dynamic measurements of the Young's modulus of grade ECA graphite made at temperatures from 1000° C to 2000° C showed an increase of the modulus with temperature. (auth)

CB 12-13-50

I. INTRODUCTION

In view of the interest in refractory materials for many high temperature applications, an investigation of the properties of graphite up to the sublimation point was undertaken. A search of the literature and inquiry[1] with the National Carbon Company indicated that no studies on the mechanical properties of graphite have been made at temperatures above 1500° C. All the grades of graphite used in this investigation were manufactured by the National Carbon Company and are listed in Table I.[2]

The anisotropic nature of graphite is well known. The tensile breaking strength of extruded graphite, for example, is higher in the direction of extrusion than perpendicular to that direction. In molded graphite the strength is greatest in a direction normal to the molding pressure vector. Such anisotropy of the polycrystalline material is attributed to a preferred orientation assumed by the individual graphite laminae during the manufacturing process. All the specimens used in the recent investigation were fabricated with their axes parallel to the direction of maximum strength.

As the investigation progressed it became evident that many factors contributed to the observed variations in the properties of graphite. In the present preliminary survey, however, no attempt was made to investigate fully the effect of such variables as grain orientation, density, particle size, types of pitch and coke, and graphitization temperature. It must be emphasized, however, that these variables (which may vary from piece to piece within a given batch as well as from grade to grade) may profoundly influence the mechanical properties of the material.

II. EXPERIMENTAL PROCEDURE

A. Tensile Breaking Strength

The tensile specimens used in the present investigation were heated by the passage of a direct current. The electrodes and graphite adapters supporting the specimen are shown in Fig. 1. This figure shows that the load was applied by weights placed in a pan. Later the pan was replaced by a bellows arrangement and the load was applied pneumatically. An even more satisfactory method of application utilized a lever mechanism actuated by a pail of lead shot. Ball-bearing universal joints at the two electrodes insured the application of a pure tensile stress. Pertinent details of the two types of specimen which were utilized are given in Fig. 2. Typical specimens which had attained a very high temperature may be seen in Fig. 3. The beads on the surface apparently were formed by the condensation of material which had migrated through the porous structure after subliming at the center of the specimen.

The specimens were tested in a helium atmosphere contained by a furnace in which the specimen assembly of Fig. 1 was suspended. The furnace, which is shown in Fig. 4, consisted essentially of a graphite cylinder insulated from an external water-cooled steel jacket by a packing of carbon black. Six windows permitted observation of the specimen and electrodes, and optical pyrometer measurement of the specimen temperature. Since the strength of graphite was found to increase with temperature at low temperatures and then to decrease sharply at very high temperatures, two methods of breaking the specimens were utilized. For temperatures above 3000° K, it was found convenient to apply a load and then raise the temperature until the specimen broke. Below 3000° K, the specimens were heated to various temperatures and then loaded to failure. Unfortunately, no strain measurements were made in these early tests.

B. Creep Characteristics

The creep measurements were made with essentially the same equipment used in the short-time tensile testing. The image of the white-hot specimen was focused on a ground glass screen about five feet from the specimen by means of an achromatic lens in one of the furnace windows. Two reference shoulders on the specimen (as shown in Fig. 5) defined the gage length used for elongation measurements. Most of the elongation measurements were made upon the screen with a micrometer microscope while the test was in progress. When the time between readings was less than two minutes, however, photographs of the screen were made with a four by five Speed Graphic camera using glass plates. A fixed reference gage of metal foil was included on the ground glass screen to permit correction for dimensional changes of the photographic emulsion during processing.

C. Young's Modulus

The Young's modulus of grade ECA graphite was determined as a function of temperature by measuring the frequency of vibration of specimens fabricated in the form of a cantilever beam. The specimens were heated in a vacuum by radiation from a surrounding cylinder carrying an electric current as is shown in Fig. 6. The experiments consisted of displacing the free end of the specimen, releasing it, and photographing the resulting motion of the beam extremity with a General Radio Company Type 651-AE oscillograph recorder. A typical trace of the incandescent tip obtained in this manner is shown in Fig. 7. The 60-cycle marks along the edge of the film (provided by a spark coil actuated by a General Radio Company Type 631-B Strobotac) furnished a time base which permitted calculation of the frequency and the Young's modulus. No calculation of the logarithmic decrement was made, since it was believed that the presumably large support losses would obscure any significant changes in the internal friction of the material. Attempts to measure the modulus dynamically at room temperature by view-

ing the vibrating specimen with the Strobotac were unsuccessful due to the rapid damping of the vibration, and so a static measurement was substituted.

D. Experimental Errors

The method of heating the specimens in the breaking strength and creep tests leads to uncertainities in the interpretation of the data. Although the core temperature of the specimen was higher than the surface temperature, no correction for this temperature difference was attempted, and the results are presented in terms of surface temperature. Due to the temperature difference, however, sublimation at the core gave rise to unknown changes in the cross-sectional area of the specimen in the tests performed above 2500° C. In addition, the rapid creep rate found at high temperatures must have had a profound effect upon the strength measurements at those temperatures. In the creep study, the indirect elongation measurements were subject to errors arising from distortion in the optical system.

The experimental errors involved in the temperature measurements are considered negligible in comparison with the other uncertainties involved. The measurements were made with a disappearing filament optical pyrometer and were corrected for the emissivity of the graphite and the transmissivity of the window.

A significant experimental error in the Young's modulus determinations may be attributed to uncertainties in measurement of the thickness of the cantilever specimen which was only about 0.050 inch. Since the moment of inertia of the beam section involves the cube of this value, an uncertainty of 0.001 inch can lead to a noticeable error in the modulus calculation. Errors of lesser magnitude were contributed by nonuniformity of the temperature distribution along the beam, and by any uncertainties in beam length measurement and in frequency comparison.

By far the largest error entering into all the graphite experiments, however, arises from the lack of homogeniety of the material. Not only may the presence of voids and fissures in the structure influence the results obtained with the thin specimens used in the modulus study, but variations in the apparent bulk density of the material lead to scatter in the experimental results which probably overshadows most of the errors mentioned above. The bulk density of the molded graphites C-18 and EBP (as measured from disks sawed from a core taken from the center of the block) is shown in Fig. 8 as a function of positions in the block. A density profile of the extruded ECA graphite rod is presented in Fig. 9.

III. RESULTS

A. Tensile Breaking Strength

The breaking strength of each of the different grades of graphite tested during present investigation is presented in Fig. 10 as a function of temperature of the specimen. It may be seen that the strength of the four grades possessing a considerable degree of grain orientation approximately doubles as the temperature rises from room temperature to about 2500° C. The abrupt decrease in strength observed at higher temperatures may presumably be attributed to the beginning of significant sublimation and of rapid creep.

The smooth curves of Fig. 10 were fitted to experimental points obtained by testing several specimens at each temperature. For all grades but EBP, four specimens were used. Measurements of the apparent (bulk) density of the specimens showed a relatively small variation, and no correlation between density and strength was apparent. The EBP specimens, however, exhibited unusually large variations in both density and strength. In this case six specimens were tested at each tem-

perature and the data obtained were corrected for density variations.

The large scatter observed in the tensile testing of the EBP specimens is shown in Fig. 11. When the strength values at each temperature were plotted against density (determined by using samples from the cores of the broken tensile specimens) a linear relationship was found. When this relationship was used to correct the observed strength values to an average observed density of 1.67 gm/cc, the scatter was reduced to a point where the average values could be approximated by a smooth curve, as is shown in Fig. 12.

B. Creep Characteristics

The curves of elongation versus time which were obtained with graphite are qualitatively similar to those characteristic of metals. A typical curve is presented in Fig. 13. The slope of the apparently linear portion of this curve defines the rate of so-called secondary steady state creep. The experimental data obtained with ECA graphite at various stresses and temperatures are presented in Fig. 14 in terms of this steady creep rate, a form of presentation which has become conventional.

C. Young's Modulus

The Young's modulus of three specimens of grade ECA graphite is presented as a function of temperature in the range from 1000 to 2000° C in Fig. 15. Temperature variations along the specimen length (of the order of 10 per cent at the greatest) required presentation of the results in terms of an average temperature, an average determined in this case by using the nominal fiber strain in the specimen as a weight function. Fig. 15 also shows values of the modulus which were obtained by measurement of static deflections of the same specimens at room temperature. Considering the brittleness of graphite in the above temperature range, it is believed that the modulus measured dynamically at the low frequency employed (about 70 cycles/sec) should not differ materially from the static modulus.

IV. DISCUSSION

A. Tensile Breaking Strength

No structural explanation for the increase in the breaking strength of graphite with increasing temperature is available at the present time. The temperature to which graphite is subjected during its manufacture is stated[1] to be between 2500° and 3000° C, so that it appears doubtful that any further graphitization can occur. On the assumption that the relief of internal stresses or changes in grain orientation might take place at high temperature, many specimens were heated (with and without applied stress) and then cooled to room temperature. No significant difference between the room-temperature strength of these samples and that of untreated graphite was found.

X-ray diffraction studies[3] were made on several specimens which had been broken at high temperature. Grain orientations parallel and perpendicular to the load direction were examined. Within the limit of accuracy of the method (0.1 per cent) no change was found in the average particle size in these specimens, as compared with unstrained graphite.

A maximum in the tensile breaking strength versus temperature curve has been reported for a sillimanite refractory at 980° C.[4] Results obtained with tin and some tin alloys indicate similar behavior at temperatures in the range of -160 to -60° C,[5,6] although the data are presented in terms of ultimate strength and area reduction. The characteristics exhibited by graphite, however, differ from those of the above materials in that the increase of short-time strength with temperature as shown by Fig. 8 appears to be limited only by the imminence of sublimation. If corrections could be made for the large influence of creep and for the decrease in effective specimen cross section due to sublimation of material at the hot core, the abrupt drop in short-time strength

might possibly appear even more precipitous.

It may be of interest to note that of the various fracture criteria* proposed in the literature, the energy relationship advanced by Saibel[9] shows promise of giving the most satisfactory qualitative agreement with the behavior exhibited by graphite. Saibel's criterion, which relates fracture to the phenomenon of melting, is based upon the assumption that "the quantity of energy required for the abolition of cohesive strength is equal to the fractional change in specific volume in passing from the solid to the liquid state multiplied by the heat of fusion".

Until Saibel's somewhat tenuous argument concerning the formation of "holes" can be clarified and extended to include phase changes from the solid to the gaseous state, however, and until stress-strain data for graphite are available, quantitative speculation concerning an energy criterion for the failure of graphite will not be justified. It is suggested that the interpretation of graphite behavior might possibly be aided by strength versus temperature measurements upon other materials (such as possibly iodine) in a thermodynamic state (relative to their triple points) where melting would not occur.

B. Creep Characteristics

The experimental data as presented in Fig. 14 may be interpreted in terms of the rate theory evolved by Eyring[10] and utilized by Kauzmann,[11] Dushman, et al,[12] and others. Such theory as applied to a shear process defines an activation energy and an entropy change which are related by the following equation:

$$\log_{10}\left(\frac{\nu s}{T}\right) = \log_{10}\left(\frac{k}{h}\right) + \frac{\Delta\eta}{2.303\,R} - \frac{Q}{2.303\,RT} \qquad \ldots(1)$$

*Comprehensive surveys have been made by Orowan[7] and Gensamer, et al.[8]

where

ν_s = zero stress intercept of constant temperature lines on graph of creep rate versus stress

T = absolute temperature

R = gas constant

k = Boltzmann's constant

h = Planck's constant

Q = activation energy

$\Delta\eta$ = change in entropy

When the graphite data are plotted in terms of $\log_{10}$ (ν_s/T) versus (1/T) as shown in Fig. 16, the slope and intercept of the "straight" portion of the curve determine the activation energy and entropy change to be:

$$Q = 226{,}000 \text{ cal/mole}$$
$$\Delta\eta = -174 \text{ cal/mole } ^\circ K \qquad \ldots(2)$$

The deviation of the data presented in Fig. 16 from a straight line may be interpreted as an indication that the rate of creep may be governed by two different mechanisms, one predominant at high temperatures, the other prevailing in the low-temperature regime. The lack of sufficient data, however, precluded calculation of the energy and entropy values for the lower temperature range. Speculation regarding the existence of a change in crystal structure at about 2500° C is encouraged by a rapid decrease in the time required for establishment of a "linear" creep rate which was noted at this temperature, and by the maximum observed in the tensile strength curve at the same temperature.

Deductions concerning the behavior of graphite based upon the Eyring-Kauzmann theory, however, cannot be accepted without reservation. In the first place, as has been suggested by Lubahn[13] and others, the

distinction between secondary, steady-state creep (upon which the rate theory is predicated) and primary, transient creep may be illusory. One of the factors contributing to the illusion is the so-called tertiary creep (as exhibited by the curve of Fig. 13), which in the present work is attributed at least in part to reduction of the specimen cross section with time due to loss of material by sublimation. Secondly, the rate theory is strictly applicable only to processes of pure shear, and graphite exhibits some properties which are difficult to explain by a shear mechanism. The fracture surfaces obtained with graphite specimen at all temperatures up to the sublimation point, for example, are typical of a brittle material even though the failure might have been preceded by a considerable amount of permanent elongation.* Typical fractures obtained in torsion and tension are shown in Fig. 17.

C. Young's Modulus

The increase in the Young's modulus with increasing temperature observed for ECA graphite cannot be explained at the present time. In spite of the scatter due to the possible experimental errors discussed earlier, the results shown in Fig. 15 are regarded as qualitatively correct. It seems apparent that the thermal dependence of the macroscopic elasticity coefficients of a pure solid[14] must be influenced by the state of the material relative to its triple point. It should be mentioned that preliminary torsion experiments have indicated the existence of a possible maximum point (at about 2100° C) in the modulus of rigidity of ECA graphite which represents an increase of about 20 per cent over the room temperature value. In this case, of course, in-

*Consideration of the porous nature of graphite may possibly permit reconciliation of the apparently brittle nature of the gross fracture surface with plastic deformation of the individual elements of the porous structure.

terpretation is complicated by the fact that the shearing stress was in the plane of minimum tensile strength, normal to the direction of extrusion. The directional dependence of the shear properties of graphite has not yet been investigated, but it is presumed that the maximum shear resistance would be found in this plane. The torsion work is continuing and will be reported in detail at a later date.

V. CONCLUSION

The experiments described above represent a preliminary investigation of some pertinent mechanical properties of graphite which was made in order to evaluate the suitability of the material for high-temperature structural applications. It was found that the tensile-breaking strength of the several grades of graphite considered assumes its maximum value at a temperature of about 2500° C. The strength measurements in this region were profoundly influenced, however, by the high creep rate which was observed and investigated at corresponding temperatures. The Young's modulus of grade ECA graphite was found to increase slowly with temperature, its value at 2000° C being about 40 per cent greater than its room temperature value.

The unusual high temperature strength properties and low density exhibited by graphite make it appear promising as a potential structural material. In order to facilitate comparison of the characteristics of graphite with those of some other materials proposed for gas turbine application, for example, the tensile data for ECA graphite are presented in Fig. 18 together with corresponding data for several ceramic oxides and a good high-temperature alloy (as reported by Norton[15]) in terms of the short-time tensile strength divided by the nominal specific gravity of the material. It is apparent that for the same creep rates, graphite can be used at temperatures much higher than those permissible with other materials.

The unique behavior which was observed in the present investigation emphasizes the desirability of further study of the properties of graphite at elevated temperatures. A need for fatigue data and accurate stress-strain information obtained under controlled conditions of strain rate and temperature is apparent, and measurements of electrical resistivity and internal friction might provide useful information in regard to possible changes in grain orientation and crystal structure.

TABLE I*

Graphite Grade	Method of Manufacture	Graphitization Temperature °C	Approximate Percentage of Oriented Graine in Final Product	Final Density gm/cm^3
AGX	Extruded	2600	40	1.58
C-18	Molded	2600	30	1.60
SA-25	Molded	3000	1	1.55
ECA	Extruded	3000	60	1.67
EBP	Molded	3000	30	1.76

*The densities as given by the National Carbon Company and presented in Table I can only be considered as average values. For example, it will be shown later that the density of EBP varies from 1.59 gm/cm^3 to 1.80 gm/cm^3 within one block.

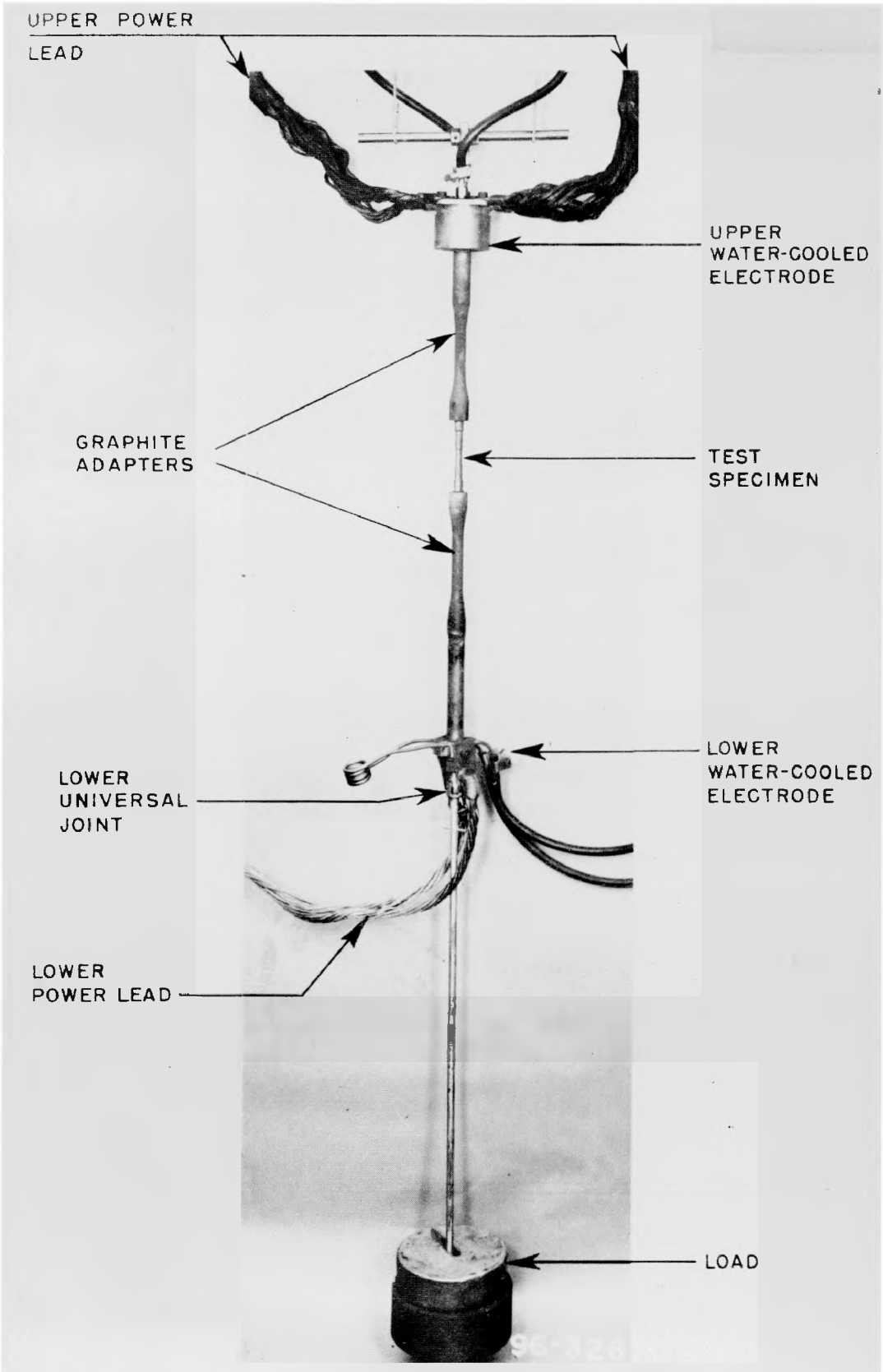

Figure 1. Specimen and Adapters

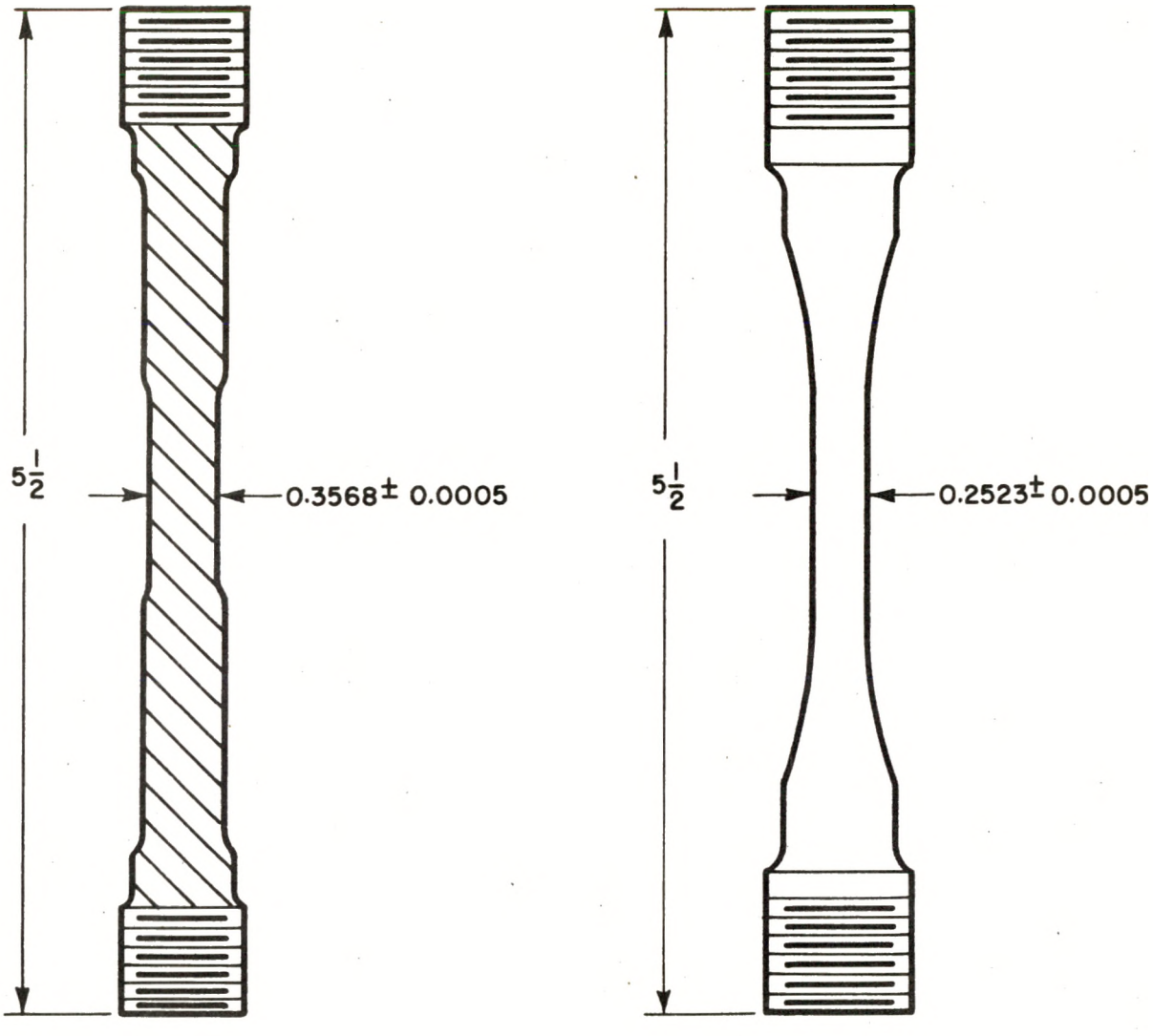

Figure 2. Specimens Utilized in Strength Measurements

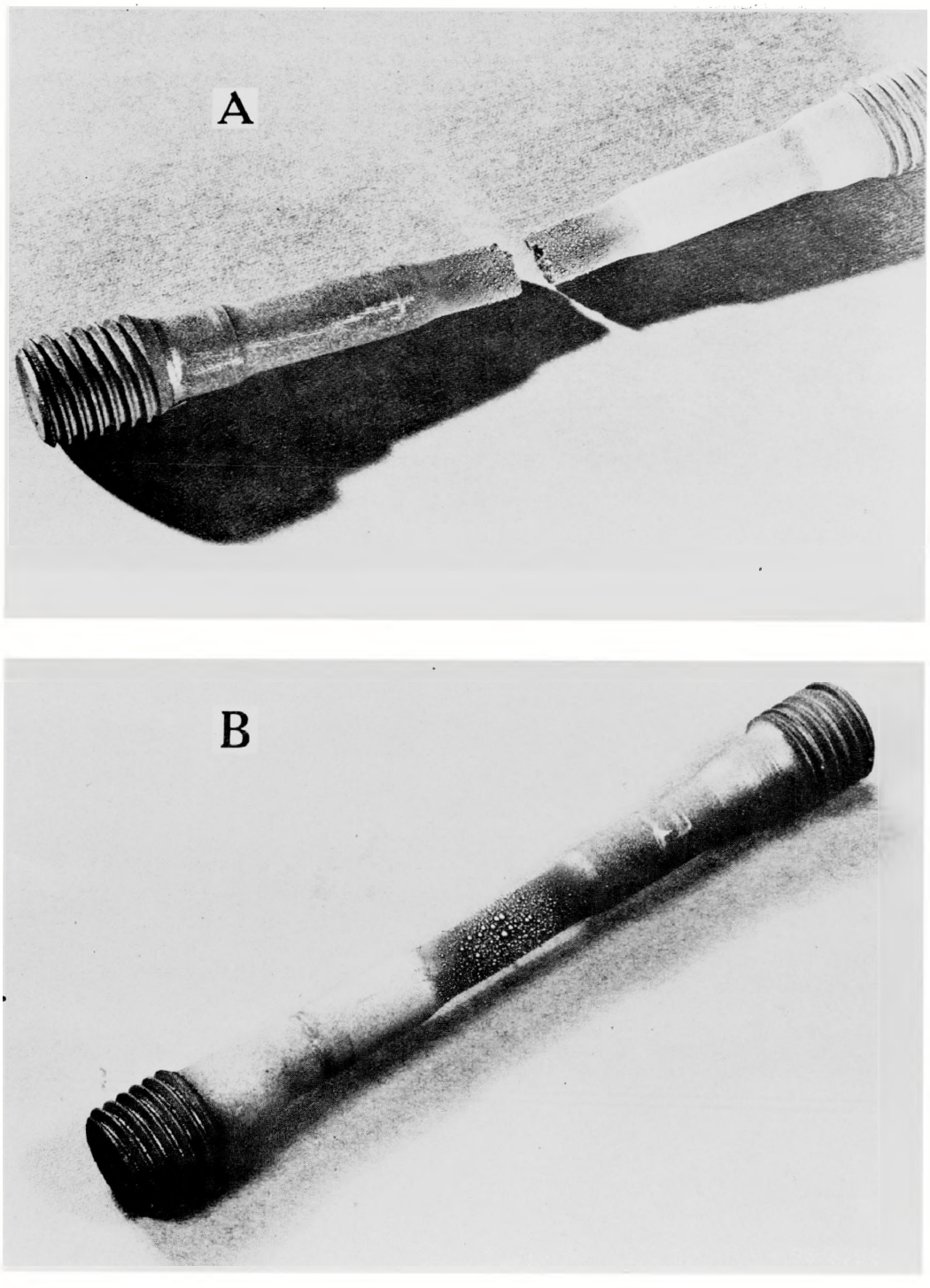

Figure 3. Broken and Unbroken Specimens

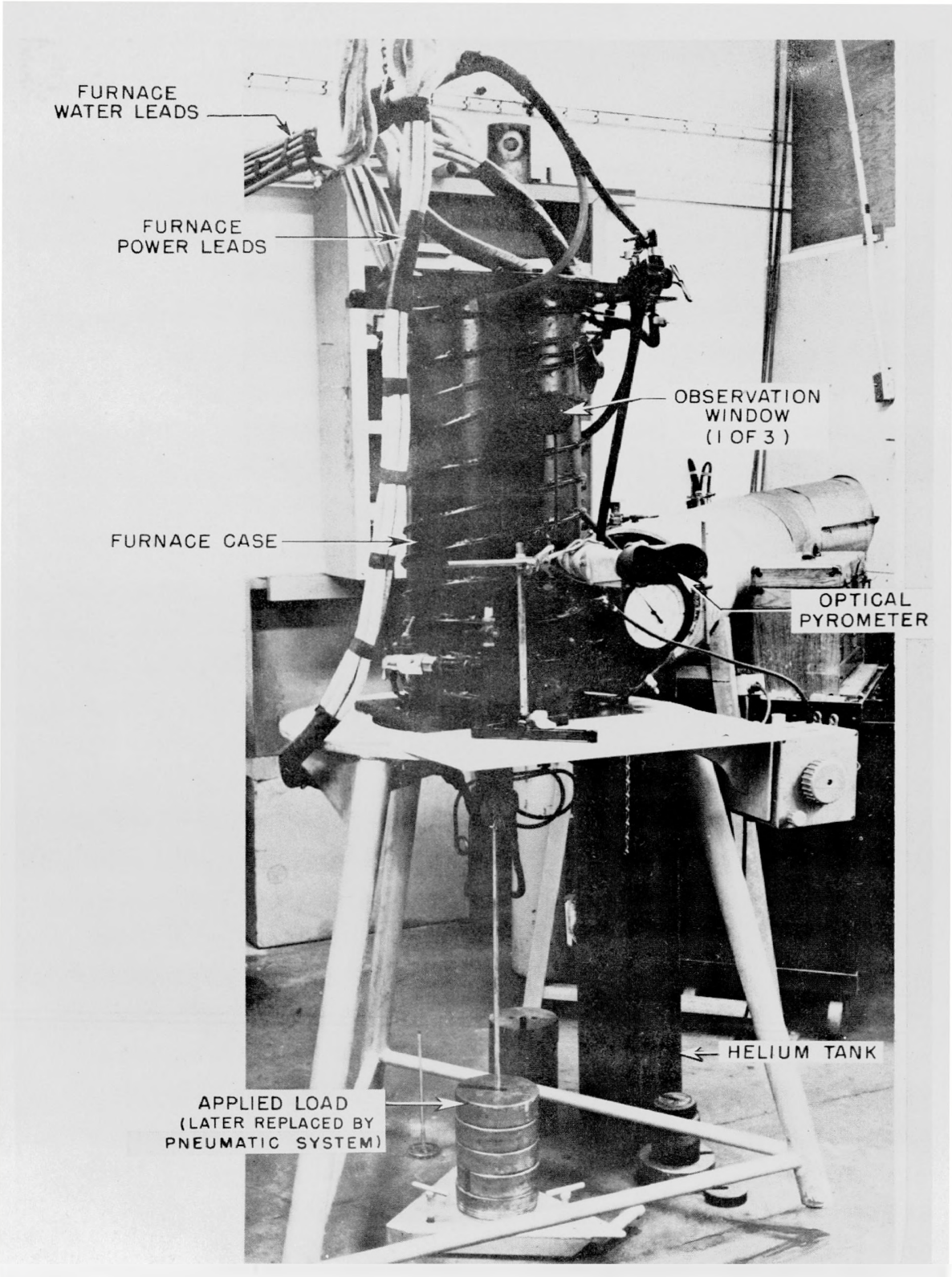

Figure 4. High Temperature Furnace

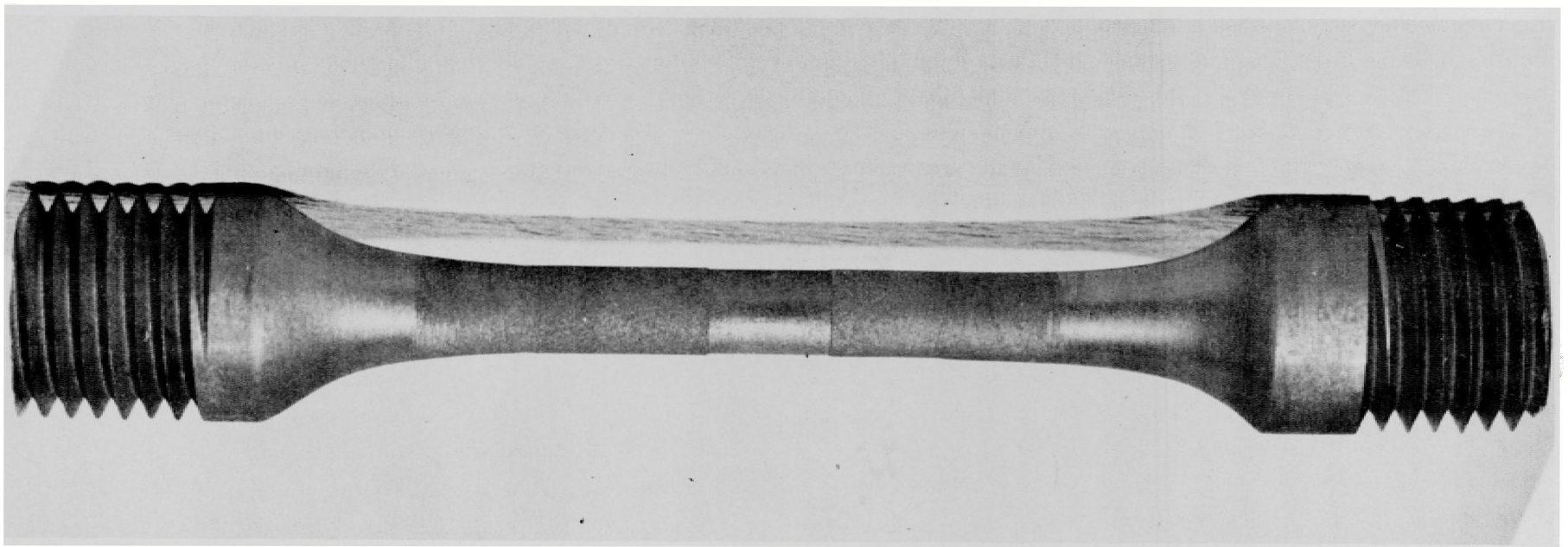

Figure 5. Graphite Creep Specimen

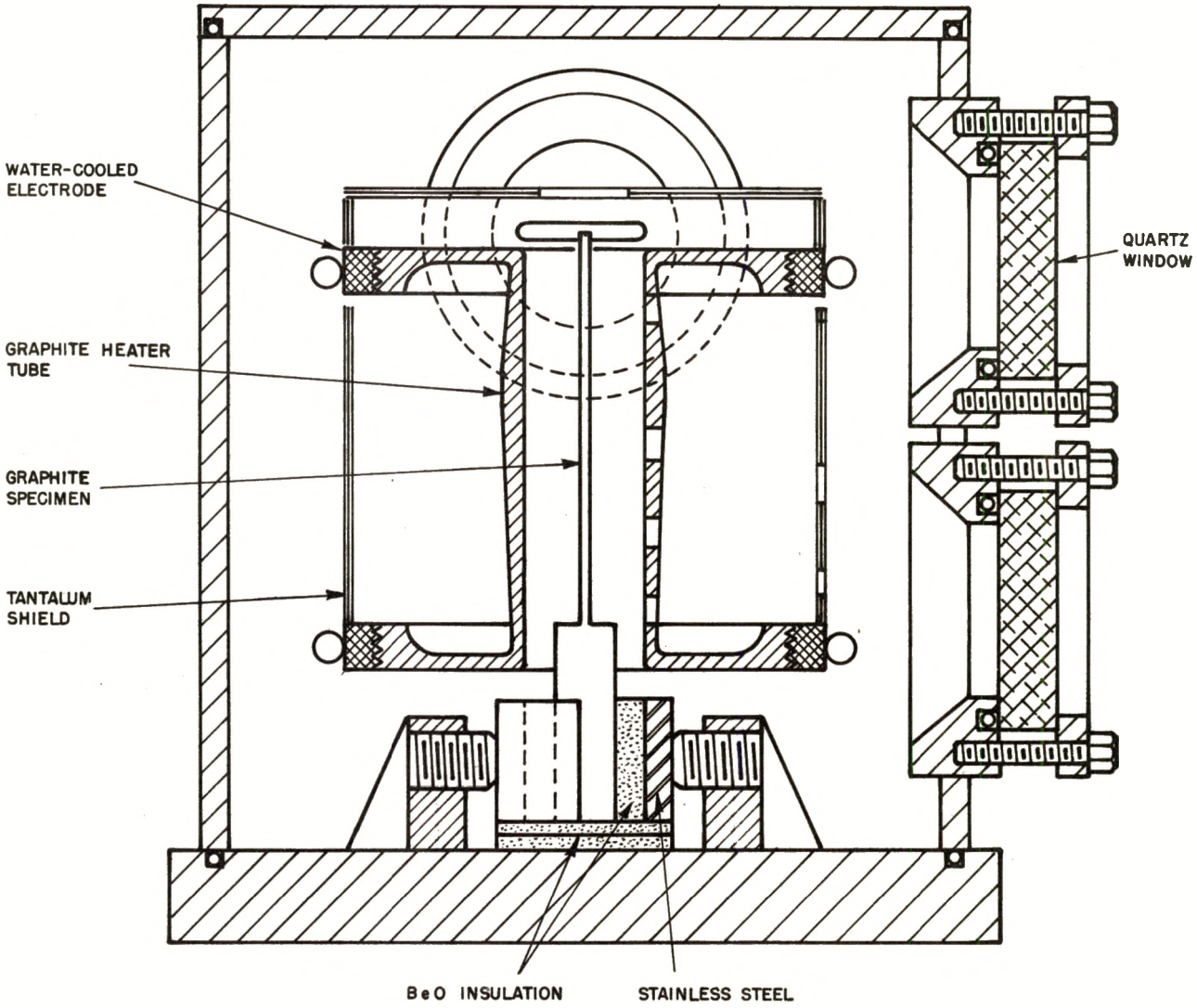

Figure 6. Cross-section of Furnace used in Young's Modulus Determinations

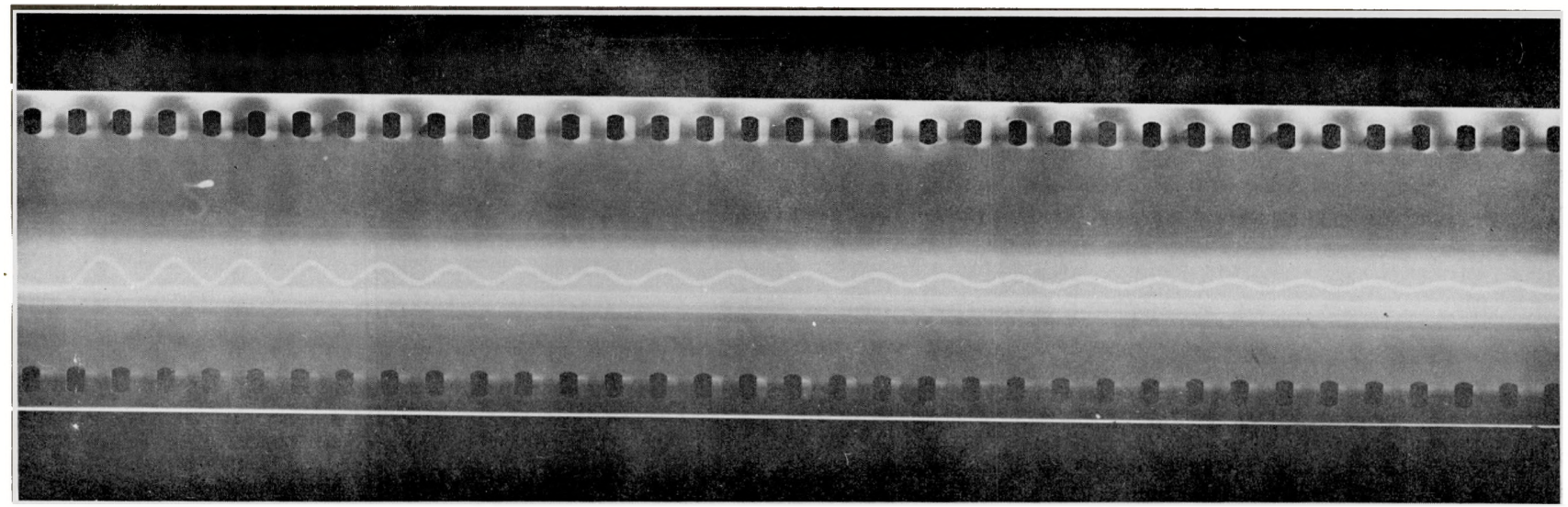

Figure 7. Initial Portion of a Typical Record Obtained in Young's Modulus Measurement

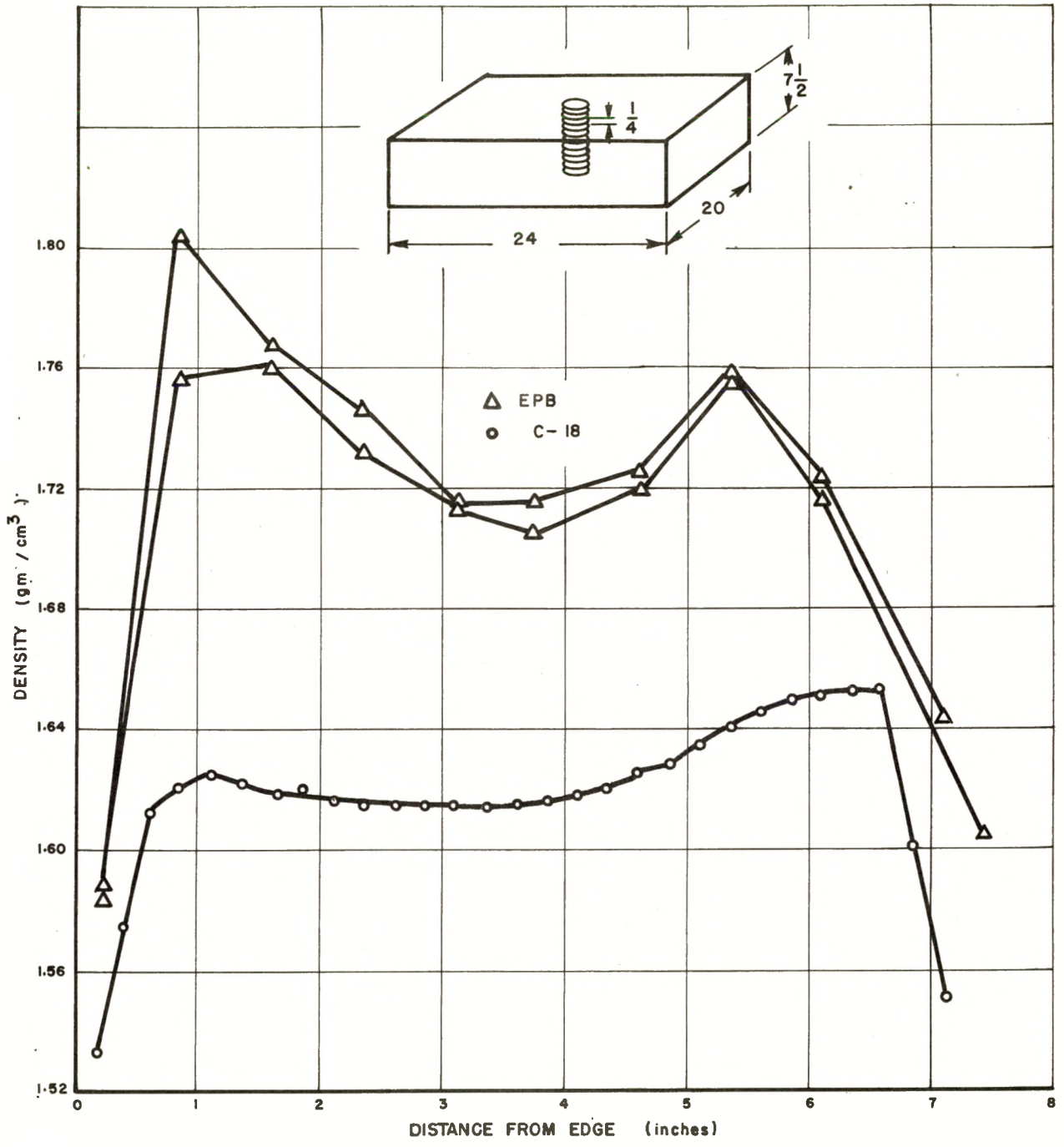

Figure 8. Density of Grades EBP and C-18 Graphite as a Function of Position in Molded Block

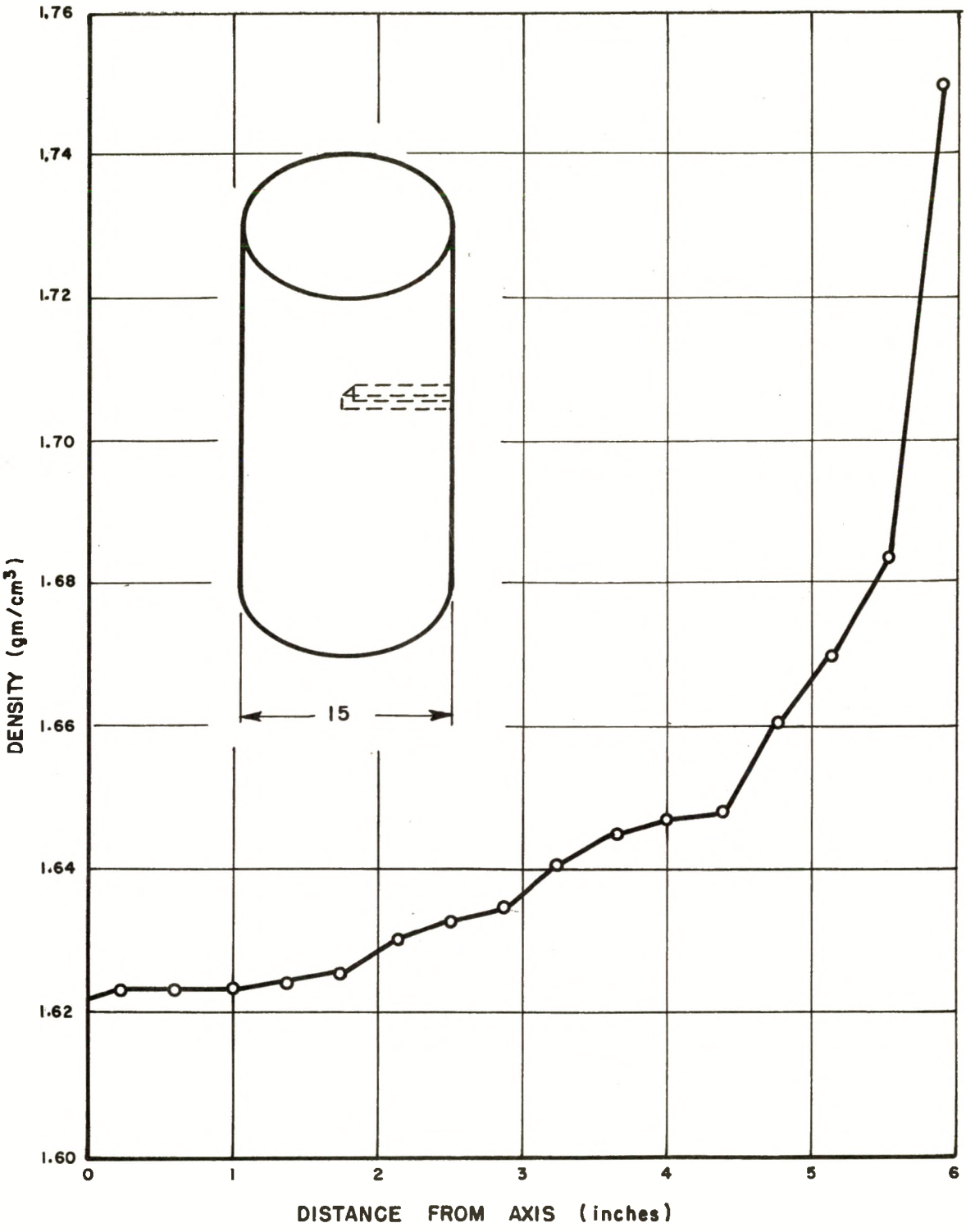

Figure 9. Density of Grade ECA Graphite as a Function of Position in Extruded Rod

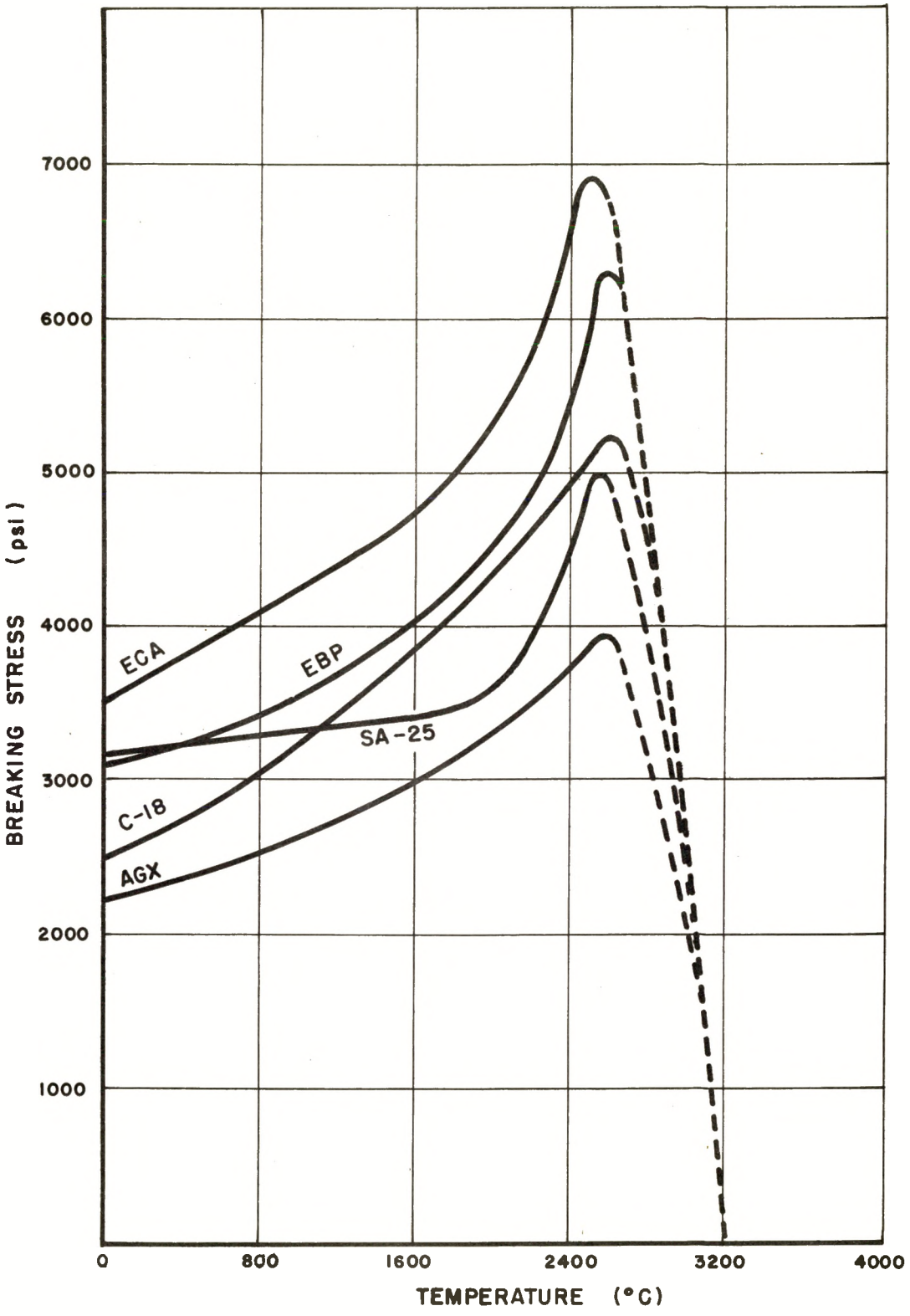

Figure 10. Short-time Breaking Strength of Various Grades of Graphite as a Function of Temperature.

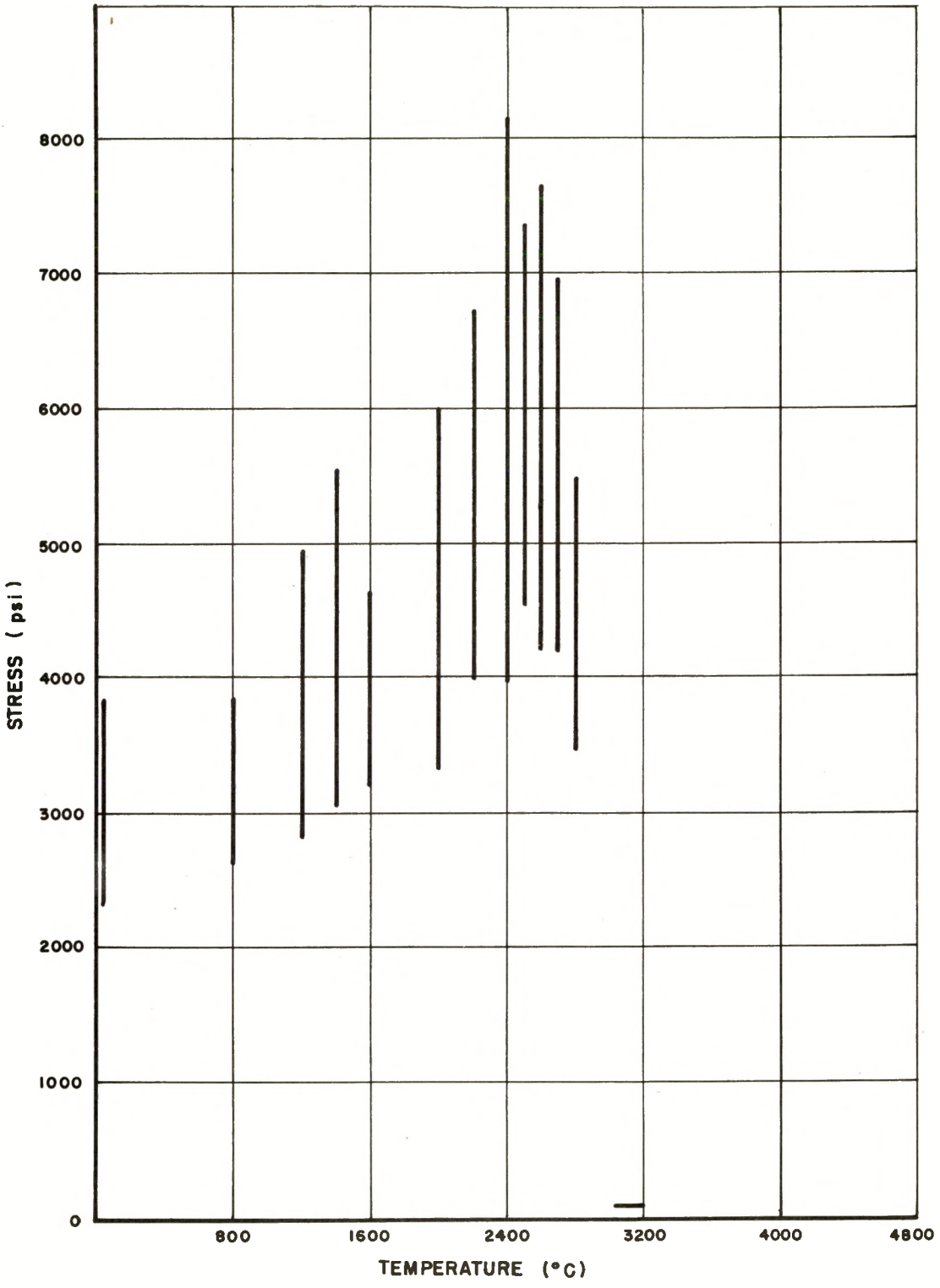

Figure 11. Range of Variation of Values Obtained in Strength Measurements with EBP Graphite

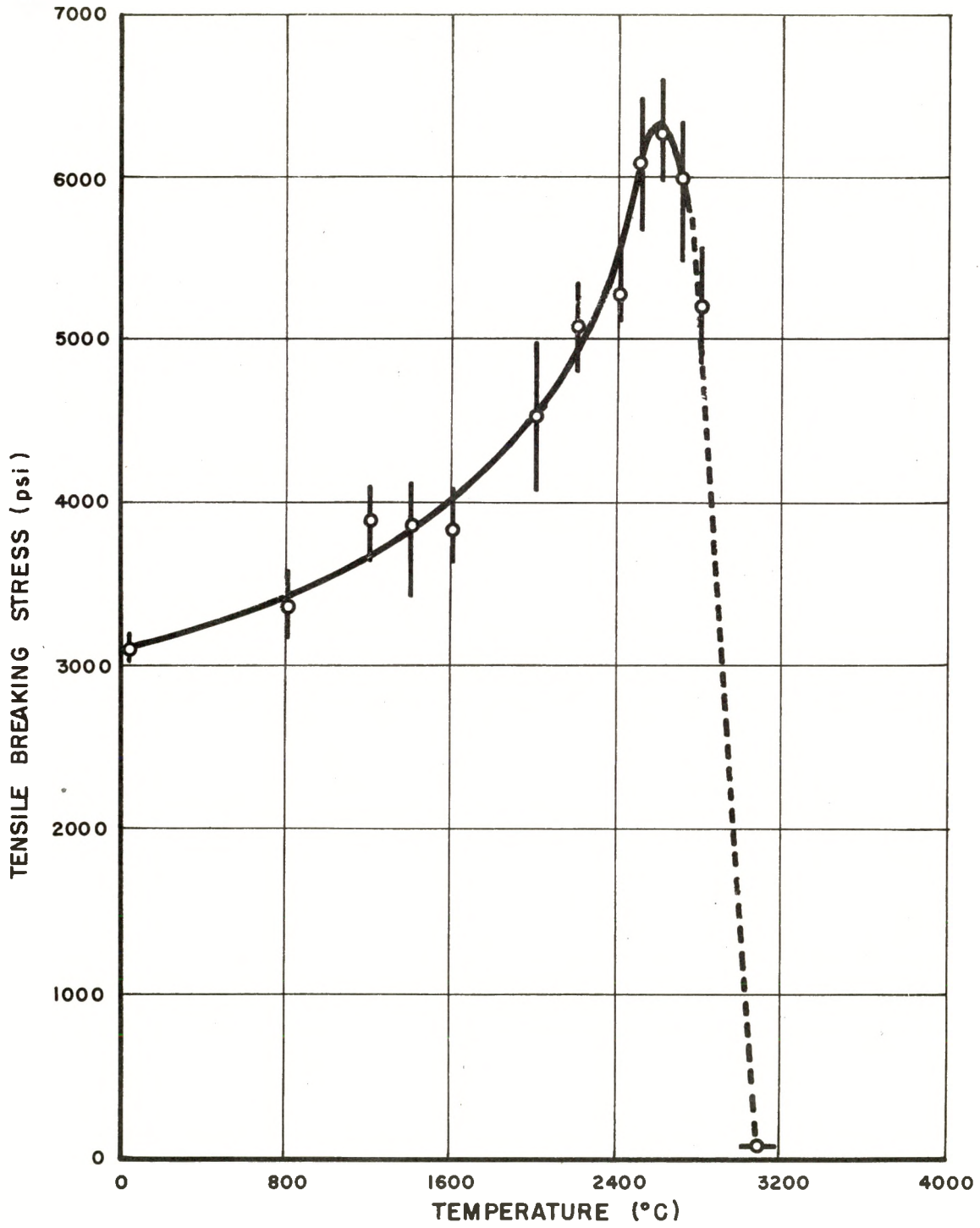

Figure 12. Strength of EBP Graphite after Correcting to a Density of 1.67 gm/cc

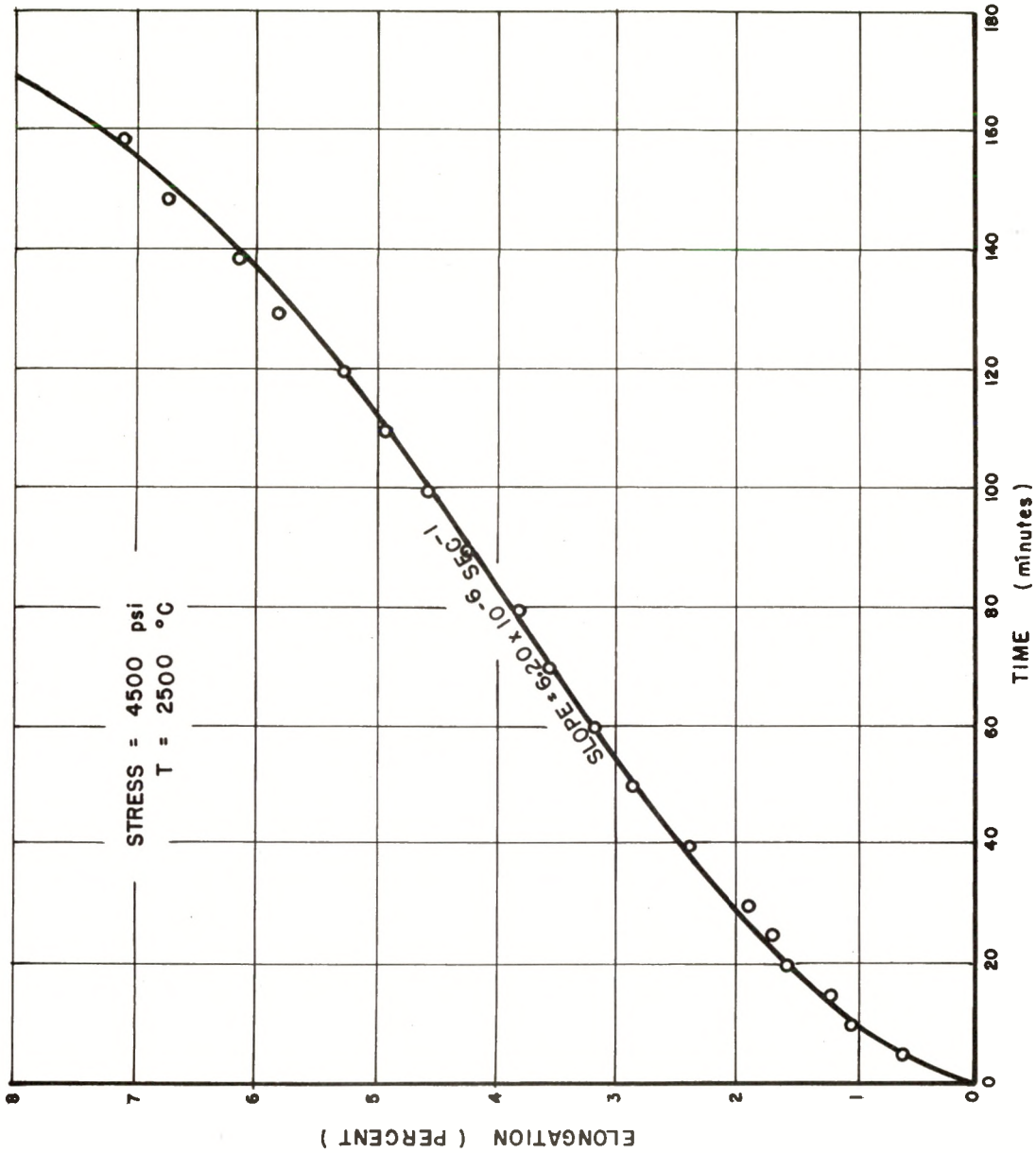

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Figure 13. Typical Creep Curve for Grave ECA Graphite

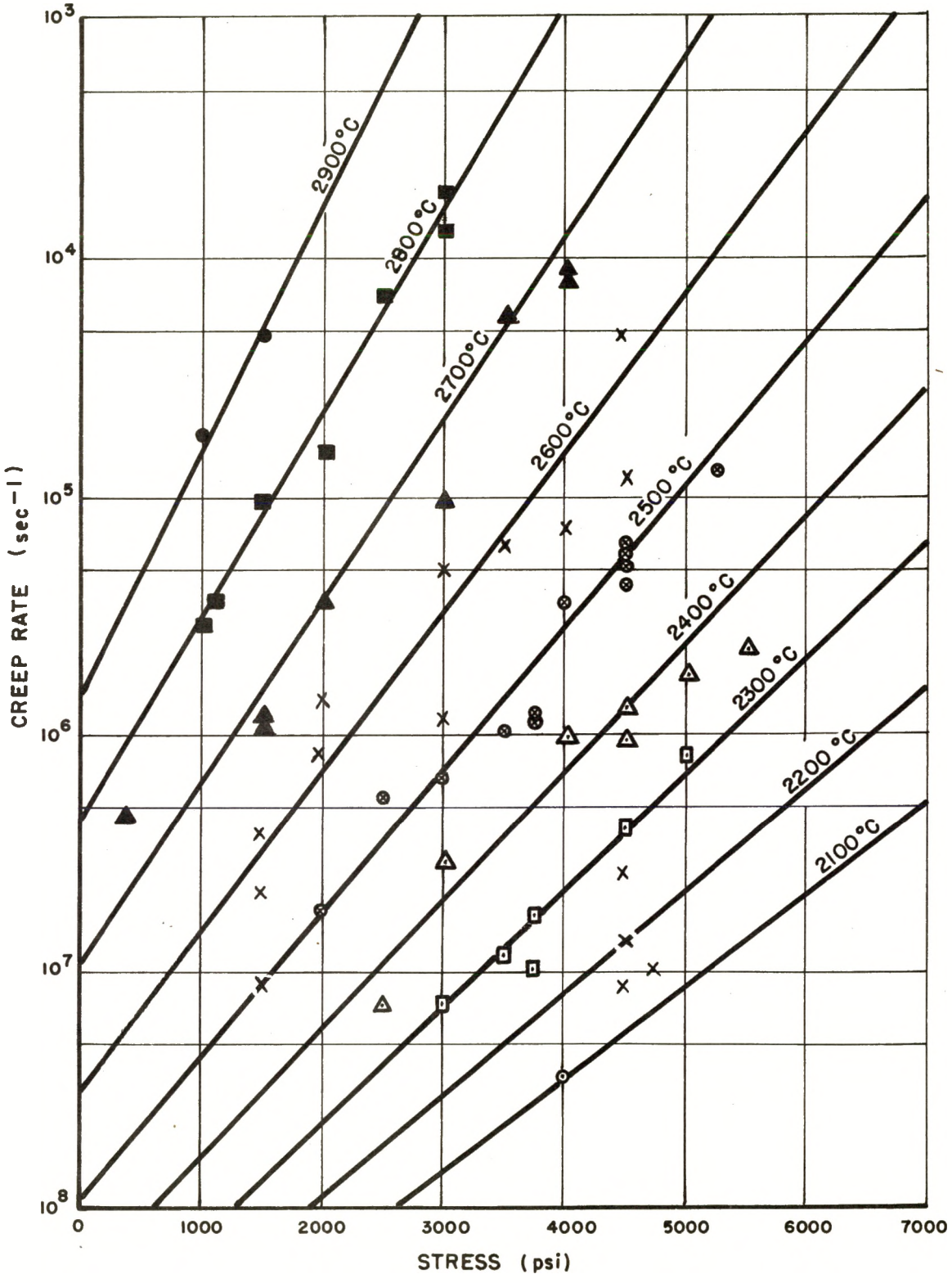

Figure 14. Steady Creep Rate Obtained with ECA Graphite as a Function of Stress for Various Temperatures

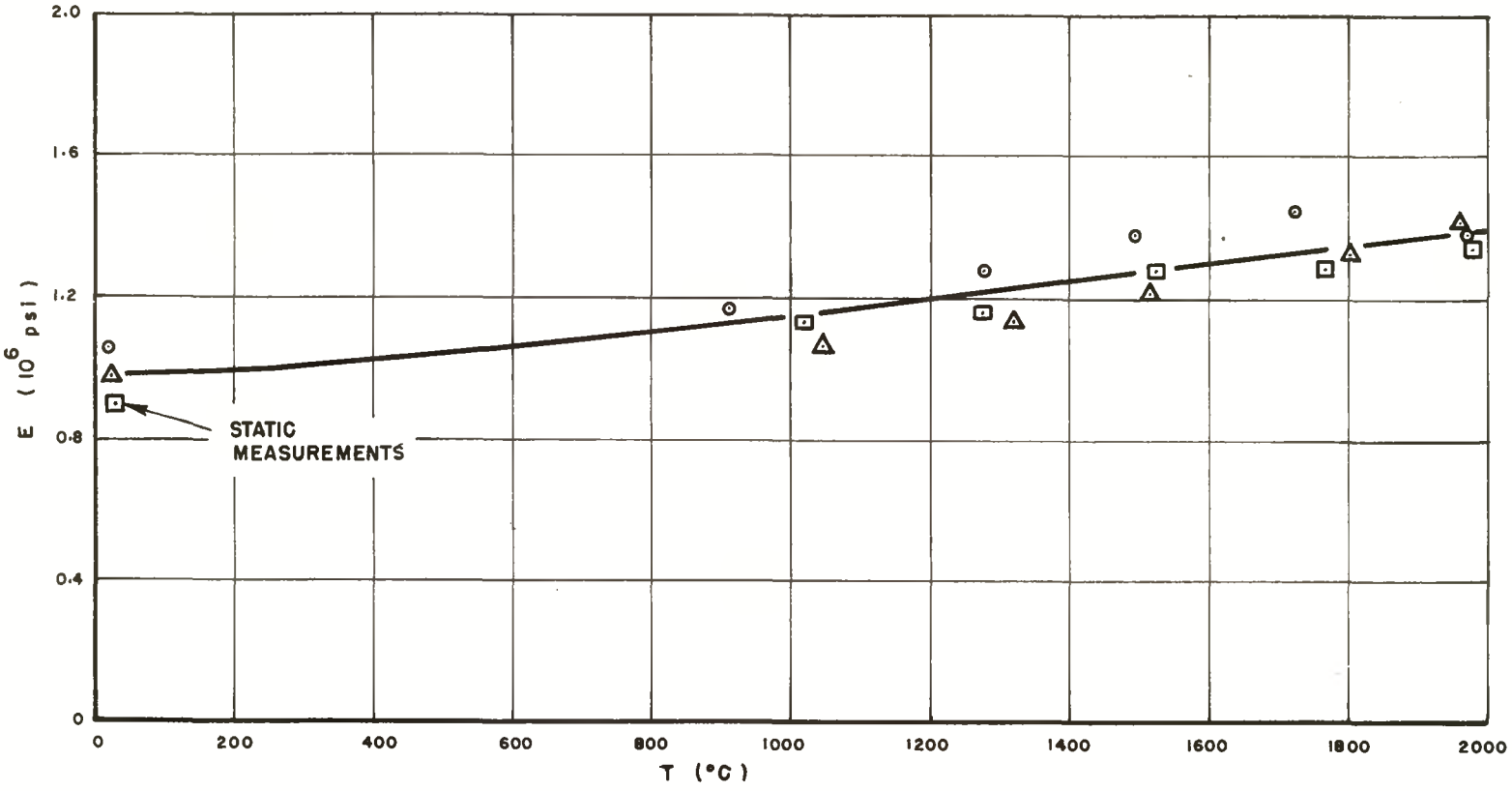

Figure 15. Young's Modulus of ECA Graphite versus Temperature

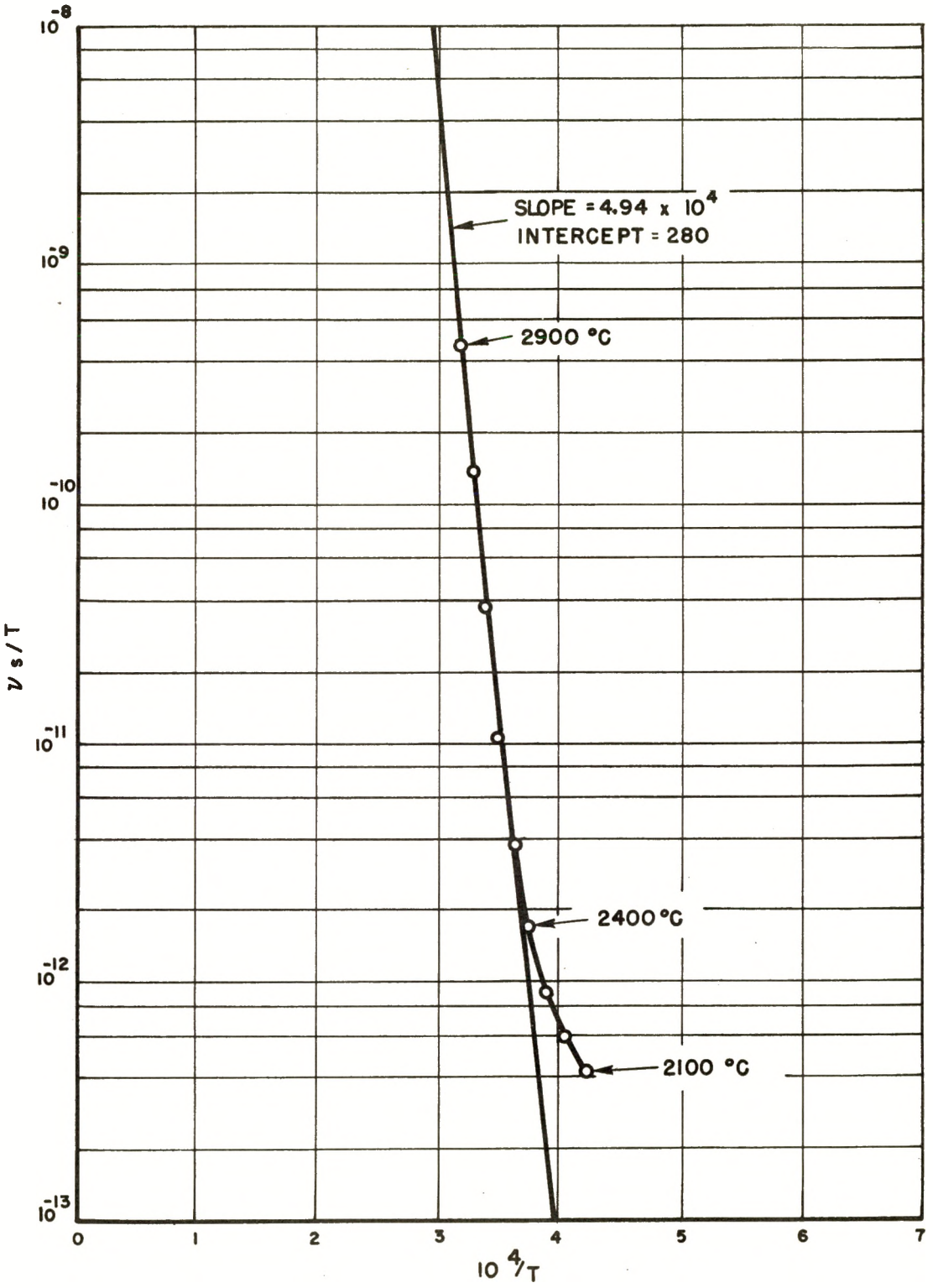

Figure 16. Determination of Activation Energy and Entropy Change for ECA Graphite

Torsion Tension

Figure 17. Typical Fractures Obtained in Torsion and Tension Tests at 2700° C

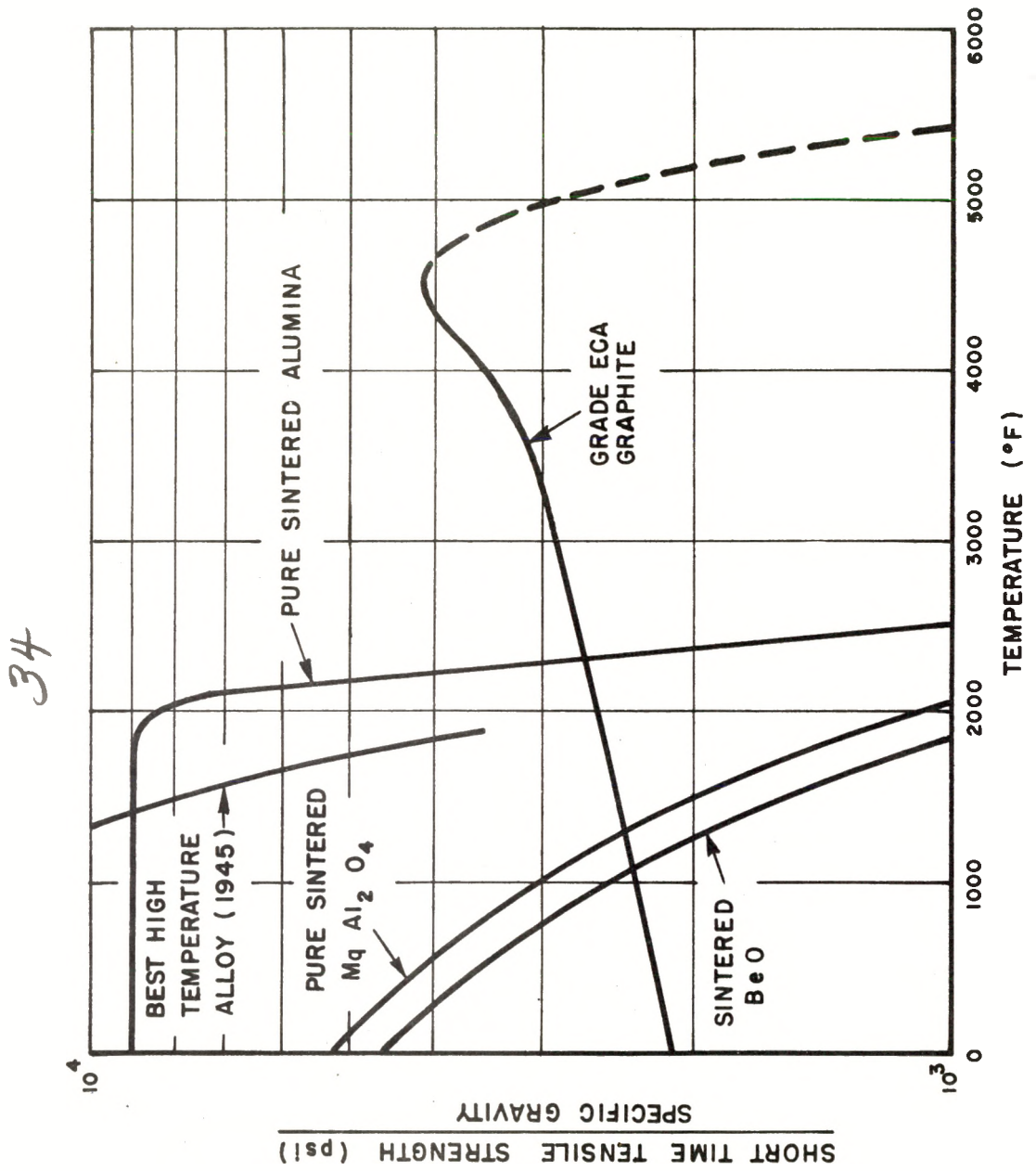

Figure 18. Strength-to-Weight Ratio for Several Materials as a Function of Temperature

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