FOREWORD

This work was conducted by 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 Non-Metallic Materials," Task No. 735002, "Graphite Materials Development"; Project No. 7381, "Materials Application," Task No. 738102, "Materials Processes"; and Project No. 7-817, "Process Development for Graphite Materials." The work was administrated under the direction of the AF Materials Laboratory, Aeronautical Systems Division, with Captain R. H. Wilson, L. J. Conlon, and W. P. Conrardy acting as Project Engineers.

Work under this contract has been in progress since May 1, 1960. The investigations covered in this report were conducted at the Research Laboratory of the National Carbon Company located at Parma 30, Ohio, under the direction of J. C. Bowman, Director of Research, and W. P. Eatherly, Assistant Director of Research.

The author wishes to thank Mr. G. Sprogis for the design and construction of the automatic temperature control device. He also wishes to thank Dr. G. B. Spence for his efforts in fitting the anelastic parts of the data to models.

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

WADD Technical Notes 61-18 and 61-18, Part II, progress reports covering work from the start of the Contract on May 1, 1960, to October 15, 1961, and the following volumes of WADD Technical Report 61-72 covering various subject phases of the work:

Volume I	Observation 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.
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, 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.
Volume XIV	Study of High Temperature Tensile Properties of ZTA Grade Graphite, by R. M. Hale and W. M. Fassell, Jr.
Volume XV	Alumina-Condensed Furfuryl Alcohol Resins, by C. W. Boquist, E. R. Nielsen, H. J. O'Neil, and R. E. Putcher - Armour Research Foundation.
Volume XVI	An Electron Spin Resonance Study of Thermal Reactions of Organic Compounds, by L. S. Singer and I. C. Lewis.
Volume XVII	Radiography of Carbon and Graphite, by T. C. Furnas, Jr., and M. R. Rosumny.

ABSTRACT

Tensile creep tests under conditions of constant load and constant temperature were performed on four different types of graphite. The temperature ranged from 2300°C to 2900°C. Some of the data were fitted to creep equations and the temperature and stress dependence of the steady-state creep rates were investigated. For National Carbon Company grade ATJ graphite, the creep rates yielded an activation energy of 124 Kcal/mole and varied with the fourth power of the stress for specimens with two different grain orientations. The activation energy was found to be the same for all the other graphites which were tested. The creep was found to be greater in the direction "against the grain" than in the direction "with the grain," and differences in creep strain and creep rate for the two orientations were found to increase as the anisotropy of the graphite was increased. For one type of graphite, the creep of specimens oriented "with the grain" decreased as the density increased and was not sensitive to differences in the proportions of filler and binder carbon.

This report has been reviewed and is approved.

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1. INTRODUCTION

Creep of graphite in tension at high temperatures has been observed and reported by several investigators.[1-8] Although the general features of the creep curves are by now well established, there are great differences in the creep behavior of different grades of graphite. The literature also shows that one can expect to find in many cases considerable scatter in the data obtained from one grade of graphite. Several means of analyzing the creep data have been proposed, but no one method is yet completely satisfactory for characterizing the data or for defining the mechanisms for creep. The equation proposed by Davidson and Losty[9] to describe the creep in graphite cantilevers and springs has also been favored by Martens[2-4] and his coworkers for describing the tensile creep of graphite. Another equation, based on a model for a viscoelastic material, was judged to be more suitable in the analysis of creep of graphite in flexure.[10] The activation energies for creep which have been reported cover a very wide range, and they have varied with the grade of graphite, the type of creep test, and the method of analysis. There is also considerable disagreement in the literature as to the stress dependence of the creep rate.

In the present work, tensile creep data are reported for several of the grades of graphite whose creep in flexure was previously investigated[10, 11] and for some low density graphites which were prepared in the laboratory. The most extensively tested grade is National Carbon Company grade ATJ graphite; the tensile creep data for this graphite have been fitted to several equations, but most of the analysis is devoted to the equation based on the model for a viscoelastic material. The other graphite grades which were tested were some very high density grade ZTA graphites and a lampblack base graphite. Activation energies have been determined for each of the graphites from the creep rates and, where possible, the stress dependence of the creep rate has also been determined. Some of the variables which are discussed are the grain type and orientation, the density, and the relative proportions of filler and binder carbon.

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2. EXPERIMENTAL

The apparatus used for tensile creep testing is illustrated in Figure 1.

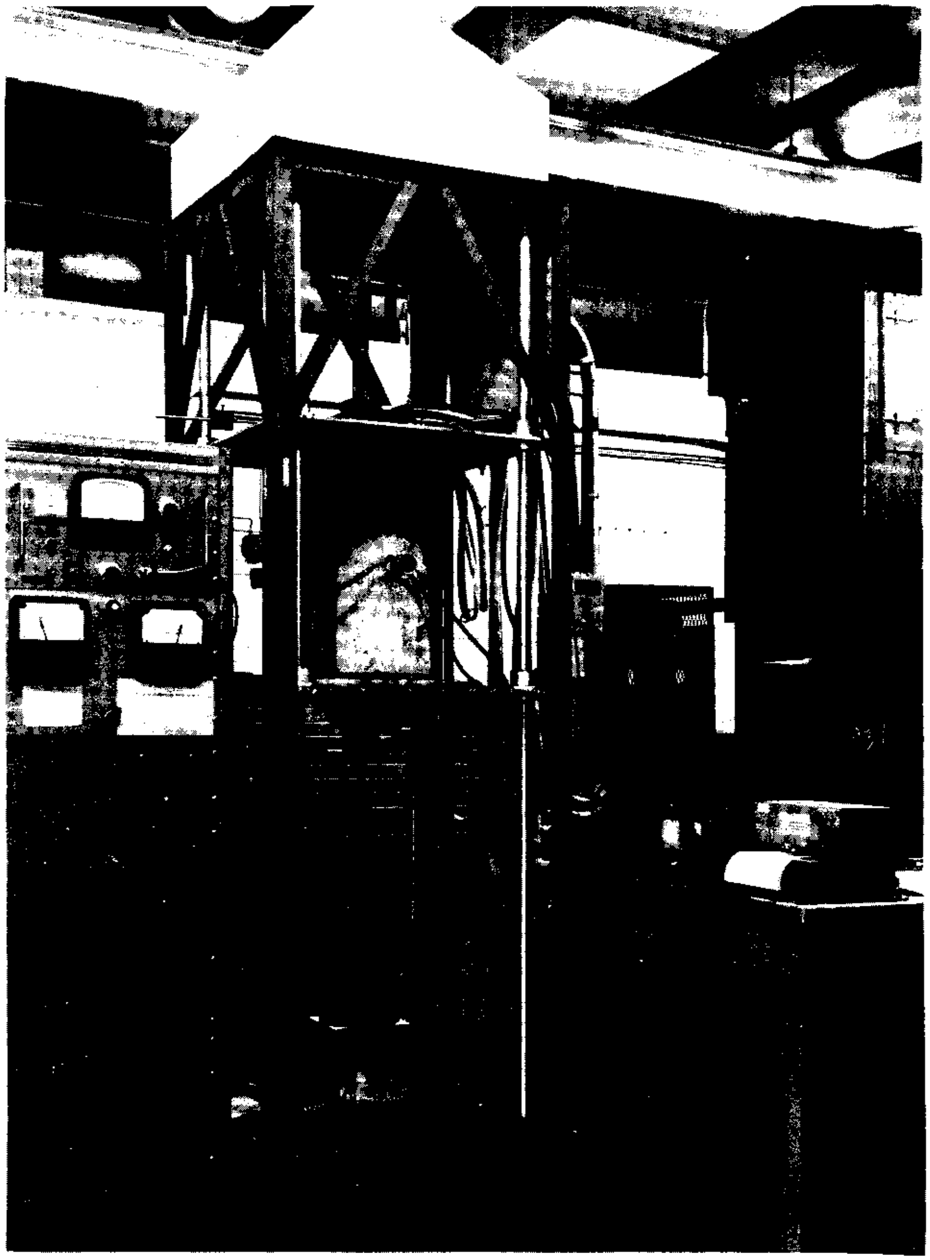

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Figure 1. Apparatus for High Temperature Tensile Creep Testing of Graphite

The major part of the apparatus is built on and around a large angle iron frame. In the center of this frame is the vertical tube furnace, which is used for heating the specimen. The heating element of the furnace is a graphite tube with dimensions of 3 inches OD by 2-3/8 inches ID by 34-1/2 inches in length. The center of the heating element has three vertical slots 1/2-inch wide by 6 inches long which are symmetrically oriented to help concentrate the hot zone at the center of the furnace. Surrounding the heating element is a carbon tube with an inside diameter of 4 inches, about five inches of carbon black insulation, and an outer shell of transite pipe. A 50-kw transformer and saturable core reactor supply the electric current for the heating element. Two sight tubes are used in order to measure and control the temperature.

The specimen and all the parts of the load train within the furnace, as shown in Figure 2, were made of graphite. The specimen, 3-1/2 inches long,

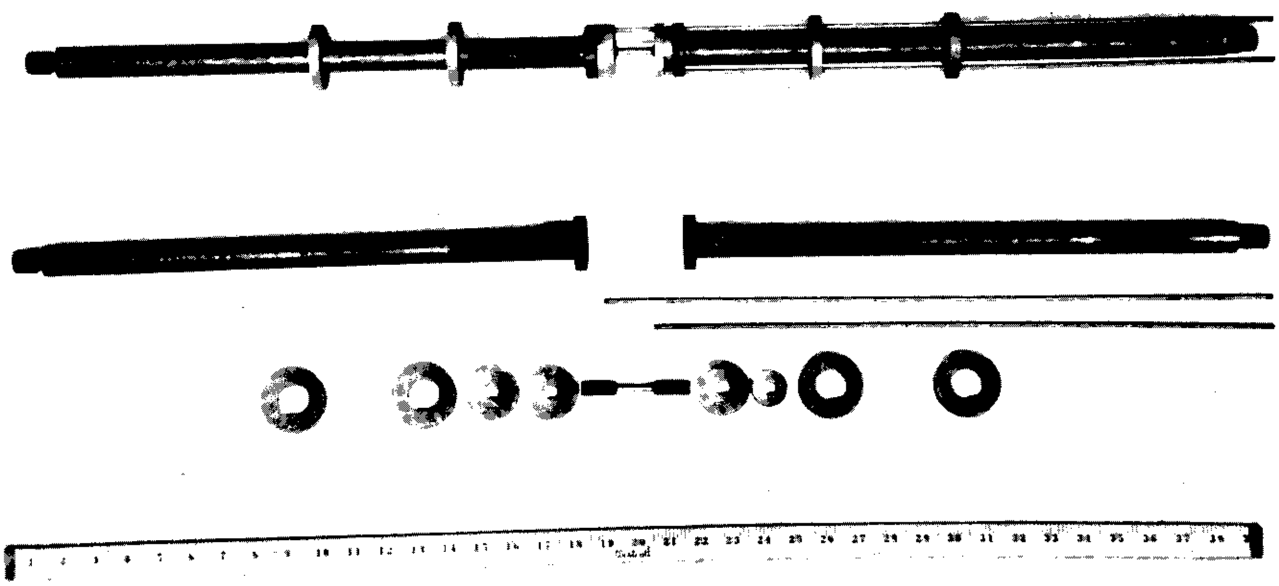

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Figure 2. Specimen, Load-transmission Rods, Lock-collars, Baffles, and Extensometer Rods Used for High Temperature Tensile Creep Tests

was threaded on both ends with 1/2-13 threads. The central gauge section of the specimen was 0.25-inch diameter and one inch long, and there was a 1/8-inch radius between each end of the gauge length and the threaded ends. Two circular collars were threaded and locked on each end of the specimen as closely as possible to the gauge section, and the ends of the specimen were connected by their threads to the main parts of the load train. Two graphite extensometer rods which projected from the top of the furnace were threaded into the upper and lower collars, respectively, and were guided by the baffles on the load train.

The furnace could be moved vertically on two screws which were driven by a small electric motor. While the furnace was in its lowered position, the load train was assembled and suspended from the top of the frame through a universal joint. The furnace was then raised so that the specimen was at the center of the furnace where it could be viewed through the two sight tubes. A graphite end cap with a plastic bellows was attached to the lower, threaded end of the heating element. The weight holder, which contained another universal joint, was threaded to the lower part of the load train; the end plate of the bellows was clamped to the load train during the assembly, thereby making a seal which kept air from entering the bottom of the furnace. The weight holder rested on a hydraulic jack below the furnace so that the specimen supported only the weight of the lower half of the load train, the end plate of the bellow, and part of the universal joint—a total load of about two pounds—before the start of the creep test. A loose-fitting graphite cap on top of the heating element permitted the load train and extensometer rods to pass freely out of the top of the furnace. An inert atmosphere was maintained inside the furnace by introducing either nitrogen or argon into the lower end of the heating element and into both sight tubes.

Two differential transformers were mounted on a common base above the furnace and the core of each transformer was connected to an extensometer rod. The sensitivities of the differential transformers were carefully matched and the difference in their outputs, which is proportional to the change of length of the gauge section of the specimen, was rectified and recorded on a strip chart recorder.

Measurement of the temperature was obtained by sighting through the front sight tube with a Leeds and Northrup optical pyrometer. The temperature could be held constant either by manually adjusting the power input to the furnace or by using the automatic temperature control device which operated from the second sight tube. The temperature sensing element of the latter was a silicon diode whose resistance decreased as the intensity of illumination increased. A telescope mounted next to the second sight tube focused the image of a small region of the specimen on the silicon diode. Automatic control was obtained by means of two transistorized circuits with highly stable d.c. voltages and a magnetic amplifier. When the optical pyrometer indicated that the desired temperature had been reached, the voltage across a potentiometer in one circuit was balanced against the voltage across a resistor in series with the silicon diode in the second circuit, and a switch was thrown to allow the control device to assume control and hold the temperature constant. Balance between the two circuits was maintained through the magnetic amplifier which controlled the saturable core reactor, which in turn controlled the main transformer. The automatic temperature control device was completed and used as part of the standard operating procedure after almost all of the testing of the ATJ graphite specimens had been completed.

In a typical creep test, the temperature was raised until the desired temperature was attained, then held constant. Load was applied by gently lowering the weights on the hydraulic jack until they were freely suspended. The furnace was maintained at constant temperature until after the load was removed by raising the weights on the jack or until the specimen broke. The testing temperature was in the range of 2300°C to 2900°C and control of the temperature with the automatic control device was good to within ±10°C.

3. RESULTS

3.1 Grade ATJ Graphite

Grade ATJ graphite is a fine grain, molded graphite which is made by National Carbon Company. All of the specimens which were tested were cut from a single large block of graphite and were oriented either "with the grain" or "against the grain." The average density of the grade ATJ graphite was 1.72 g/cc. The test temperature was varied from 2300°C to 2900°C and the tensile stress was varied from 1200 lbs/in^2 to 4800 lbs/in^2. For this graphite, the load was maintained constant during the creep test for a period of 90 minutes or until the specimen broke.

The creep curves which were obtained for the specimens oriented "with the grain" are shown in Figures 3 through 9, the curves being grouped according to test temperature. The creep curves which were obtained for the specimens oriented "against the grain" are shown in Figures 10 through 16. The amount of creep was found to increase as both the stress and temperature were increased. Under the same conditions of stress and temperature, the specimens oriented "against the grain" had greater creep than the specimens oriented "with the grain."

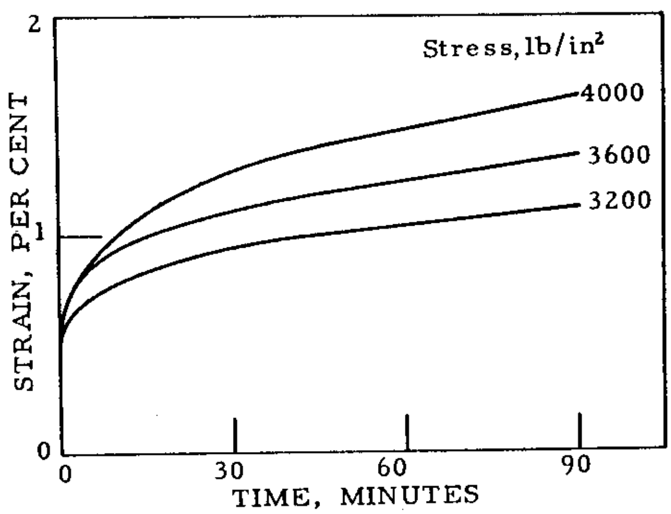

Figure 3. Strain vs Time at 2300°C for Various Stresses. ATJ Graphite, with grain.

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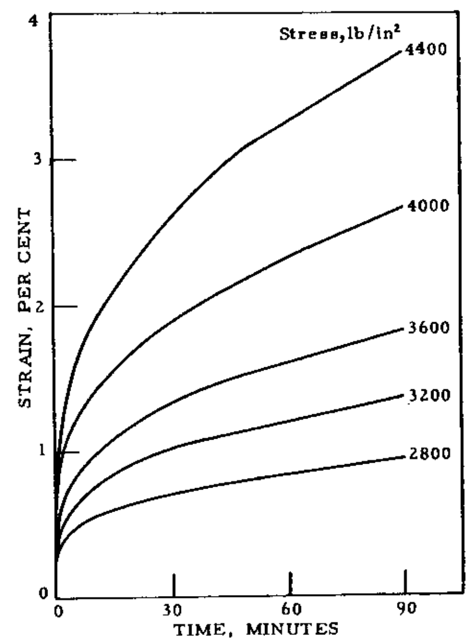

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Figure 4. Strain vs Time at 2400°C for Various Stresses. ATJ Graphite, with grain.

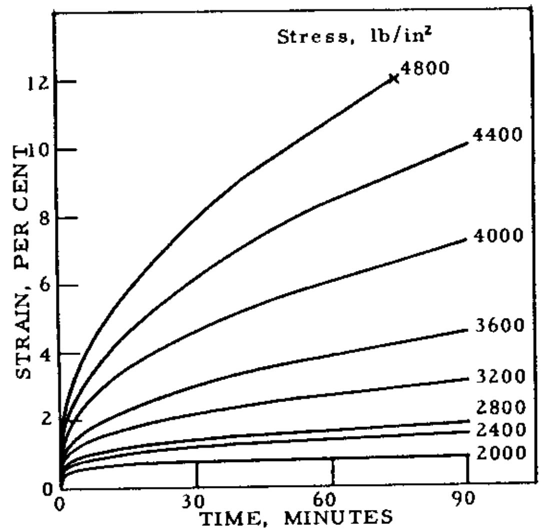

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Figure 5. Strain vs Time at 2500°C for Various Stresses. ATJ Graphite, with grain.

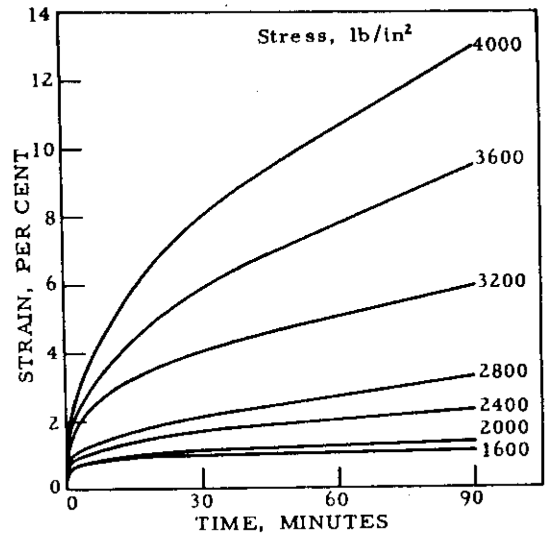

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Figure 6. Strain vs Time at 2600°C for Various Stresses. ATJ Graphite, with grain.

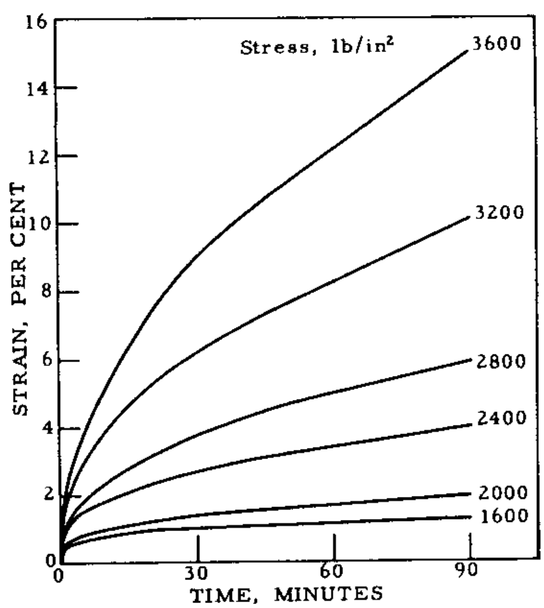

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Figure 7. Strain vs Time at 2700°C for Various Stresses. ATJ Graphite, with grain.

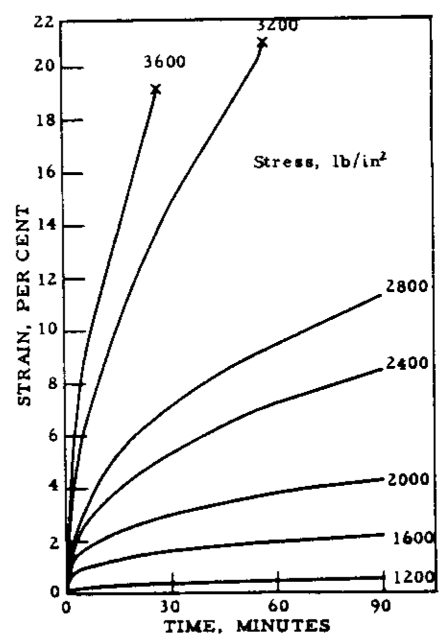

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Figure 8. Strain vs Time at 2800°C for Various Stresses. ATJ Graphite, with grain.

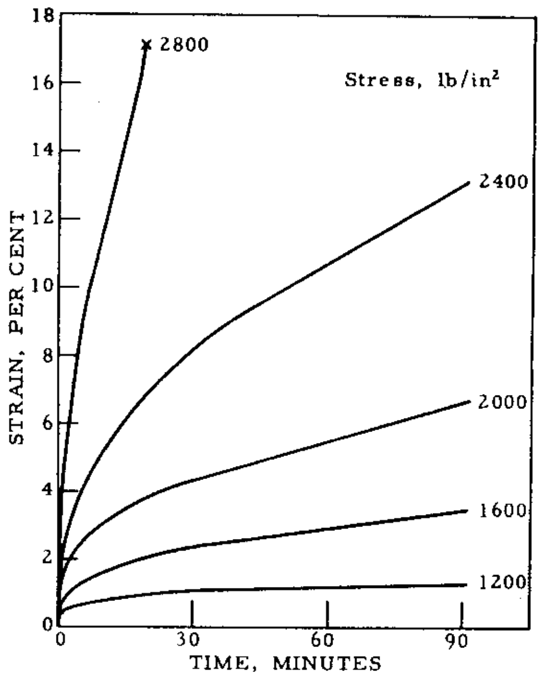

Figure 9. Strain vs Time at 2900°C for Various Stresses. ATJ Graphite, with grain. N-2356

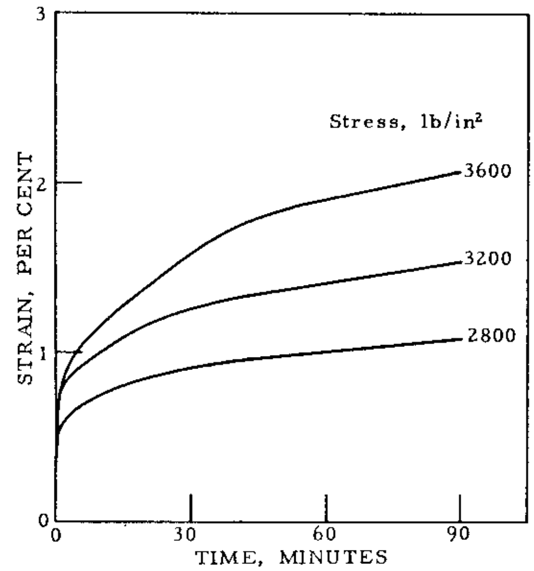

Figure 10. Strain vs Time at 2300°C for Various Stresses. ATJ Graphite, against grain. N-2705

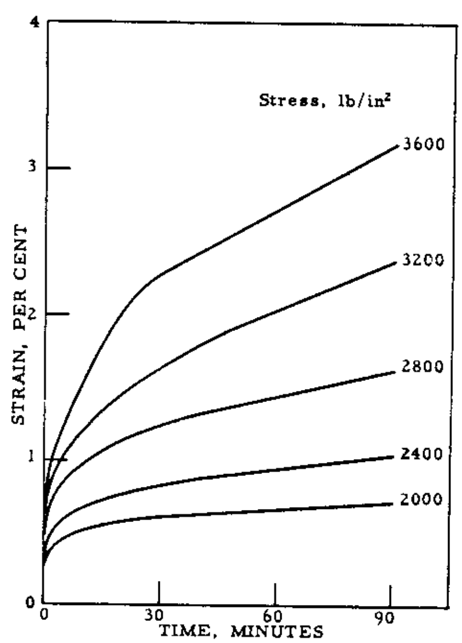

Figure 11. Strain vs Time at 2400°C for Various Stresses. ATJ Graphite, against grain. N-2706

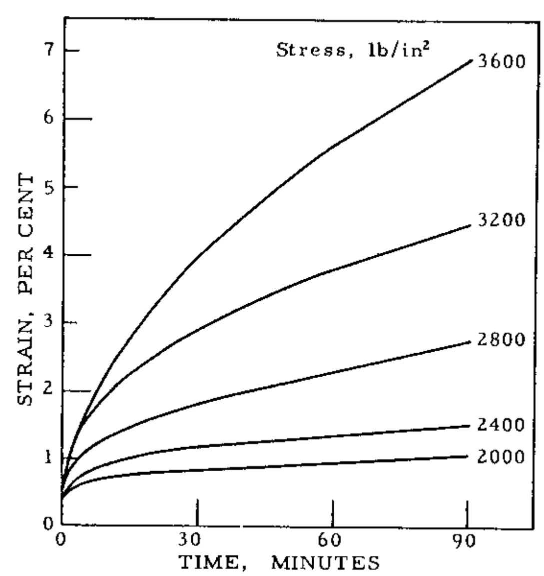

Figure 12. Strain vs Time at 2500°C for Various Stresses. ATJ Graphite, against grain. N-2485

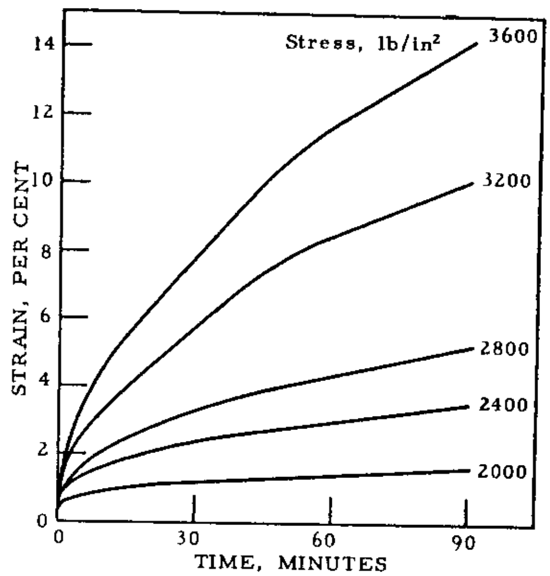

Figure 13. Strain vs Time at 2600°C for Various Stresses. ATJ Graphite, against grain. N-2486

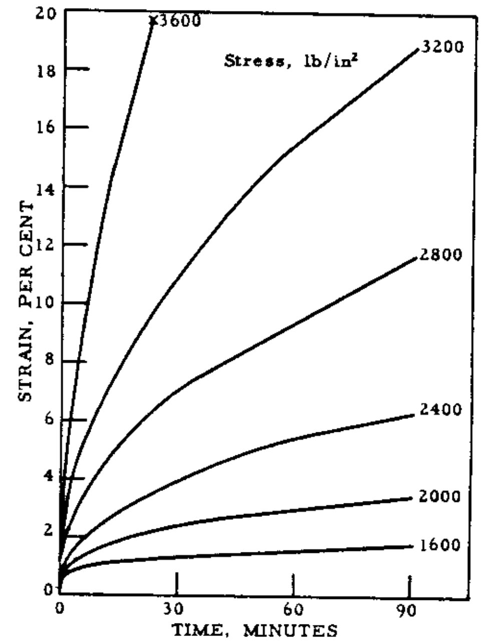

Figure 14. Strain vs Time at 2700°C for Various Stresses. ATJ Graphite, against grain. N-2707

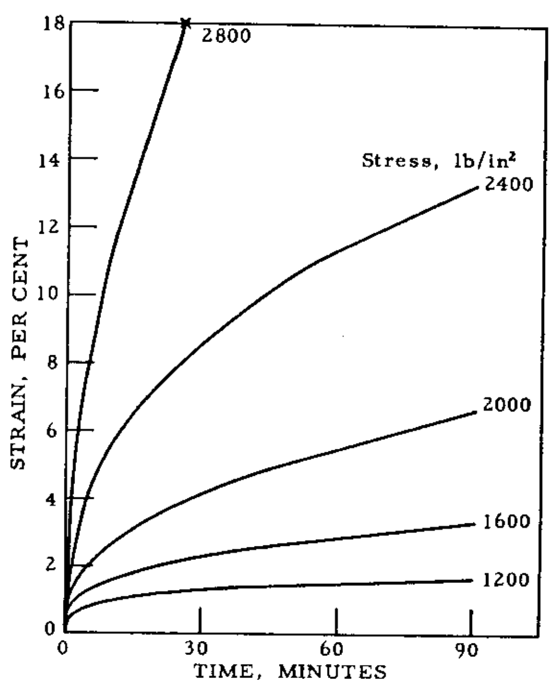

Figure 15. Strain vs Time at 2800°C for Various Stresses. ATJ Graphite, against grain. N-2708

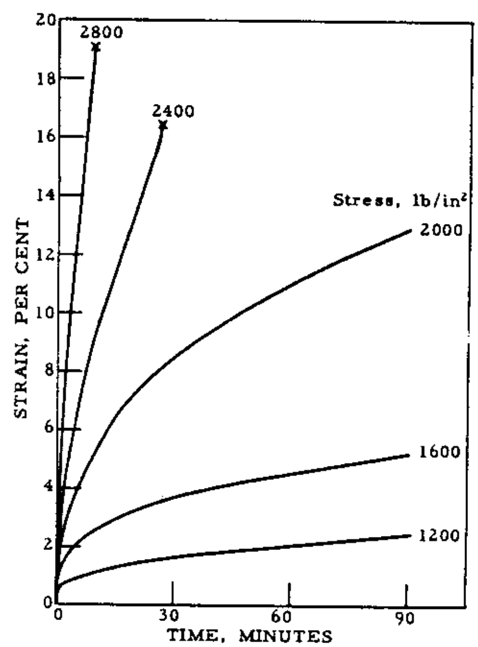

Figure 16. Strain vs Time at 2900°C for Various Stresses. ATJ Graphite, against grain. N-2709

The creep data for the grade ATJ graphite specimens were fitted to the several equations which were previously used[10, 11] in the analysis of the flexural creep data. These equations, which give the strain X in time t, are as follows:

$$(1) \quad X = A + Bt + C \log t$$

$$(2) \quad X = D + Et + F \cdot f(t)$$

$$(3) \quad X = D + Ht^{\beta}$$

$$(4) \quad X = Kt^{\gamma}$$

Equation (1) was proposed by Davidson and Losty[9] to describe the creep of graphite cantilevers and springs. The fits obtained with the ATJ tensile creep data were so poor that no serious consideration has been given to this equation.

Equations (3) and (4) were tested by plotting the data on log-log plots of X-D vs t, and X vs t, respectively. The value of D was read from the original data as the magnitude of the initial instantaneous deformation. The equations gave surprisingly good fits to the data. In the majority of cases, equation (4) gave a slightly better fit than equation (3). The value of γ was found to increase as the stress was increased and varied over a wide range from0.13 to 0.54. The value of β for any creep curve was always slightly greater than the value of γ. These results are considerably different from the results obtained by the analysis of the flexural creep data, where β and γ were lower and did not appear to vary with the stress.[10]

Equation (2) was judged to give the best description of the creep data. This equation, which was previously used in the analysis of the flexural creep data for grade ATJ graphite,[10] is based on a simple viscoelastic model containing two springs and two dashpots. The model consists of a spring and dashpot in series with a parallel combination of a spring and dashpot: the term D arises from the single spring; the term E arises from the single dashpot; and the parallel combination of spring and dashpot gives rise to the function f(t), which has the form $[1 - \exp(-\alpha t)]$ when the spring and dashpot constants are both linear in the stress and strain. The simple exponential function given by the linear viscoelastic model has been replaced by the more general function f(t) because it has been found by the analysis of the tensile as well as the flexural creep data that the simple exponential function given above does not fit the data exactly. However, the function f(t) has the same general form as the exponential function: f(t) is zero when t = 0 and approaches unity as t becomes large. The analysis which follows is based on equation (2).

The parameter D is considered to be the elastic strain since it is the deformation which occurs immediately after the load is applied. The value of this deformation is read directly from the creep curve. Since the creep rate in the first few seconds is very high, it is difficult to determine accurately where elasticity ends and creep begins; for this reason, the value of D as obtained from the creep curve is subject to considerable experimental error. The values of D were plotted against the stress, and an elastic modulus was determined at each temperature from the slope of the straight line which gave the best fit on each plot. While it appears that D is proportional to the stress, this modulus is defined hereafter as an apparent Young's modulus because of the uncertain contribution of the creep to the initial deformation and the lack of knowledge of the true stress-strain behavior before the onset of creep. Table 1 gives the values of the apparent modulus at each

Table 1. Apparent Young's Modulus From Initial Deformation

Temperature °C	Apparent Young's Modulus (lbs/in^2) With the Grain	Apparent Young's Modulus (lbs/in^2) Against the Grain
2300	$.77 \times 10^6$	$.66 \times 10^6$
2400	.76	.66
2500	.64	.65
2600	.62	.60
2700	.54	.50
2800	.48	.36
2900	.48	.41

temperature as obtained for the two groups of specimens. The modulus tends to decrease as the temperature is increased and is generally smaller "across the grain" than "with the grain." If D is truly an elastic term, the elastic part of the recovery of the deformation which takes place immediately as the load is removed should be equal to the corresponding elastic deformation when the load is applied. It has been found that the elastic recovery is generally smaller than the initial elastic deformation. This type of effect may be expected if the high temperature stress-strain curves are nonlinear in a manner similar to the room temperature stress-strain curves. The difference between the initial elastic deformation and the elastic part of the recovery would then correspond to the permanent set which is found in graphites strained at room temperature. If it is assumed that creep deformation does not enter into the observed value of D, the apparent modulus which has been determined can be considered to be a secant modulus, for it is equal to the stress divided by the total elastic strain.

The parameter E is the steady-state creep rate. The creep tests were of 90 minutes duration, and the rates tended to be linear over approximately the last 30 minutes of each test. The values of E were determined directly from the final linear slopes of the creep curves. Figures 17 and 18 are Arrhenius plots of E vs 1/T for the two groups of specimens oriented "with the grain" and "against the grain" respectively. Fairly good fits have been obtained with sets of parallel lines, indicating that the activation energy for

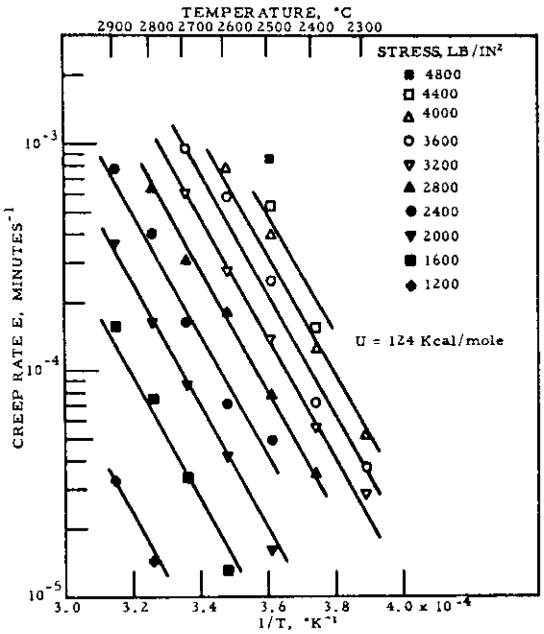

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Figure 17. Creep Rate E vs 1/T for Various Stresses. ATJ Graphite, with grain.

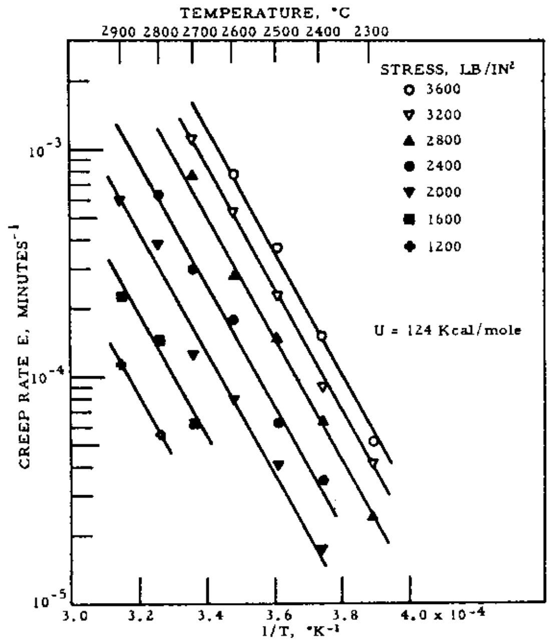

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Figure 18. Creep Rate E vs 1/T for Various Stresses. ATJ Graphite, against grain.

creep is independent of the applied stress. The activation energy obtained from the slopes of the lines is 124 Kcal/mole and is the same for both groups of specimens. This value is considerably higher than the 70 to 76 Kcal/mole activation energy which was obtained from the creep of grade ATJ graphite in flexure.[10] Specimens oriented "with the grain" were stronger than those oriented "against the grain" and were therefore subjected to higher maximum stress at each temperature. Over the range of stresses from 2000 lb/in^2 to 3600 lb/in^2, the steady-state creep rate at any given temperature and stress was lower for the specimen oriented "with the grain" than for the specimen oriented "against the grain," and the ratio of the steady-state creep rates for specimens with the two grain orientations was about 1.8. At lower stress values, the ratio was higher; it was equal to 2 at 1600 lb/in^2 and 3.5 at 1200 lb/in^2.

The stress dependence of the creep rate at each temperature is shown in Figures 19 and 20 for the groups of specimens with the two different grain orientations. The figures show that the stress dependence of the creep rate for both grain orientations is given by a power law in which the creep rate varies as the fourth power of the stress. Combining the temperature and stress dependence, the creep rate E can be expressed as

$$(5) \qquad E = \text{Const.} \times S^4 \exp[-U/RT]$$

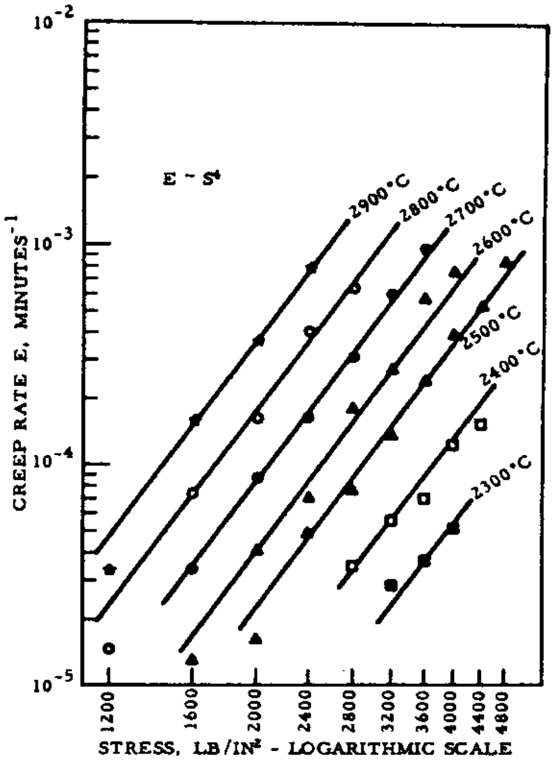

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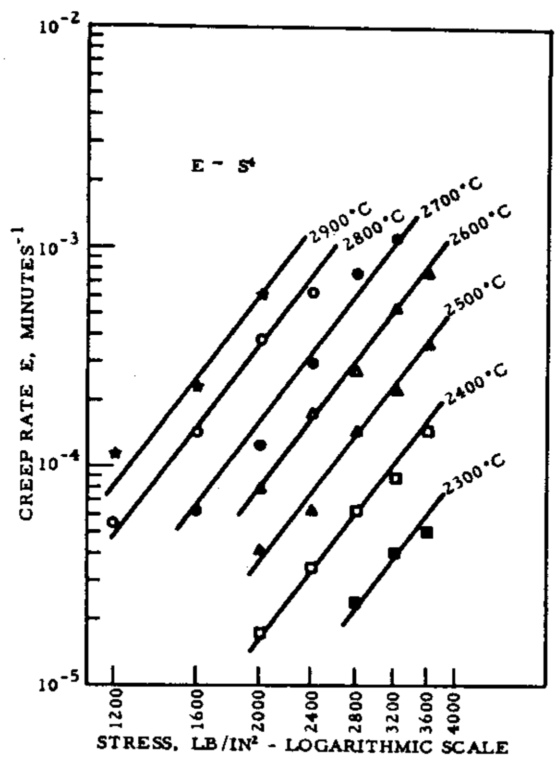
Figure 19. Creep Rate E vs Stress for Various Temperatures. ATJ Graphite, with grain.



N-2753

Figure 20. Creep Rate E vs Stress for Various Temperatures. ATJ Graphite, against grain.

where S is the stress, T is the absolute temperature, R is the general gas constant, and U is the activation energy, which is equal to 124 Kcal/mole. The fourth power stress dependence of the steady-state creep rate found in this investigation is close to that reported by Wagner, Driesner and Haskin[8] who determined the creep rate to be proportional to the stress to the 3.8 power. On the other hand, Martens et al,[4] have reported the creep rate to be proportional to the square of the stress.

One possible objection that may be raised to our treatment is that the creep rates, which have been determined from the slopes of the creep curves at the end of 90 minutes, may not truly be steady-state creep rates. Some longer tensile creep tests lasting up to six or seven hours have been reported which indicate that the creep rate continues to decrease with time.[2] The creep rate in these tests either decreases until failure occurs by rupture or the rate becomes constant after a few hours or so very slowly varying with time that it seems to be constant. It is typical, in fact, of graphite that there is no prolonged period of third-stage creep; when the creep rate does increase, it is always associated with crack formation and subsequent failure by rupture, and the period of increasing creep rate is generally no longer than about three minutes. The question of whether the creep rate as determined at the end of longer testing times would satisfy the same temperature and stress relationship as given in relation (5) cannot be fully answered at this time. There is evidence, however, from some four-hour creep tests on grade ATJ graphite*, using only one value of stress, that the creep rate does continue to decrease slightly but that the activation energy based on the creep rates at the end of four hours is the same 124 Kcal/mole.

The third term on the right-hand side of equation (2) represents, according to the viscoelastic model, the anelastic part of the creep. The parameter F was determined from the creep curve by taking the value of the X intercept at $t = 0$ of the extrapolated linear part of the creep curve and subtracting from it the value of D. Errors in D and E therefore contribute to errors in F. The value of F was found to increase with increasing temperature and stress. The stress dependence, shown in Figures 21 and 22, indicates that although there

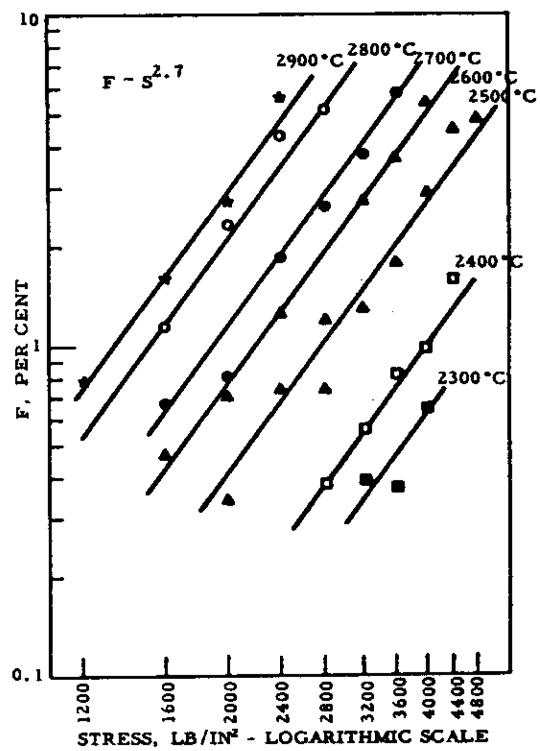


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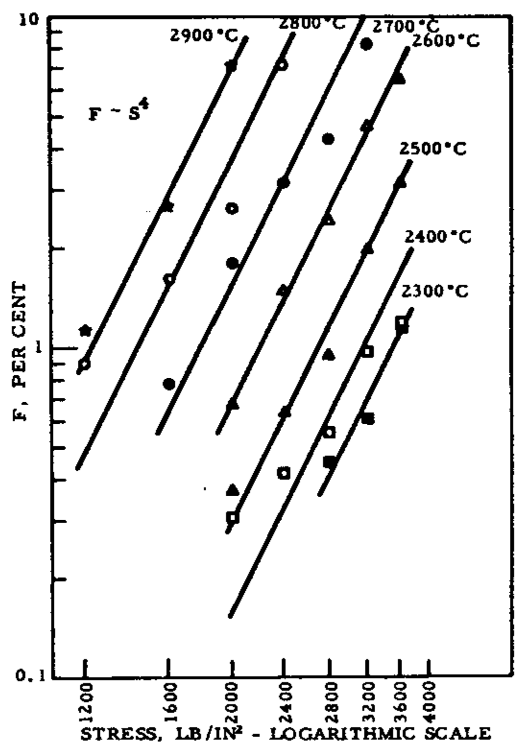
Figure 21. F vs Stress for Various Temperatures. ATJ Graphite, with grain.



N-2757

Figure 22. F vs Stress for Various Temperatures. ATJ Graphite, against grain.

is considerable scatter of the points about the straight lines, F can be approximated by a power law of the stress. For the case of the specimens oriented "against the grain," as shown in Figure 22, F varies with

*See Appendix I.

the same fourth power of the stress as does the parameter E. However, for the specimens oriented "with the grain," as shown in Figure 21, F varies with the 2.7 power of the stress.

The analysis indicates that the function f(t) as obtained from the data does not fit perfectly the exponential function given by the simple linear viscoelastic model. Some attempts were made to modify the model and thus change the form of f(t) in order to obtain better fits to the data. One attempt, suggested by the literature on linear viscoelastic materials, consists of replacing the parallel combination of a spring and dashpot with a series of such combinations. Since the total anelastic strain is equal to the sum of the strains of each parallel combination, the third term on the right-hand side of equation (2) must be replaced by a series of terms such that

$$(6) \qquad F \cdot f(t) = \Sigma F_i \, [1 - \exp(-t/\tau_i)] \, ,$$

where the parameter F is equal to the sum of the F_i, and relaxation times τ_i have been used instead of their reciprocals α_i. Analysis of the creep curves indicates that improved fits to the data can be obtained by using only two terms in the above series (6) such that there is a large member F_1 associated with a relaxation time τ_1 of the order of 10 to 20 minutes and a smaller member F_2 associated with a much shorter relaxation time τ_2 of one to two minutes. For purely mathematical reasons one would expect to obtain even better fits by increasing the number of terms in the series.

The term $F \cdot f(t)$ has generally been interpreted on the basis of the viscoelastic model as an anelastic term. As such, it should also describe the recovery of the strain when the load is removed. According to the model, one should observe in recovery the same relaxation time or distribution of relaxation times that is observed in creep, and all of the anelastic deformation should be recoverable after the load is removed. This interpretation is incompatible with experimental observations. Recovery after creep has been observed in several cases for long periods of time, indicating that recovery can continue for possibly several hours with a corresponding relaxation time or times of the order of hours. Also, only a fraction of what has been considered to be the anelastic part of the creep strain can be recovered completely. There are, therefore, two serious points of disagreement in the correlation of the anelastic terms in creep and recovery: they do not agree either in magnitude or in the distribution of relaxation times.

A further objection to using a linear viscoelastic model is that the parameters E and F are not linear functions of the stress. Therefore, another attempt at fitting the anelastic part of the creep curve and thus obtaining a different form for the function f(t) was made using a model which consisted of a parallel combination of a spring and dashpot, where the constants of the two elements were no longer assumed to be linear functions of the stress and strain or strain rate. The deformation of the spring was assumed to be proportional to an integral power of the stress and the deformation rate of the dashpot was assumed to be proportional to another integral power of the stress. Several different combinations of powers were tried, each of which gave a parameter F proportional to the fourth power of the stress and a slightly different function f(t).

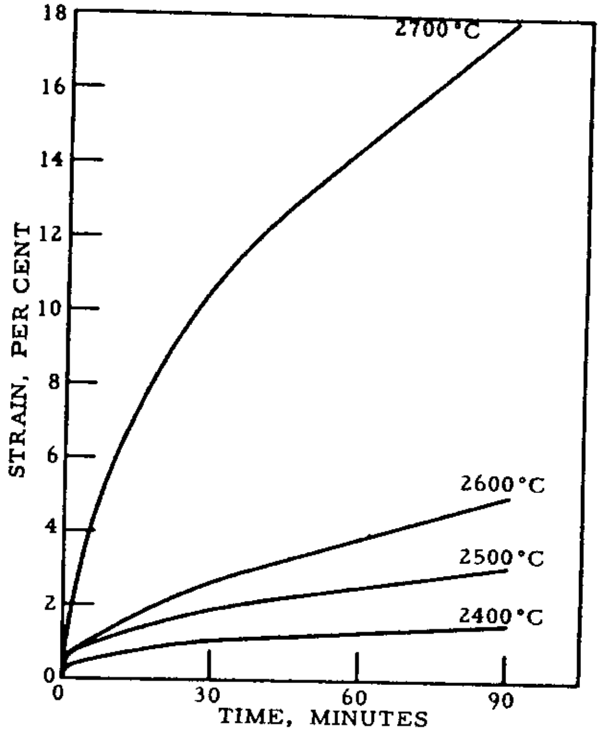
It was found that no great change in the form of the function f(t) was effected by raising or lowering by one the assumed power of one of the elements with an appropriate change in the power of the other element, and no single combination of powers stood out as being better than all others. The net result of this model for anelasticity is a function containing four arbitrary parameters, namely, the spring and dashpot constants and the two exponents of the stress. These functions f(t) were used to fit the data for the specimens oriented "against the grain." There were a few excellent fits of the data, but no single combination of parameters gave a function which would satisfy all of the data. The data for specimens oriented "with the grain" require a different combination of parameters because of the different stress dependence of F, and no attempt was made to fit these data. The model at the present time is useful because it gives a good qualitative description of the creep, but there still remains the problem of correlating the anelastic part of the creep curve to the recovery curve and the much more general problem of attaching physical meaning to the model parameters.

3.2 Grade ZTA Graphite

Grade ZTA graphite is a high-density, fine-grain, highly anisotropic graphite whose grain size is comparable to that of grade ATJ graphite. Two different pieces of this graphite were used. Piece No. FE-1721 was thick enough to furnish specimens oriented "against the grain," but there was considerable variation in density in this piece from top to bottom. Some specimens oriented "with the grain" were cut from this piece in order to observe the effect of density variation on the creep behavior of this graphite. Piece No. FE-1745 was more uniform in density, and specimens oriented "with the grain" were taken from this piece in order to determine the temperature dependence of the creep for constant density.

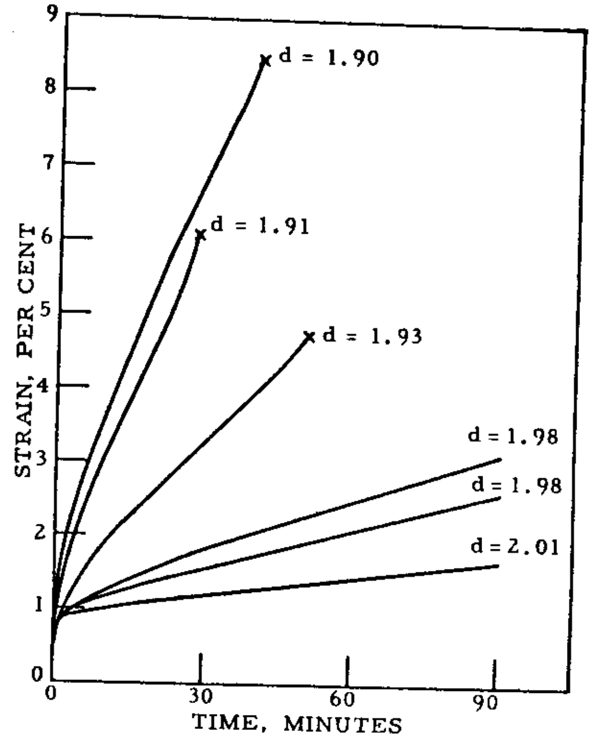
The density of piece No. FE-1721 varied from 1.89 to 2.01 g/cc from top to bottom. The specimens oriented "against the grain" all had the same density variation from end to end, with an average density in the gauge length of about 1.95 g/cc. Figure 23 shows the creep curves obtained for different specimens between 2400°C and 2700°C with an applied stress of 2000 lb/in^2. The breaking strength of the grade ZTA graphite with this orientation was between 2400 lb/in^2 and 2800 lb/in^2 at a temperature of 2500°C. The 2000 lb/in^2 stress was therefore close to the breaking strength. Comparison with the corresponding creep curves for grade ATJ graphite of the same grain orientation indicates that the grade ZTA graphite exhibits considerably greater creep.

On the other hand, the specimens oriented "with the grain" exhibit very high creep resistance. Figure 24 shows creep curves obtained for specimens cut from piece No. FE-1721 and oriented "with the grain." All tests were run at a temperature of 2800°C with a stress of 4400 lb/in^2. The creep decreased as the density increased, and the curves demonstrate how rather small differences in density can account for very large differences in creep behavior. In this particular figure, unlike any other in this paper, most of the curves were obtained on specimens which were previously tested at



N-3036

Figure 23. Strain vs Time at 2000 lb/in^2 for Various Temperatures. ZTA Graphite (FE-1721), against grain. Average Density 1.95 g/cc

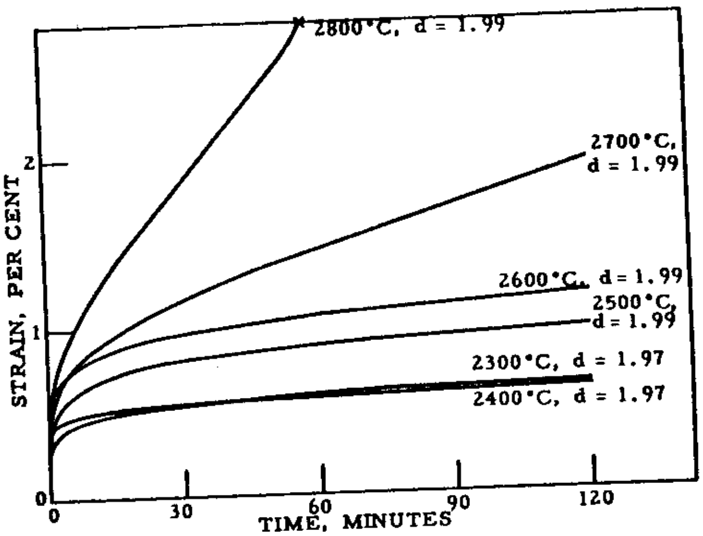


N-3032

Figure 24. Strain vs Time at 2800°C and Stress of 4400 lb/in^2 for Specimens with Different Densities. ZTA Graphite (FE-1721), with grain.

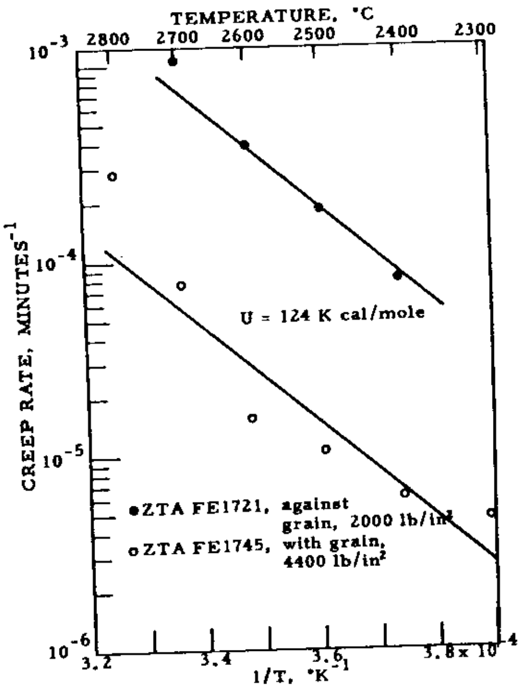
either lower temperature or lower stress prior to these tests.

Figure 25 shows some creep curves which were obtained with specimens cut from piece No. FE-1745 and oriented "with the grain." Density here varied between 1.97 and 1.99 g/cc. The tests were run at temperatures ranging from 2300°C to 2800°C with a stress of 4400 lb/in^2. There is sufficient variation of density within a block of this graphite (and most other graphites as well) to make it extremely difficult to reproduce any of these creep curves exactly. The overlap of the curves at 2300°C and 2400°C is not a real effect, but is rather a demonstration that the creep behavior is not much different at these temperatures. A comparison of the creep curves of ZTA graphite oriented "with the grain" at 2400°C and 2500°C with the corresponding creep curves of ATJ graphite shows that ZTA graphite with this orientation underwent about one-tenth of the maximum strain given the ATJ graphite at the end of the creep test. Under a stress of 4400 lb/in^2, the steady-state creep rate of ZTA graphite having a density of 1.98 g/cc was smaller by a factor of 2.5 than that of ATJ graphite having the same grain orientation and a density of 1.72 g/cc. The ZTA graphite can sustain a higher stress than ATJ graphite, but it fails after a lower maximum strain.



N-3608

Figure 25. Strain vs Time at 4400 lb/in^2 for Various Temperatures. ZTA Graphite (FE-1745), with grain.



N-3609

Figure 26. Creep Rate vs 1/T. ZTA Graphite.

An Arrhenius plot of the steady-state creep rate vs 1/T for the grade ZTA specimens is shown in Figure 26. For the specimens oriented "against the grain" an activation energy of 124 Kcal/mole, identical to that of the grade ATJ graphite, was obtained. The points for the specimens oriented "with the grain" show a considerable amount of scatter, due either to density differences or to variations in grain orientation, but it seems reasonable that the creep rates for this orientation follow the same activation energy.

Appendix II contains a sketch of piece No. FE-1721 and a tabulation of the densities and creep rates for all of the tests run on this material. The stress dependence of the creep rate at 2500°C was investigated for FE-1721 specimens oriented "against the grain" and for some FE-1745 specimens oriented "with the grain." These data are given in Appendix II. No definite conclusions about the stress dependence of the creep rate of grade ZTA graphite could be drawn from the data.

3.3 Extruded Graphite Rods

A series of tensile creep tests was made on graphite rods which were prepared from different mixtures of the same coke filler particles and of the same binder. The filler particles were a Continental petroleum coke flour and the binder was coal tar pitch. This coke is of dendritic (needle) structure, and the particle sizes were all less than 0.0164 inch diameter. The binder was varied from 30 to 38 parts by weight to 100 parts of coke in the green mixes, which also contained three to four parts of lubricating oil. Mixing conditions were maintained as uniform as possible for all mixes and 9/16-inch diameter rods were extruded from each of the mixes. The rods, baked in closed saggers to 800°C, were graphitized in a single large furnace to 3000°C. After graphitization, the rods were machined into 1/2-inch diameter cylinders and densities were determined. From each set of rods of a given binder content, rods having approximately the same density were selected from which specimens were prepared. The rods were of relatively poor graphite by commercial standards; the grain was coarse as compared to a standard fine-grain graphite such as ATJ, and the densities were relatively low. The object of this series of tests was to take several graphites made from the same filler coke and binder in order to observe the influence of density on the creep behavior and to see what effect the varying proportions of filler and binder coke had on the creep.

The densities or ranges of densities of the different groups of graphite rods are shown in Table 2. It was found that the density was about

Table 2. Bulk Density of Extruded Graphite Rods

No. of Parts Binder to 100 parts by Weight of Coke in Green Mix	Density g/cc
30	1.48 - 1.49
32	1.44
34	1.44
36	1.44 - 1.45
38	1.38

1.44 g/cc for the intermediate binder contents; it was higher for the lower binder content, and lower for the higher binder content. The net effect on the effective filler density d_0, which is the ratio of the weight of the filler particles to the total volume, was to cause it to increase as the binder content was decreased. In other words, the particles were packed more tightly together as the binder content was decreased.

Figures 27 to 31 show the creep curves for the specimens grouped according to binder content. Each of the figures presents the results of tests at temperatures ranging from 2300°C to 2700°C for a stress of 2000 lb/in^2. Where there is no creep curve at 2700°C, as in the figures for the two lower binder contents, the specimen broke when load was applied at that temperature. In addition, a series of tests in which the stress was varied was run at 2500°C for each group of specimens with a different binder content. Figure 32 shows the results of tests run on the group of specimens

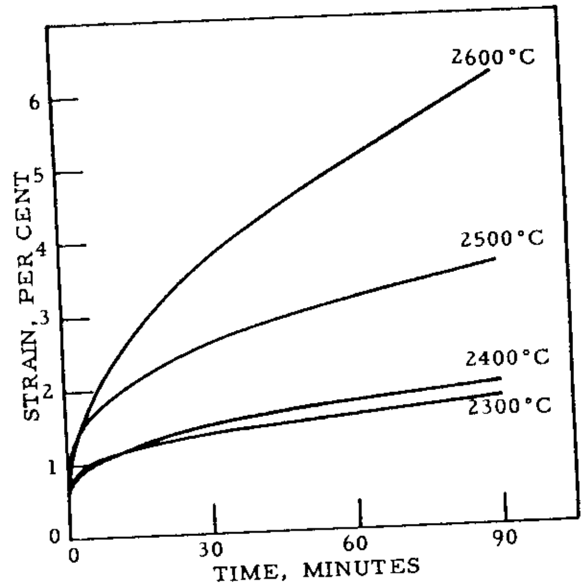


Figure 27. Strain vs Time at 2000 lb/in² for Various Temperatures. Extruded Graphite, with grain. (30 parts binder to 100 parts coke in green mix.) N-3508

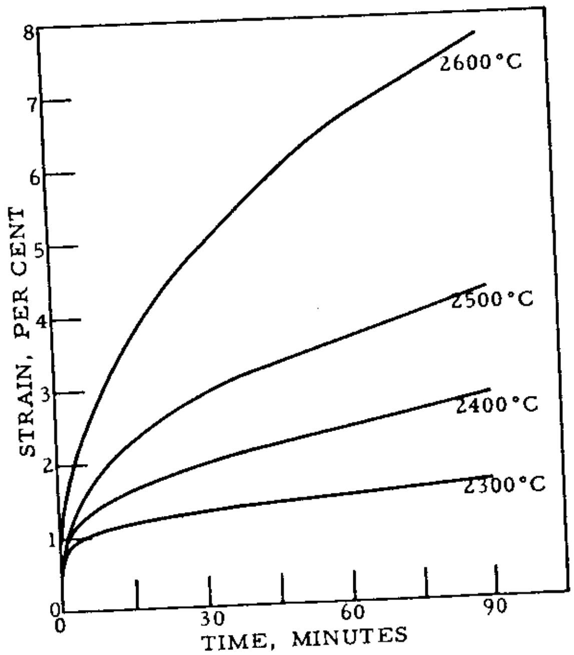


Figure 28. Strain vs Time at 2000 lb/in² for Various Temperatures. Extruded Graphite, with grain. (32 parts binder to 100 parts coke in green mix.) N-3510

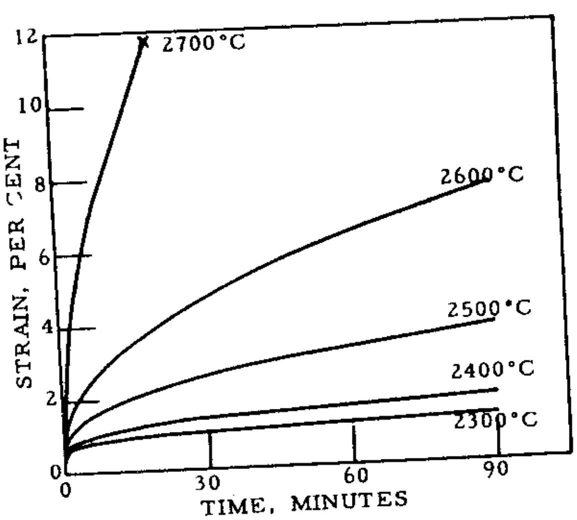


Figure 29. Strain vs Time at 2000 lb/in² for Various Temperatures. Extruded Graphite, with grain. (34 parts binder to 100 parts coke in green mix.) N-3512

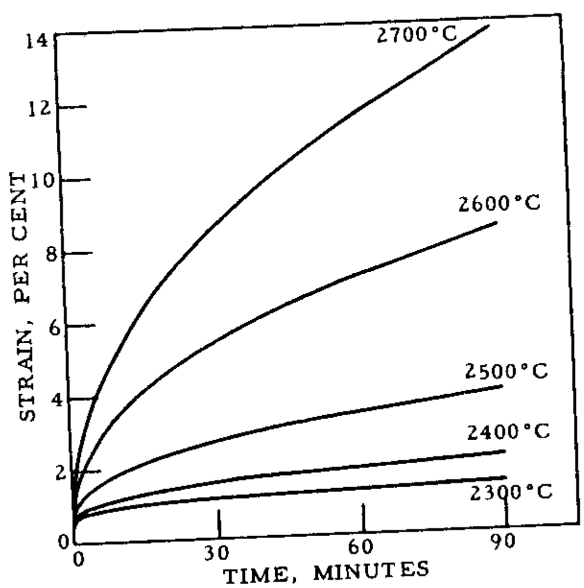
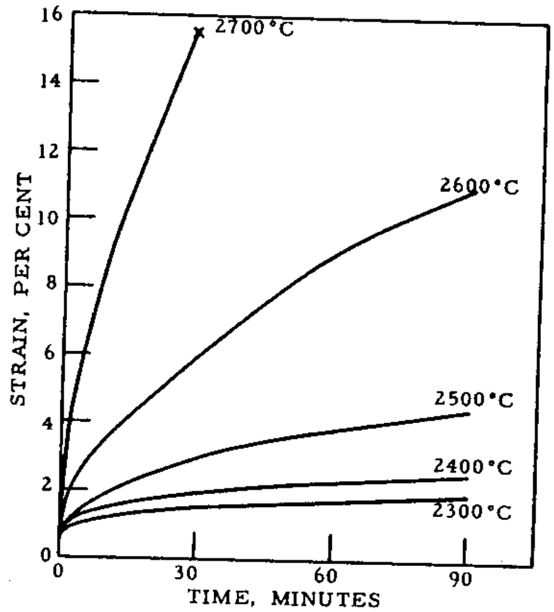
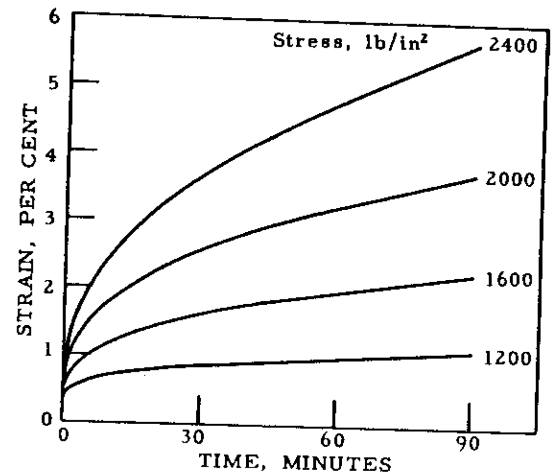


Figure 30. Strain vs Time at 2000 lb/in² for Various Temperatures. Extruded Graphite, with grain. (36 parts binder to 100 parts coke in green mix.) N-3514



N-3516

Figure 31. Strain vs Time at 2000 lb/in^2 for Various Temperatures. Extruded Graphite, with grain. (38 parts binder to 100 parts coke in green mix.)



N-3513

Figure 32. Strain vs Time at 2500°C for Various Stresses. Extruded Graphite, with grain. (34 parts binder to 100 parts coke in green mix.)

made with 34 parts binder. The results are typical of those obtained for specimens having other binder contents.

A comparison of Figures 27 through 31 shows that the creep depends strongly on density, decreasing as the density increases; however, the creep is not very sensitive to the relative proportions of filler and binder carbon. The figures indicate that for graphites having the same bulk density but different ratios of filler-to-binder carbon (as, for example, the rods made with 32, 34, and 36 parts binder), there is not much difference between the creep curves obtained under the same conditions of temperature and stress. From a comparison of the curves for the specimens made with 32 parts binder with those made with 34 and 36 parts binder, there is a slight indication that, for the same density, the creep might be a little lower if the carbon is distributed more in the form of binder carbon than in the form of filler carbon. However, comparison of the creep curves for the specimens made with 34 parts binder with those for the specimens made with 36 parts binder indicates that the situation might be quite the reverse.

The temperature dependence of the steady-state creep rate is shown in Figure 33. A straight line is drawn on the plot which has a slope corresponding to an activation energy of 124 Kcal/mole. A fairly good fit to the points is obtained at all but the highest and lowest temperatures. The

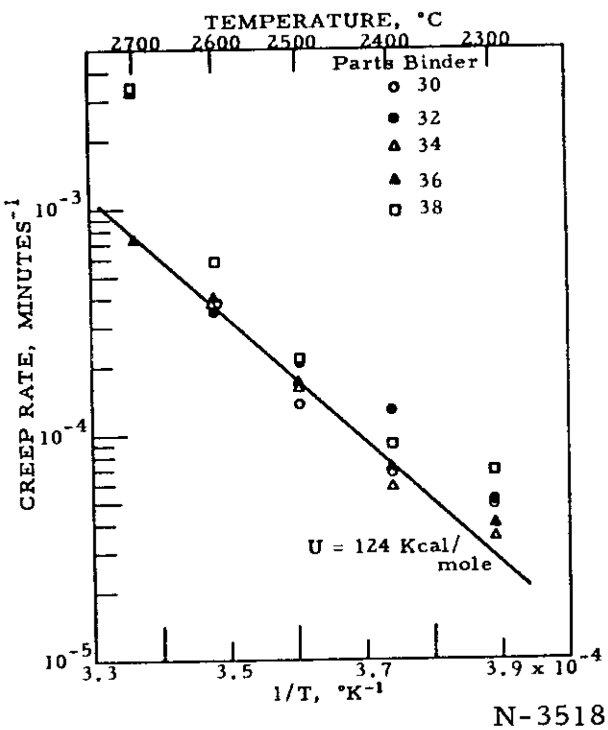


Figure 33. Creep Rate vs 1/T for Stress of 2000 lb/in². Extruded Graphite, with grain, various binder contents.

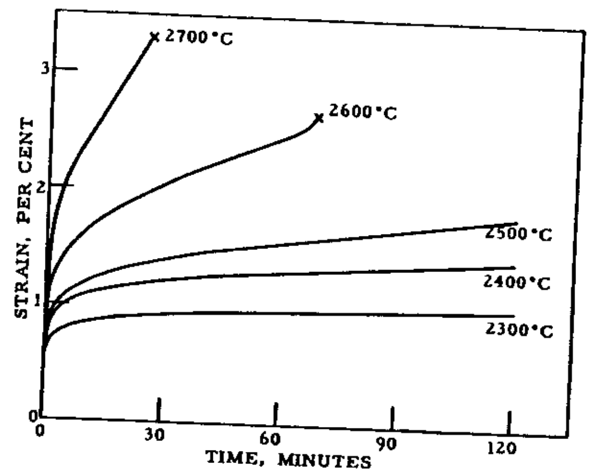
activation energy is the same as that which was obtained for the grade ATJ and ZTA graphites. The stress dependence of the steady-state creep rate at 2500°C was also investigated for each of the groups of specimens over the range of 1200 lb/in² to 2400 lb/in². Over this range of stresses, the creep rate was found to vary with the stress S approximately as S^n with n equal to 2.6. It may be recalled that in the case of grade ATJ graphite, n was found to be equal to 4.

By fitting the data to equation (2), the parameter F was also evaluated for each of the creep curves. The stress dependence of F could not be determined with any great accuracy from the creep curves which were obtained at 2500°C, but F could be approximated by a function of the form S^m where m lies between 2.2 and 2.9. F also increased with increasing temperature, and on an Arrhenius plot, F may be said to have an "activation energy" of about 100 K cal/mole.

3.4 Grade CEP Graphite

Grade CEP graphite is a molded lampblack-base graphite. It is graphitized to 3000°C and is very nearly isotropic in its mechanical, thermal, and electronic properties. The material which was tested had a density of 1.58 g/cc, and all of the tensile specimens were oriented in the "with the grain" direction as determined by the direction of the force applied during molding. No specimens were prepared which were oriented "against the grain," but flexural creep tests have indicated that the creep curves for this graphite should be almost independent of the orientation.(11)

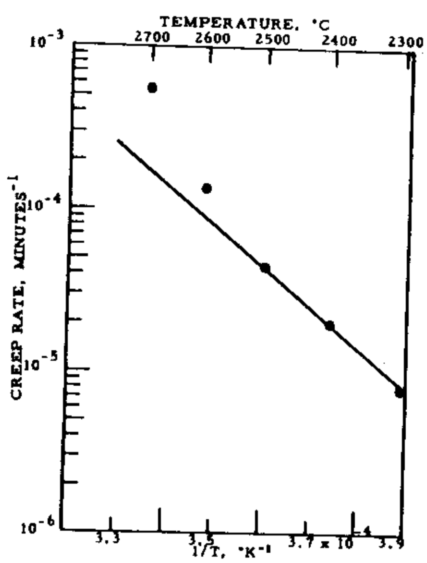
Figure 34 shows the creep of CEP graphite at temperatures ranging from 2300°C to 2700°C under a stress of 3600 lb/in², which is close to the high temperature breaking strength of this graphite. The grade CEP graphite is lower in density than grade ATJ graphite and has a lower tensile strength, but it undergoes much less creep for a given stress than ATJ graphite with either grain orientation. Only ZTA graphite oriented "with the grain" showed less creep for a given combination of temperature and stress than did CEP graphite.



N-4005

Figure 34. Strain vs Time at 3600 lb/in² for Various Temperatures. CEP graphite, with grain.

Figure 35 is a plot of the creep rate vs 1/T for the specimens whose creep curves are shown in the previous figure. Again, a line which corresponds to an activation energy of 124 Kcal/mole has been drawn on the Arrhenius plot. This line gives a fair fit to the points for the three lower temperatures. At the higher temperatures the line does not fit, but the points at these temperatures undoubtedly do not represent true steady-state creep rates because the specimens broke in relatively short times. It is characteristic of all creep data which has been observed in graphite that when a specimen breaks in less time than that of a standard creep test (i.e., in less than 90 to 120 minutes) the final creep rate is always greater than that which would be expected from an extrapolation of the line on the Arrhenius plot which fits the data at the lower temperatures.



N-4006

Figure 35. Creep rate vs 1/T for Stress of 3600 lb/in². CEP graphite, with grain.

The problem of obtaining accurate determinations of the creep rates of grade CEP graphite presents the same type of experimental difficulty as is encountered in the determination of creep rates of grade ZTA graphite. The graphite will sustain very little creep deformation and the resulting creep rates are relatively low. As the creep rate decreases, the possible error in the measurement of the creep rate increases. Some additional creep tests, not shown here, were run on CEP graphite specimens over the same temperature range with a lower stress. While the results are not conclusive, the data indicate that the activation energy for creep as determined from the steady-state creep rates in tension is the same for this graphite as for all of the other graphites which were tested.

4. DISCUSSION

Most theories of creep are based on the motion of dislocations.[12] In graphite, as in other hexagonal crystals, slip by dislocation motion can only take place along the basal planes. In polycrystalline materials creep may also take place by motion along grain boundaries; but in metals the contribution of grain boundary creep is generally considered to be less than 10 per cent of the total creep.[13] The most generally accepted theories of creep are based on the concept of dislocation climb as proposed by Mott.[14] Weertman[15, 16] has developed a theory based on a model of dislocation climb which predicts a fourth power stress dependence such as was found for graphite; but Weertman[16] states that his model is not applicable to the creep of hexagonal crystals even if nonbasal slip occurs because it is impossible to form immobile dislocations in such a system. Mott[14] had previously come to the same conclusion and had stated that in hexagonal crystals the slip distance (the distance a dislocation moves before it is pinned) for the edge dislocations must be the same as the linear dimension of the crystal and that apart from oxide layers there is nothing to prevent their passing right out of the crystal. Direct observations of the motion of dislocations in single crystals of graphite in the electron microscope have led to the conclusion that the dislocations move quite freely through the crystal and that the closest spacing of dislocations is of the order of 1000 Å.[17] Since the sizes of the largest crystallites in polycrystalline graphite are of the order of several thousand Angstroms, one would expect to find few, if any, immobile dislocations within the crystallites. If a dislocation is then assumed to spend within the crystallite only the very short time necessary for it to travel from one boundary to another, the rate controlling mechanism must be the rate of generation of dislocations at the boundaries, which must act as sources and sinks for the dislocations. The boundaries may also be regions which store the internal stresses that account for the long lasting recovery which has been observed in graphite. Since there is no direct evidence at the present time of grain boundary motion in graphite, these remarks concerning the influence of the grain boundaries must be considered to be only speculative.

Dorn[18] has shown that the activation energy for steady-state creep in metals is about equal to that for self-diffusion. Dienes[19] has performed theoretical calculations of activation energies for self-diffusion in graphite by three different mechanisms: vacancy diffusion, direct interchange of atoms, and diffusion by interstitial atoms. The calculations were based on the value of 124 Kcal/mole for the heat of sublimation of graphite. Kanter,[20] using the more presently acceptable value for the heat of sublimation of 170 Kcal/mole,[21, 22] corrected the values of the activation energies given by Dienes. Table 3 shows the results obtained by Dienes and Kanter. In experiments on the diffusion of C^{14} through crystals of natural graphite over the temperature range of 1995°C to 2347°C, Kanter[20] obtained an activation energy of 163 ± 12 Kcal/mole. In experiments on the diffusion of C^{14} through polycrystalline graphite (AUF graphite) over the temperature range of 1835°C to 2370°C, Feldman et al[23] assumed the diffusion to be due to a combination of volume diffusion and grain boundary diffusion. Since Dienes had concluded that the preferred mechanism for self-diffusion in graphite was

Table 3. Energy Parameters for Diffusion in Graphite

	Kcal/mole	
	Dienes	Kanter
Activation Energy for Direct Interchange	90.4	113
Activation Energy for Motion of a Vacancy	71.4	93
Energy of Formation of a Vacancy	119.4	170
Activation Energy for Self-Diffusion by a Vacancy Mechanism	190.8	263
Activation Energy for Motion of an Interstitial	---	---
Energy of Formation of an Interstitial	417.0	467
Activation Energy for Self-Diffusion by an Interstitial Mechanism	>417.0	>467

Activation Energy for Creep = 124 Kcal/mole.

the direct interchange mechanism, Feldman et al used Dienes's value of 90 Kcal/mole for the activation energy for volume diffusion to calculate on the basis of this value and their experimental data a value of 75.4 Kcal/mole for the activation energy for grain boundary diffusion. By using Kanter's corrected value of 113 Kcal/mole for the activation energy for volume diffusion, the above value for the activation energy for grain boundary diffusion can be corrected to yield a value of 86.6 Kcal/mole. However, both of the above values for the activation energy for grain boundary diffusion may be in error because of the possible wrong choice of an activation energy for volume diffusion. Kanter [20] states that the experimental values for the activation energy for diffusion in graphite are compatible with existing theoretical calculations for either the vacancy or the direct interchange mechanism. On the basis of independent calculations, Baker and Kelly[24] believe that self-diffusion in graphite occurs by a vacancy mechanism and they calculate an activation energy for self-diffusion of 149 ± 33 Kcal/mole. If the activation energy for creep is to be related to that for self-diffusion, it is most likely related to self-diffusion by a vacancy mechanism because the direct interchange mechanism does not lead to the transport of matter. The value of 124 Kcal/mole which was obtained for the activation energy for creep can be seen to fall within the range of activation energies which have been calculated for self-diffusion, but it is impossible at this time to deduce from this value and present theories of creep any specific mechanism for the creep of graphite.

5. SUMMARY AND CONCLUSIONS

Tensile creep tests, usually of 90 minutes duration, were conducted on several types of graphite under conditions of constant load and constant temperature. The materials which were tested were fine-grain grade ATJ graphite, high density grade ZTA graphite, some coke-base extruded graphite rods having low density, and lampblack-base grade CEP graphite. The testing temperature varied from 2300°C to 2900°C. The results led to the following conclusions:

1. For a given stress, the creep rate increases as the temperature is increased. From the temperature dependence of the creep rate, an activation energy of 124 Kcal/mole was obtained, which is the same for all of the materials which were tested. The activation energy was found to be independent of the grain orientation and the crystalline structure of the graphite. In flexural creep, on the other hand, a different activation energy was obtained for each grade of graphite, and all of the values for activation energies which were obtained were lower than that obtained for the tensile creep tests.(11)

2. At a given temperature, the creep rate of grade ATJ graphite was found to vary with the fourth power of the stress for specimens oriented both "with the grain" and "against the grain." On the other hand, the creep rates of the extruded graphites oriented "with the grain" were found to vary with the 2.6 power of the stress. No stress dependence was determined for the grade ZTA and CEP graphites.

3. The creep strain and creep rate are greater for specimens oriented "against the grain" than for specimens oriented "with the grain." The differences in creep behavior for the two orientations become greater as the anisotropy of the graphite increases, as can be seen from a comparison of the creep curves of grade ATJ graphite with those of the more highly anisotropic grade ZTA graphite. Under the same conditions of temperature and stress, the steady-state creep rate for a specimen of ATJ graphite oriented "against the grain" was 1.8 times as great as that of a specimen oriented "with the grain" over most of the range of stresses.

4. The equation based on the model of a linear viscoelastic material gives the best fit and most satisfying description of the creep data of all the equations which were used. However, the model must be modified in order to satisfy the data. Since the parameters of the equation which represent the creep rate and the magnitude of the anelastic part of the strain vary with powers of the stress, the model elements must be made non-linear. By adjusting the stress dependence of the model constants, some improvements can be made in the fit of the equation to the data. However, no unique combination of constants for the model elements has been found to satisfy all of the creep data, and no correlation has yet been made between the elements of the model and physical mechanisms giving rise to the creep. The main advantage of retaining the model at the present time lies in its use as a phenomenological description of the data.

5. For graphites with different proportions of filler and binder, the magnitudes of the creep strain and the creep rate depend on the average bulk density, remaining insensitive to the relative proportions of filler and binder carbon. As the density increases, the creep strain and creep rate for specimens oriented "with the grain" both decrease. The activation energy and the stress dependence of the creep rate were found to be independent of the density.

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APPENDIX I

EXTENDED CREEP TESTS

Some four-hour long creep tests were run on specimens of ATJ graphite oriented "with the grain." For each test, a stress of 3600 lb/in^2 was applied. The temperature was varied in this series of tests from 1700°C to 2700°C. Figure 36 is an Arrhenius plot of the creep rate vs 1/T in which the rates at the end of 90 minutes and at the end of four hours are both plotted. There are only two exceptions: at 2700°C there is only a 90-minute rate because the specimen broke shortly after 90 minutes under load, and at 2600°C the rate at the end of three hours is plotted. In general, the creep rate did decrease in the interval between 90 minutes and four hours, except at the very lowest temperatures where a condition of steady-state creep was reached after a relatively short period of time. Two conclusions can be drawn from these results. First, above 2300°C the activation energy as determined from the four-hour creep rates is the same as that which was determined from the 90-minute creep rates. Secondly, the activation energy for creep appears to decrease below about 2100°C.

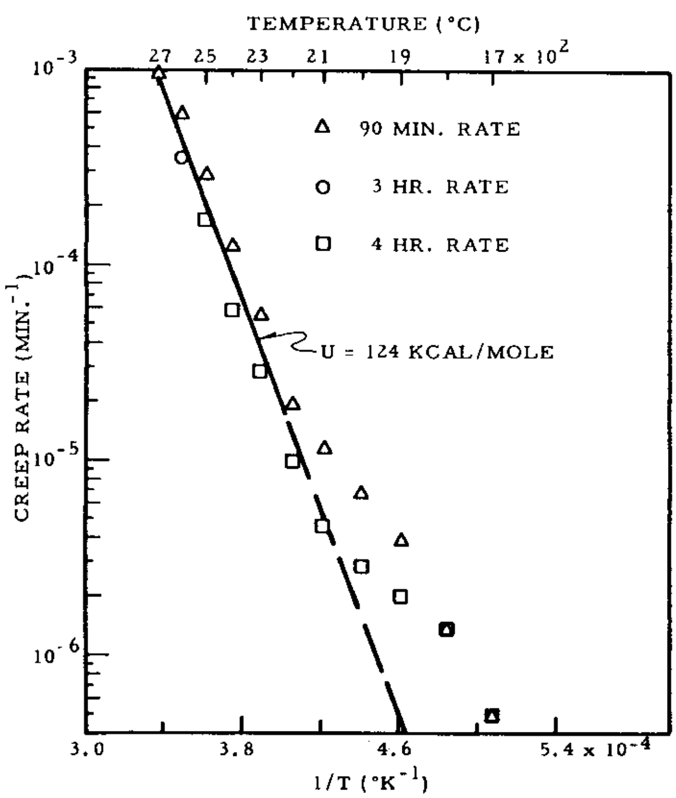

Figure 36. Creep Rate vs 1/T. ATJ Graphite, with grain. Stress = 3600 lb/in^2.

APPENDIX II

TENSILE CREEP DATA FOR GRADE ZTA GRAPHITE

A sketch of the block of ZTA graphite, FE-1721, is shown in Figure 37.

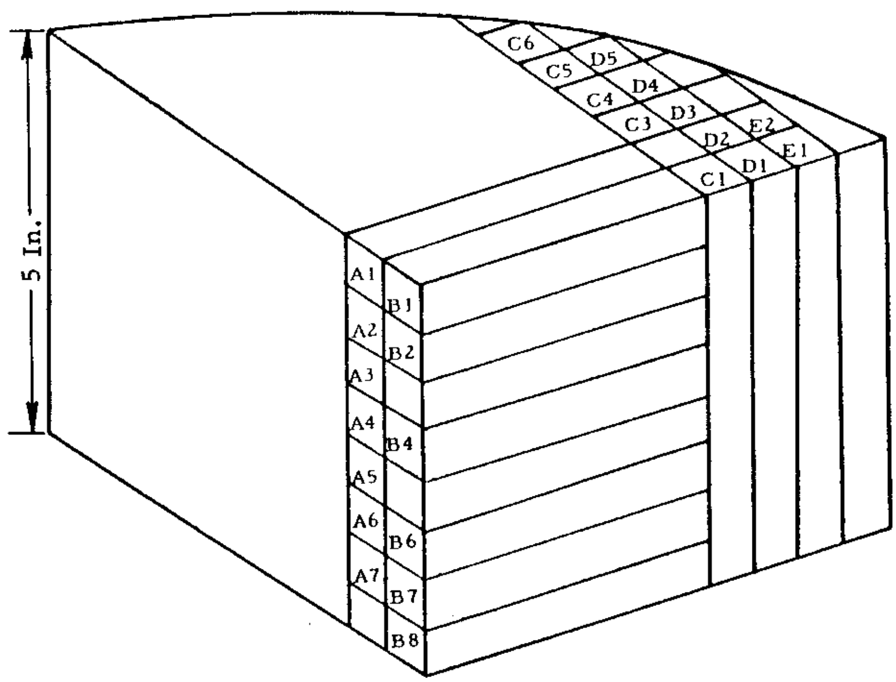

Figure 37. Sketch of ZTA Graphite Piece No. FE-1721.

The block was five inches thick, and specimens having both grain orientations were prepared. Table 4 summarizes the creep data obtained for the specimens oriented "with the grain" and Table 5 summarizes the data obtained for the specimens oriented "against the grain."

Table 6 gives the creep rates which were obtained for specimens of ZTA graphite, FE-1745, oriented "with the grain" for various values of stress at 2500°C.

Table 4. Creep Data for ZTA Graphite, FE-1721, With the Grain. Creep Tests Were 90 Minutes Long.

Specimen No.	Density (g/cc)	Test No.	Temp. (°C)	Stress (lb/in^2)	Creep Rate (min^{-1})
B 1	1.89	1	2400	4400	0.352×10^{-4}
		2	2500	4400	0.566×10^{-4}
		3	2600	4400	1.28×10^{-4}
		4	2700	4400	5.12×10^{-4}
A 2	1.90		2800	4400	15.8×10^{-4}
B 2	1.90	1	2500	4400	0.453×10^{-4}
		2	2600	4400	1.09×10^{-4}
		3	2700	4400	4.55×10^{-4}
A 3	1.91	1	2600	4400	1.28×10^{-4}
		2	2800	4400	16.0×10^{-4}
A 4	1.93	1	2600	3600	0.207×10^{-4}
		2	2800	4400	6.45×10^{-4}
B 4	1.94		2700	4400	1.91×10^{-4}
A 5	1.96		2600	2800	0.139×10^{-4}
A 6	1.98	1	2600	4400	0.278×10^{-4}
		2	2700	4400	0.449×10^{-4}
		3	2800	4400	1.60×10^{-4}
B 6	1.98	1	2500	4400	0.255×10^{-4}
		2	2800	4400	2.27×10^{-4}
B 7	2.00	1	2500	4400	0.182×10^{-4}
		2	2600	4400	0.299×10^{-4}
		3	2700	4400	0.366×10^{-4}
		4	2800	4400	2.10×10^{-4}
B 8	2.01	1	2800	3600	0.136×10^{-4}
		2	2800	4000	0.290×10^{-4}
		3	2800	4400	0.860×10^{-4}
		4	2800	4800	3.2×10^{-4}

Table 5. Creep Data for ZTA Graphite, FE-1721, Against the Grain. Creep Tests Were 90 Minutes Long.

Specimen No.	Density (g/cc)	Test No.	Temp. (°C)	Stress (lb/in^2)	Creep Rate (min^{-1})
C 1	1.95	1	2500	1600	0.308×10^{-4}
C 3	1.95	1	2500	1200	$\sim 0.2 \times 10^{-4}$
C 5	1.95	1	2500	2000	1.82×10^{-4}
D 2	1.95	1	2500	2400	6.8×10^{-4}
D 3	1.95	1	2700	2000	11.0×10^{-4}
D 4	1.95	1	2400	2000	0.800×10^{-4}
D 5	1.95	1	2600	2000	3.96×10^{-4}

Table 6. Creep Data for ZTA Graphite, FE-1745, With the Grain. Creep Tests Were 120 Minutes Long. T = 2500°C

Density (g/cc)	Stress (lb/in^2)	Creep Rate (min^{-1})
1.98	3200	3.33×10^{-6}
1.98	3600	6.0×10^{-6}
1.98	4000	1.92×10^{-5}
1.97	4000	1.1×10^{-5}
1.99	4400	1.04×10^{-5}
1.97	4800	2.86×10^{-5}