

FOREWORD

This report was prepared by the Carbon Research Laboratory at the University of Buffalo, Buffalo, New York under USAF Contract No. AF 33(616)-7791. This contract was initiated under Project No. 7350, "Refractory Inorganic Nonmetallic Materials", Task No. 735002, "Refractory Inorganic Nonmetallic Materials: Graphitic". The work was administered under the direction of the Directorate of Materials and Processes (Materials Central), Deputy Commander/Technology, Aeronautical Systems Division, with Messrs. K. S. Mazdidasni and C. A. Pratt, Jr. as project engineers.

This report covers work conducted from November 1960 to January 1962.

The authors would like to thank Mr. J. Distanto for collaboration on one phase of the project.

contrails.iit.edu

ABSTRACT

In continuation of the work on the elastic properties of carbons, families of curves of the dependence of Young's modulus on temperature and on the heat treatment have been obtained for four basic types of carbons, using the shortly completed high temperature equipment. The decisive influence of filler type has been established. As a result of gained experience, improvements in the associated electronics were introduced. The amplitude dependence of the internal friction and dynamic Young's modulus were, for the first time, investigated at room temperature for vibrations in the sonic range of frequencies for several types of carbons. Reproducibility has been achieved in measurements of thermal conductivity with the new high temperature test apparatus and families of curves of the dependence of the conductivity on temperature and on heat treatment obtained for soft carbon. A new technique of determining the heat conductivity of carbons without internal heat generation has been tried and found to work up to 1800°C. Improved results were obtained with the transient state technique of determination of the heat diffusivity by using a new fast responding pyrometer and a new improved apparatus for the "steady" sinusoidal wave technique constructed.

PUBLICATION REVIEW

This technical documentary report has been reviewed and is approved.

FOR THE COMMANDER:



W. G. RAMKE
Chief, Ceramics and Graphite Branch
Metals and Ceramics Laboratory
Directorate of Materials and Processes

TABLE OF CONTENTS

	Page
I. INTRODUCTION	1
II. ELASTIC PROPERTIES AT ROOM TEMPERATURE	4
A. Room Temperature Elasticity Apparatus	4
B. Studies of Young's Modulus at Room Temperature	4
III. ELASTIC PROPERTIES AT HIGH TEMPERATURE	8
A. Apparatus and Samples	8
B. Young's Modulus of Graphite Rods at High Temperature	8
C. Changes in Young's Modulus of Carbon Rods during the Heat Treatment Process	9
D. Improved Electronics for the High Temperature Elasticity Apparatus	14
IV. MECHANICAL ENERGY LOSSES	18
A. Experimental Method	18
B. Specimens	19
C. Results	20
1. Graphite	22
2. Baked carbon	23
3. Glassy carbon	23
D. Concluding Remarks	23
V. THERMAL CONDUCTIVITY MEASUREMENTS	24
A. Tests to Establish Reproducibility	24
B. Test to Determine Effect of Heat Treatment	27
C. Measurements without Internal Heat Generation	30
VI. THERMAL DIFFUSIVITY	33
A. Introduction	33
B. Transient Heat Flow Method	33
C. "Steady" Sinusoidal Heat Flow Method	40
VII. REFERENCES	45

LIST OF FIGURES

Figure		Page
1	Block diagram of the New Electronics Associated with the Room Temperature Elasticity Apparatus . . .	4
2	Electronic Circuit for the New Electronics Associated with the Room Temperature Elasticity Apparatus	5
3	Young's Modulus vs. Heat Treatment Temperature for Pitch Impregnated Rods	7
4	Young's Modulus vs. Ambient Temperature for Four types of Graphitized Carbon Bodies	10
5	Typical Curve for the Dependence of Young's Modulus on Temperature for a Soft Filler-Soft Binder rod having an Original Heat Treatment Temperature of 1800°C.	11
6	Young's Modulus as a Function of the Ambient Temperature for a 1/2 inch Diameter Extruded National Carbon Company Baked Carbon for Increasing Temperatures	12
7	Young's Modulus vs. Ambient Temperature for a 1/2 inch Diameter Extruded Laboratory-Prepared Hard Filler-Hard Binder Baked Carbon for Increasing Temperatures	13
8	Electronic Circuit of 400 c/s Oscillator and Chopper Driver Amplifier Associated with the High Temperature Elasticity Apparatus	15
9	Amplifier and Phase Sensitive Detector Associated with the High Temperature Elasticity Apparatus . . .	16
10	Diagram showing the Zeroing LVDT's and the Deflection Averaging Circuit	17
11	Electric Block Diagram of Apparatus for Measurement of Internal Friction and Dynamic Modulus	18
12	Strain Amplitude Dependence of Mechanical Energy Loss in Graphite Specimens	20
13	Strain Amplitude Dependence of Mechanical Energy Loss in Baked Carbons	21

LIST OF FIGURES (Cont'd.)

Figure		Page
14	Strain Amplitude Dependence of Mechanical Energy Loss in Glassy Carbons	22
15	Thermal Conductivity vs. Temperature of Graphite made from Soft Filler and Soft Binder Carbons	25
16	Thermal Conductivity vs. Temperature of Graphite made from Soft Filler and Hard Binder Carbons	26
17	Thermal Conductivity and Electrical Resistivity vs. Temperature of Graphite made from Soft Filler and Hard Binder Carbons	28
18	Thermal Conductivity vs. Temperature for Various Heat Treatments of Carbons made from Soft Filler and Soft Binder Carbon	29
19	Electrical Resistivity vs. Temperature for Various Heat Treatments of Carbon made from Soft Filler and Soft Binder Carbons	30
20	Arrangement for Conductivity Measurement using Thick-Walled Cylinder Method	31
21	Thermal Conductivity vs. Temperature as Determined by Thick-Walled Cylinder Method	32
22	Thermal Diffusivity vs. Temperature for Various Graphites in the Direction Perpendicular to Extrusion Axis. Transient Heat Flow Method	34
23	Thermal Diffusivity vs. Temperature for Various Graphites in the Direction Perpendicular to Extrusion Axis. Transient Heat Flow Method	36
24	Thermal Conductivity vs. Temperature for Various Graphites in the Direction Perpendicular to the Extrusion Axis	37
25	Specific Heat vs. Temperature plotted according to data from ref. 12 and 13	38
26	Ratio of the Thermal Conductivity to Density and Specific Heat vs. Temperature for Various Graphites	39

LIST OF FIGURES (Cont'd.)

Figure		Page
27	Percentage Deviation ($\alpha - K/dc$) / K/dc vs. Temperature for Various Graphites	39
28	Schematic of the Furnace for the "Steady" Sinusoidal Heat Flow Method	41
29	Schematic of the Apparatus for Thermal Diffusivity Measurements	42
30	$\sqrt{\omega/2\alpha}$ vs. the Heat Loss Factor b	43
31	$\sqrt{\omega/2\alpha}$ vs. the Heat Loss Factor b	43
32	$\sqrt{\omega/2\alpha}$ vs. the Heat Loss Factor b for various values of A_{01} and $(\Delta\phi)_{01}$	44

I. INTRODUCTION

The aim of this project is to study elastic properties of carbons and graphites and to investigate the heat conduction and heat diffusivity of graphite in a wide range of temperatures in relation to the main fabrication variables and to the basic types of carbons tested. With the exception of the mechanical strength, all the factors affecting the resistance of the carbon materials to temperature shocks are being subjected to testing in our laboratory. In the previous Technical Reports, Part I¹, Part II², and Part III³ the results of the previous work up to October 1960 were described. This included the construction of a novel high precision equipment for studies of deformation at room temperature. With this equipment broad studies of the creep and permanent set, as well as of the elastic moduli E and G in relation to the type of carbon material and its heat treatment temperature, were carried out. Some studies of Poisson ratio were made. The four basic types of carbon investigated were made of: 1) soft filler-soft binder, 2) soft filler-hard binder, 3) hard filler-soft binder, and 4) hard filler-hard binder. In addition to these, carbons made using graphitized filler (soft and hard) as well as impregnated carbons and graphites were investigated. The experience gained in operation of the room temperature equipment permitted the successful construction of the high temperature elasticity apparatus, of a completely novel design and of a very high sensitivity and precision. The first results obtained for graphite rods with this equipment were reported in form of force-deflection loops and of one curve of temperature dependence of the Young's modulus up to 2200°C. In addition, some studies of ultrasonic attenuation in relation to the porosity of the carbons and frequency of vibrations used were carried out. They led to a clarification of the scattering mechanism in carbons and of the nature of the true attenuation being caused by elastic hysteresis.

A thermal conductivity apparatus for studies in the temperature range 1000° - 3000°C. was constructed first of a conventional design and studies of the influence of the environmental atmosphere on the measured conductivities, as well as measurements of the temperature dependence of the conductivity for the four basic types of carbons, were carried out with this apparatus. Having gained experience with the simplified apparatus, the construction of a new greatly improved conductivity test equipment was carried out, the novel feature of the equipment

"Manuscript released by authors March 1962, for publication as a ASD Technical Report."

being its great flexibility, permitting heating of the rod directly by the flow of electric current as well as indirectly by a helical heating coil, or by a combination of both. The equipment was tested by performing a number of preliminary runs which showed it satisfactory operation.

A thermal diffusivity apparatus permitting study of the propagation of periodic sinusoidal heat waves through a carbon was constructed and measurements of the heat diffusivity carried out in the temperature range 100° - 650°C . Since the extension of this technique to higher temperatures encountered serious difficulties, another apparatus based on a transient heat flow method was constructed and tested in operation in the temperature range 800° - 2800°C . It has been found that the values obtained directly for the thermal diffusivity α are consistently lower than the values calculated from the corresponding heat conductivities K , specific heat C , and density d , according to the well-known formula $\alpha = K/dC$.

In the present report results obtained in continuation of this work are presented. First the results of some additional more precise studies of impregnated carbons and graphites at room temperature were performed (II), since the results reported in Part III did not seem to be sufficiently satisfying. The main work, however, centered around the studies of the Young's modulus at high temperature (III). Graphitized samples of the four types of carbons were investigated in the range 800° - 2000°C , the important and novel result obtained is that graphites made using a hard filler do not exhibit the well-known maximum in Young's modulus (at about 1800°C .). Furthermore, for the first time, complete families of curves were obtained for the dependence of Young's modulus on temperature and heat treatment for soft and hard carbons, and the presence of maxima in modulus was found to have a quite general occurrence. It has been found, however, that the operation of the equipment presents some difficulties as far as obtaining the more precise shape of these curves is concerned and for that end the electronics associated with this equipment has been rebuilt just at the end of this report period.

Since previous work on attenuation of ultrasonic waves has shown that the attenuation is predominantly caused by scattering due to the inhomogeneous structure of carbons, in continuation of the work on internal friction, we have limited ourselves to the sonic range of frequencies. For this purpose an apparatus with associated electronics was set up and studies of the dependence of internal friction and of the dynamic Young's modulus on strain amplitude were performed for several types of carbons at room temperature (IV). The studies and curves here reported are the first of their kind for carbons and graphite.

Before attempting to obtain a family of curves for the dependence of the heat conductivity on temperature and heat treatment using the new heat conduction test equipment, extended experiments were conducted to check the reproducibility of the results when samples are repeatedly investigated and heated for a considerable time in the apparatus (V). This is a very important feature of these experiments since in previous work at other laboratories, measurements were run along the curves only once without any guarantee of reproducibility which might be responsible for the disagreements in results found in the literature. Having achieved satisfactory reproducibility a corresponding family of curves was obtained for a soft carbon, the system of such curves being for the first time obtained since the unsuccessful attempt was made by Powell and Schofield. Some successful experiments were also performed in trying out a novel arrangement for determination of heat conductivity without internal heat generation and are reported in the same section.

The thermal diffusivity determinations, using the transient heat method, have been considerably improved by the use of a new radiation pyrometer with a much faster response and as a result, the discrepancy between directly measured and calculated values of α greatly reduced (VI). A new furnace was built for the periodic heat method increasing the temperature range to 900°C. and using an IBM 1620 computer, charts were prepared for a rapid determination of α from experimental data.

Having obtained roughly all the basic correlations and dependencies, further advances in the field will mostly depend on our ability to increase the precision of all measurements. Certainly this is a painstaking work with an apparently slower rate of progress, but much more promising and worthwhile than collecting wide data of lower precision and trying to draw conclusions on the basis of statistical considerations.

II. ELASTIC PROPERTIES AT ROOM TEMPERATURE

A. Room temperature Elasticity Apparatus

The electronics associated with the Linear Variable Differential Transformers on the room temperature elasticity apparatus was recently improved. The new model contains two independent channels which one measures the force applied to the specimen and the other the corresponding deflection. The force-deflection curve is recorded on an X-Y recorder and Young's modulus is obtained from the slope of the unloading portions of the curve. Fig. 1 shows a block diagram of the new electronics. The complete electronic circuit is shown in Fig. 2. A full description of this transistorized electronics and its operation has been published by D. C. Wobschall in the Rev. Sci. Inst. 32, 71-73 (1961) in a paper entitled "Sensitive Output Indicator for Differential Transformer Displacement Determination" to which the reader is referred.

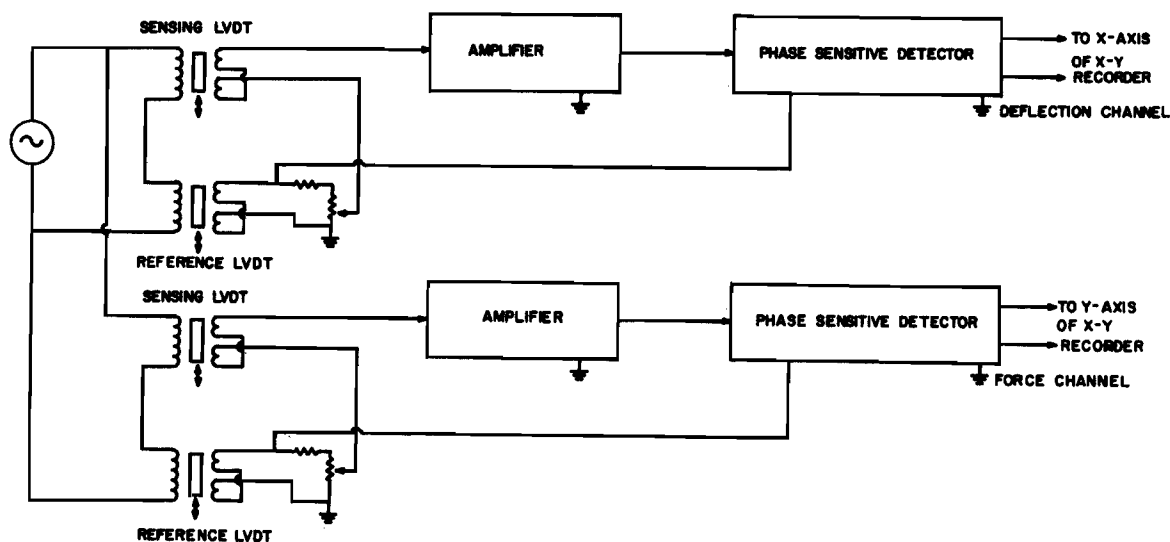


Figure 1 Block diagram of the New Electronics Associated with the Room Temperature Elasticity Apparatus

B. Studies of Young's Modulus at Room Temperature

The variation of Young's modulus with heat treatment was investigated for pitch impregnated soft filler-soft binder rods. The preparation details of these rods was described previously². Barrett Resin C Coal tar pitch was used as the impregnant. Several rods baked to 1000°C. and several rods graphitized to

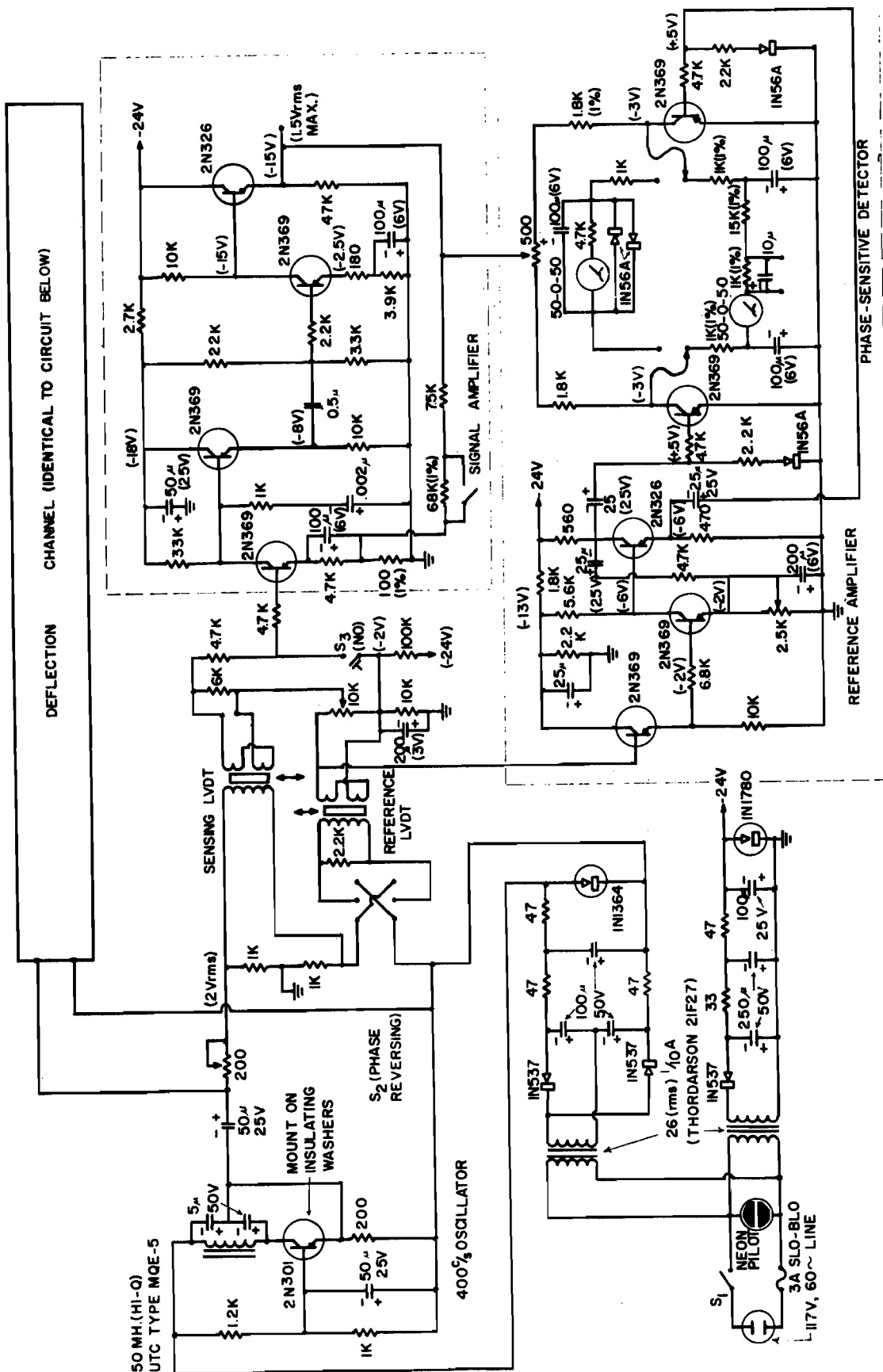


Figure 2 Electronic Circuit for the New Electronics Associated with the Room Temperature Elasticity Apparatus

3000°C. were impregnated. Impregnation was accomplished by submerging the specimens in liquid pitch under vacuum and then opening the chamber to atmospheric pressure.

Table 1 shows the apparent density and Young's modulus before impregnation, together with the same quantities after impregnation and heat treatment to 500°C. Also given are the changes of these quantities in per cent. For the graphitized rods the Young's modulus consistently increases as the weight of pitch introduced into the rod goes up. Surprisingly, such a consistent trend does not appear for the baked rods.

Table 1.

Density, Young's Modulus and Impregnation Data for 1/2 inch diameter extruded rods made with soft filler-soft binder and pitch impregnated after baking or after graphitization

Rod No. and H.T.	Apparent Density g/cm ³	E before impreg. dynes/cm ²	Weight increase after impreg. and 500°C. H.T. per cent	E after impreg. and 500°C. H.T. dynes/cm ²	Increase of E per cent
R3-1(1000°C)	1.470	10.28x10 ¹⁰	3.7	13.42x10 ¹⁰	30.5
R3-2(1000°C)	1.477	10.71	3.3	13.64	27.4
R3-4(1000°C)	1.462	10.47	6.5	13.45	28.5
R3-3(3000°C)	1.475	6.46	7.7	9.31	44.2
R3-7(3000°C)	1.465	6.41	3.3	8.97	40.0
R3-8(3000°C)	1.466	6.25	2.2	8.28	32.5

Fig. 3 shows the dependence of Young's modulus on the heat treatment for the pitch impregnated rods as well as the values of the modulus before impregnation. Rods baked to 1000°C. and then impregnated have a Young's modulus which increases slowly from a heat treatment temperature of 500°C. to a maximum value at around 800°C. Subsequently, a monotonic decrease starts down to a minimum value at 2200°C, after which the modulus rises to a new level at about 2400°C. From 2400°C. up to 3000°C. a small decrease is observed. The general shape of such curves from 1000°C. up reproduces that for soft filler-soft binder un-

impregnated rods.

Fig. 3 shows also the dependence of the Young's modulus for rods heat treated to 3000°C. before impregnation. A steady decrease in the modulus throughout the range of heat treatments investigated is observed. In comparing the effect of impregnation on both kinds of rods it is interesting to note that there is an increase in the elastic modulus in the range 500° to 800°C. for the baked rods, whereas the graphitized rods show a decrease in the same temperature range. This may be due to the incompatibility of the binder with the graphitized filler. However, at the final 3000°C. heat treatment the Young's modulus for both sets of rods with nearly the same density has approximately the same value. This shows that as far as highly heat treated (> 900°C.) materials are concerned, it does not matter whether the rods are impregnated in the baked or graphitized state.

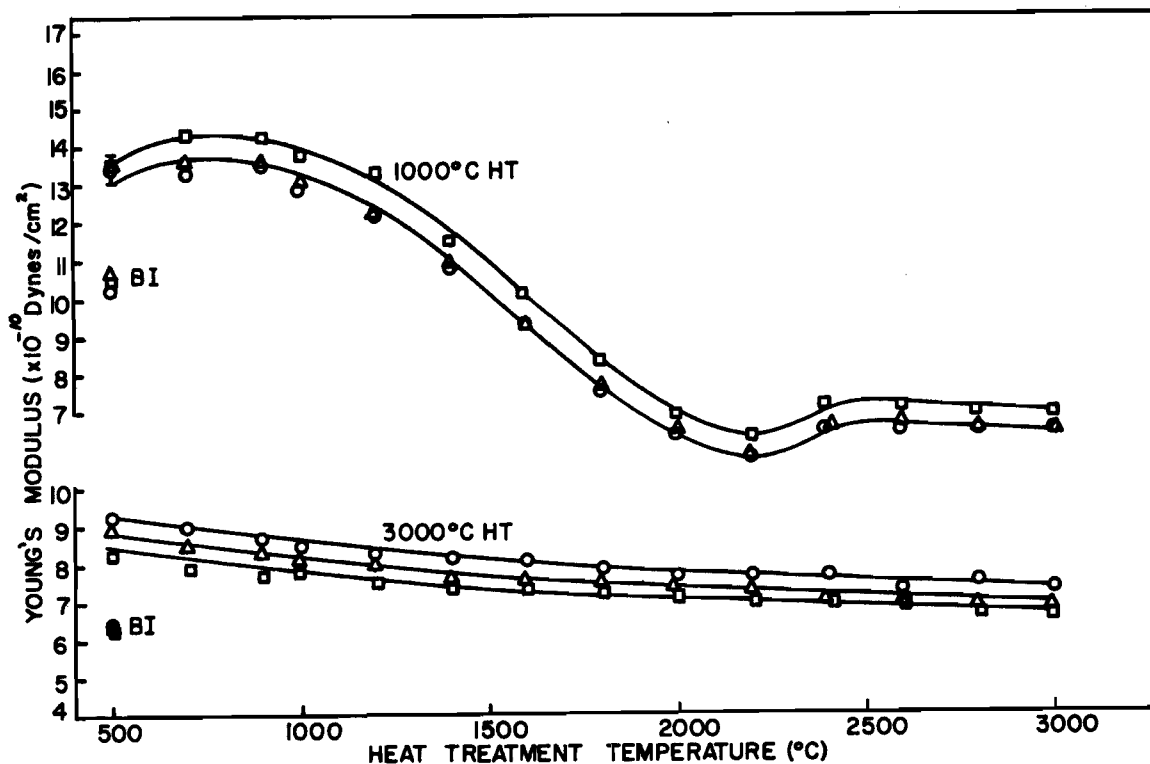


Figure 3 Young's Modulus vs. Heat Treatment Temperature for Pitch Impregnated Rods

III. ELASTIC PROPERTIES AT HIGH TEMPERATURE

A. Apparatus and Samples

The graphite tube furnace designed for the high temperature elasticity studies was described fully in previous reports^{2,3}. Therefore a brief description of its operation is included here only. The specimen to be investigated (1/2" diameter and 8" long) is held by graphite supports at two points about 7" apart. The carbon sample mounted on these supports is placed in a 2" OD graphite tube which can be heated electrically to any desired temperature up to about 3000°C. The bending force is applied at the midpoint between the supports and the curvature of the specimen is measured by three Linear Variable Differential Transformers (LVDT)* placed 3" apart. Contact between the specimen and LVDT cores is made by the intermediary of graphitized lampblack rods. The force is also applied to the specimen through a similar lampblack rod. The two outer LVDT's are used to correct for any displacement which may be caused, for instance, by crushing or viscous creep at the sample-support contacts. Electronic circuits average out the two differences in deflection and an X-Y recorder gives the force-deflection curve whose slope is a measure of Young's modulus.

In the experiments reported here the total deflection of the sample was kept small, of the order of 0.05 mm. The force was applied gradually to the rod by evacuating a bellows. The force vs. averaged deflection was in most cases recorded on an X-Y recorder as mentioned earlier. The time required for recording half of the stress-strain loop at a given temperature was about 15 seconds. Under these conditions creep effects were found to be negligible for ambient temperatures below 2000°C.

The preparation details of the carbon bodies used in this work were described previously². These carbons were of the following four types: soft filler-soft binder (SF-SB), soft filler-hard binder (SF-HB), hard filler-soft binder (HF-SB), and hard filler-hard binder (HF-HB). The soft filler was a calcined petroleum coke (Texas coke) and the soft binder was a coal tar pitch (Barrett M-30). The hard filler was from a phenolformaldehyde resin (Durez No. 175) and the hard binder was a partially polymerized phenol benzaldehyde (laboratory prepared).

B. Young's Modulus of Graphite Rods at High Temperature

Rods of each of the four types of carbons were heat treated to 3000°C. before mounting in the high temperature elasticity

*Schaevitz Engineering Company, Camden, New Jersey

apparatus (SF-SB, SF-HB, HF-SB, HF-HB). Fig. 4 shows the Young's modulus data for these rods as a function of temperature up to 2000°C. The measurements were not carried to higher temperature at this time because of the onset of viscous creep at about 2000°C. The usual maximum in Young's modulus is observed only for the rods with soft filler whereas bodies with hard filler show a continuous decrease in modulus with increasing temperature. The fact that there is a maximum for the soft filler bodies and no maximum for the hard filler bodies shows that the behavior of the Young's modulus vs. temperature depends primarily on the filler material. A similar conclusion was reached previously by Davidson⁴ and also in our experiments on the dependence of the room temperature Young's modulus on the heat treatment temperature². These results show that a study of the dependence of the strength of hard filler bodies on temperature would be a worth while undertaking (to check if such carbons show a maximum of strength around 2500°C. as the soft filler graphites do).

Preliminary measurements on National Carbon Company type ZTA graphite showed for the strain direction parallel to graphite planes an increasing modulus with a maximum around 2200°C. Both pyrolytic and ZTA graphite are quite stiff and therefore are difficult to measure. Further measurements will be made with the improved measuring equipment described below (Section D).

C. Changes in Young's Modulus of Carbon Rods during the Heat Treatment Process

In this work a specimen which was previously heat treated to about 1000°C. was mounted in the furnace and force-deflection measurements made throughout the temperature range 800° - 1200°C. after which the rod was cooled to room temperature without removing it from the furnace. Following the same procedure, force-deflection measurements were made throughout the temperature range 800° - 1400°C. and the rod was again cooled to room temperature. Measurements were continued, using the same procedure, increasing stepwise the maximum temperature by 200°C. in each run. The heating rate of the furnace was about 200°C. per hour in each run and the holding time at the maximum temperature was about 20 minutes. A typical curve obtained in a single run up

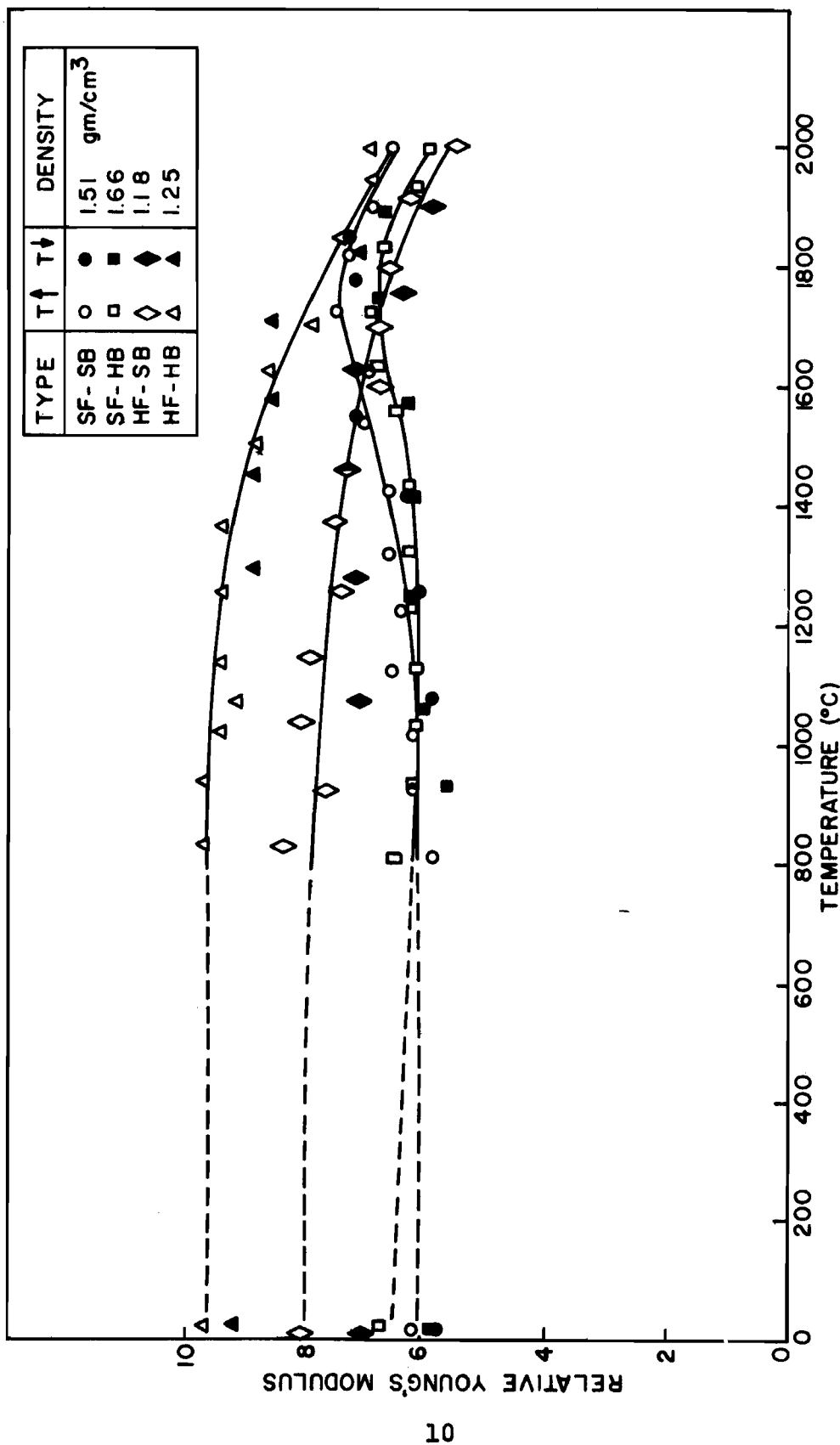


Figure 4 Young's Modulus vs. Ambient Temperature for Four types of Graphitized Carbon Bodies

and down is shown in Fig. 5 for a SF-SB rod having a heat treatment temperature of 1800°C. For increasing temperature the Young's modulus follows the upper curve through a maximum up to the original heat treatment temperature of 1800°C, and then above up to 2000°C; for decreasing temperature it follows the lower curve (again through a maximum).

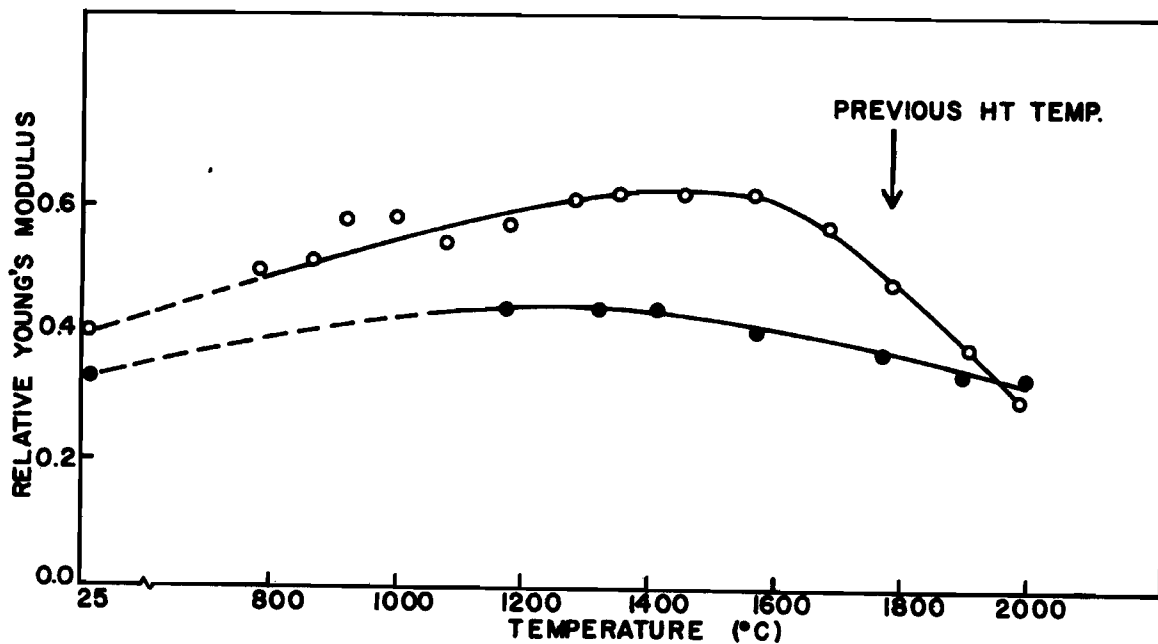


Figure 5 Typical Curve for the Dependence of Young's Modulus on Temperature for a Soft Filler-Soft Binder rod having an Original Heat Treatment Temperature of 1800°C.

An interesting feature in all of our measurements of the high temperature modulus of elasticity is the time behavior of the modulus when the ambient temperature is held constant at its highest value. It was found that under these conditions the elastic modulus gradually increases between successive measurements (observed over a total period of 20 minutes). This fact is evidenced on Fig. 5 for the ambient temperature of 2000°C. This modulus change is probably not due to an actual work hardening of the sample since the deflection under load is of such a small magnitude, but possibly in some way connected with the actual growth of the graphite crystallites, which is known to occur over long periods of time^{5,6}.

A complete family of curves for a commercial SF-SB rod is shown in Fig. 6. The heat treatment temperature of the specimen when first inserted into the furnace was about 1000°C. For the sake of clarity the figure shows only the curves obtained for increasing temperature. The heavy broken line on the figure shows the variation of Young's modulus for continually increasing heat treatment temperature. The essential features

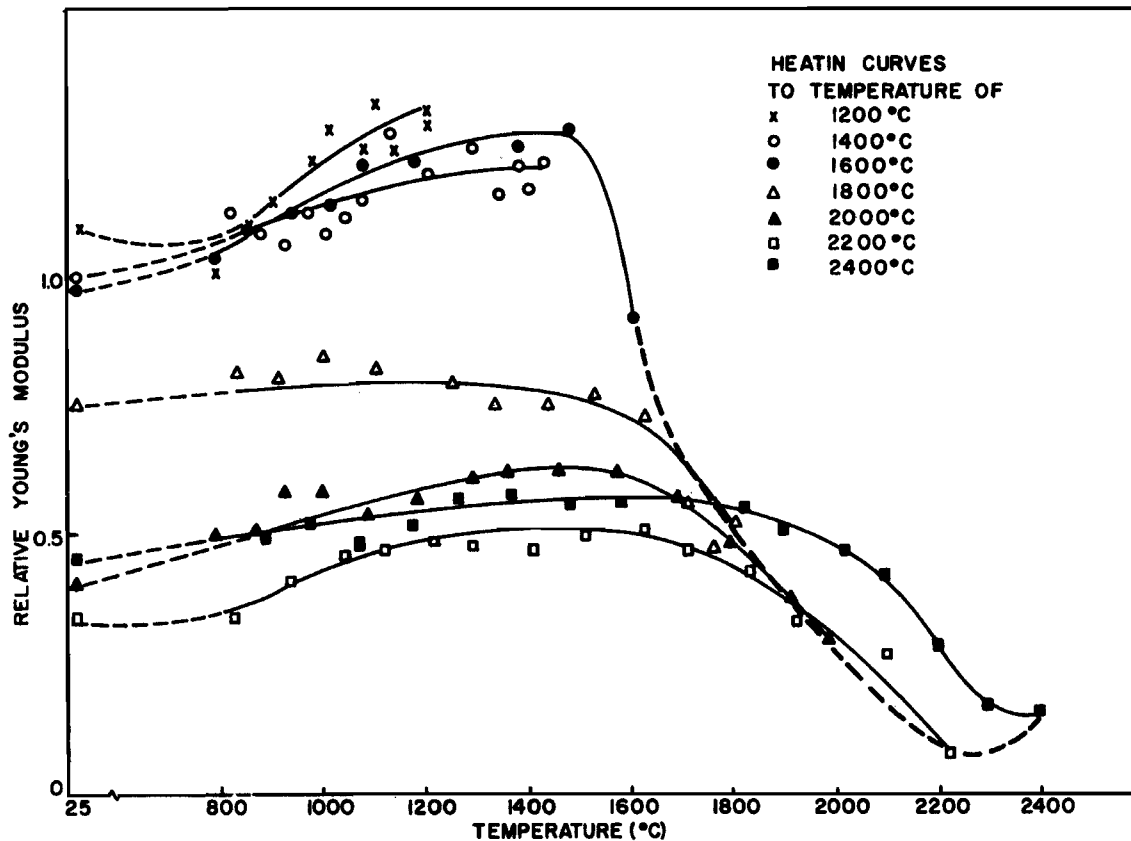


Figure 6 Young's Modulus as a Function of the Ambient Temperature for a 1/2 inch Diameter Extruded National Carbon Company Baked Carbon for Increasing Temperatures

of the results obtained are: 1) a general decrease in the Young's modulus with increasing temperature when the rod is being heat treated, that is, for temperatures above the highest previous heat treatment temperature but probably only up to about 2200°C, 2) the presence of maxima in Young's modulus curves at a temperature somewhat below the maximum heat treat-

ment temperature for all variously heat treated samples of such carbons. The dependence of the room temperature values of Young's modulus on the maximum heat treatment temperature as obtained in this work follows the general behavior previously reported². Similar systems of curves have been obtained for the laboratory-prepared SF-SB carbon specimens.

Similar elasticity measurements have also been made on a hard filler-hard binder (HF-HB) rod whose original baking temperature was 500°C. The results are shown in Fig. 7 (PHT refers to previous heat treatment). It is interesting that possibly

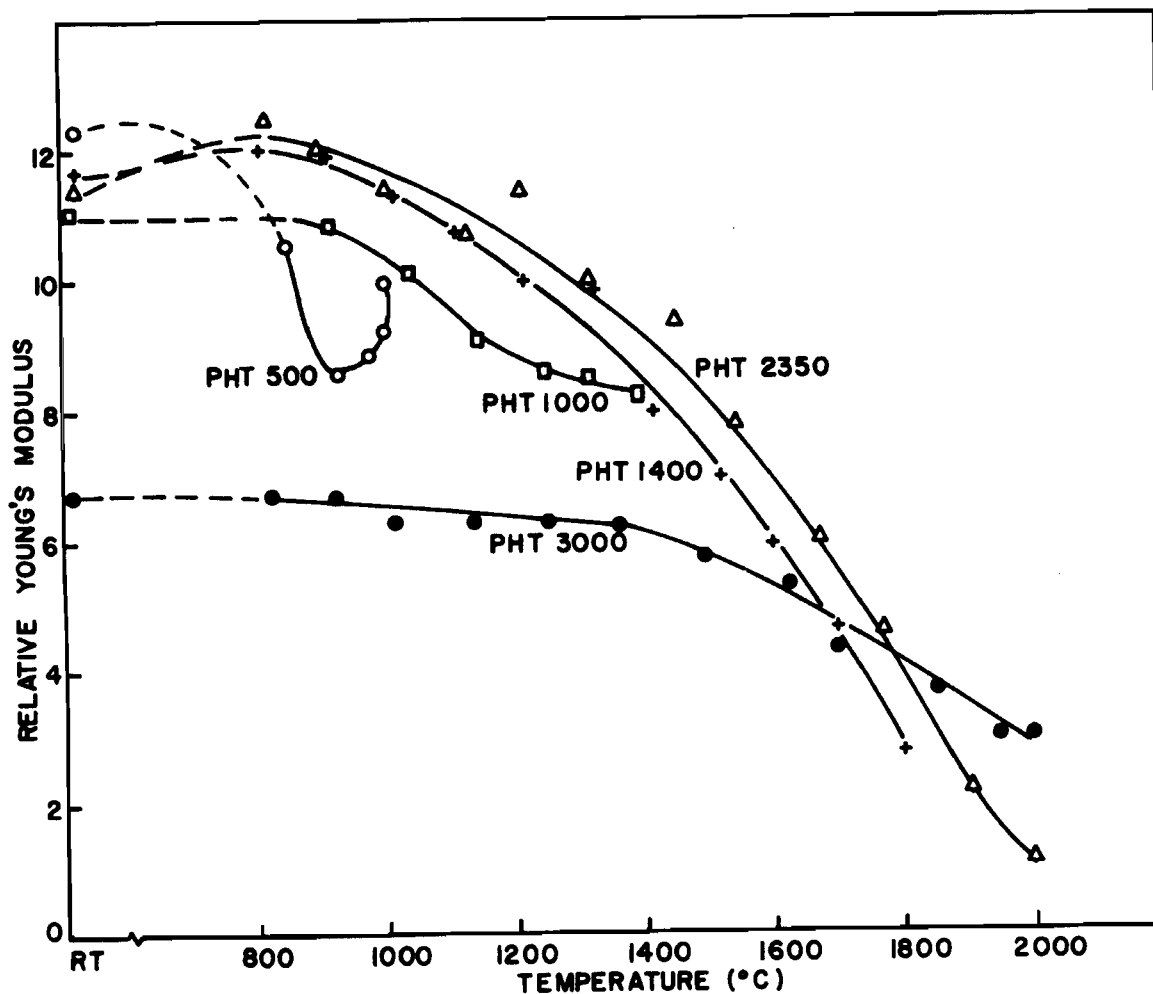


Figure 7 Young's Modulus vs. Ambient Temperature for a 1/2 inch Diameter Extruded Laboratory-Prepared Hard Filler-Hard Binder Baked Carbon for Increasing Temperatures

all of the curves show a slight maximum except the curve for the highest heat treatment. The curve PHT 500°C. is most peculiar and shows the large changes occurring in the process of baking between 500° and 1000° C. One can see that after that, during heat treatment, the modulus decreases continuously up to 2000°C. and possibly still beyond.

D. Improved Electronics for the High Temperature Elasticity Apparatus

Since the servo-system had a narrow range of reproducible operation, the system had to be reset several times during one series of experiments. This led to scattering in obtained data. Due to the great difficulty in setting up the sample, the lamp-black rods and the LVDT's and due to the criticalness of their alignment in the high temperature elasticity equipment, as well as to the difficulties in realigning of the servo-motors themselves, new electronics had to be designed which eliminates the troublesome servo-motor system and the alignment problems involved therein. In the new design the displacements which are measured by Linear Variable Differential Transformers are properly averaged before being amplified, rectified, and recorded. The diagrams of the electronics circuits are shown in Figs. 8, 9, and 10. The new apparatus will increase the accuracy of the force and strain measurements; unfortunately, up-to-date no complete curves were yet obtained to be included in this report.

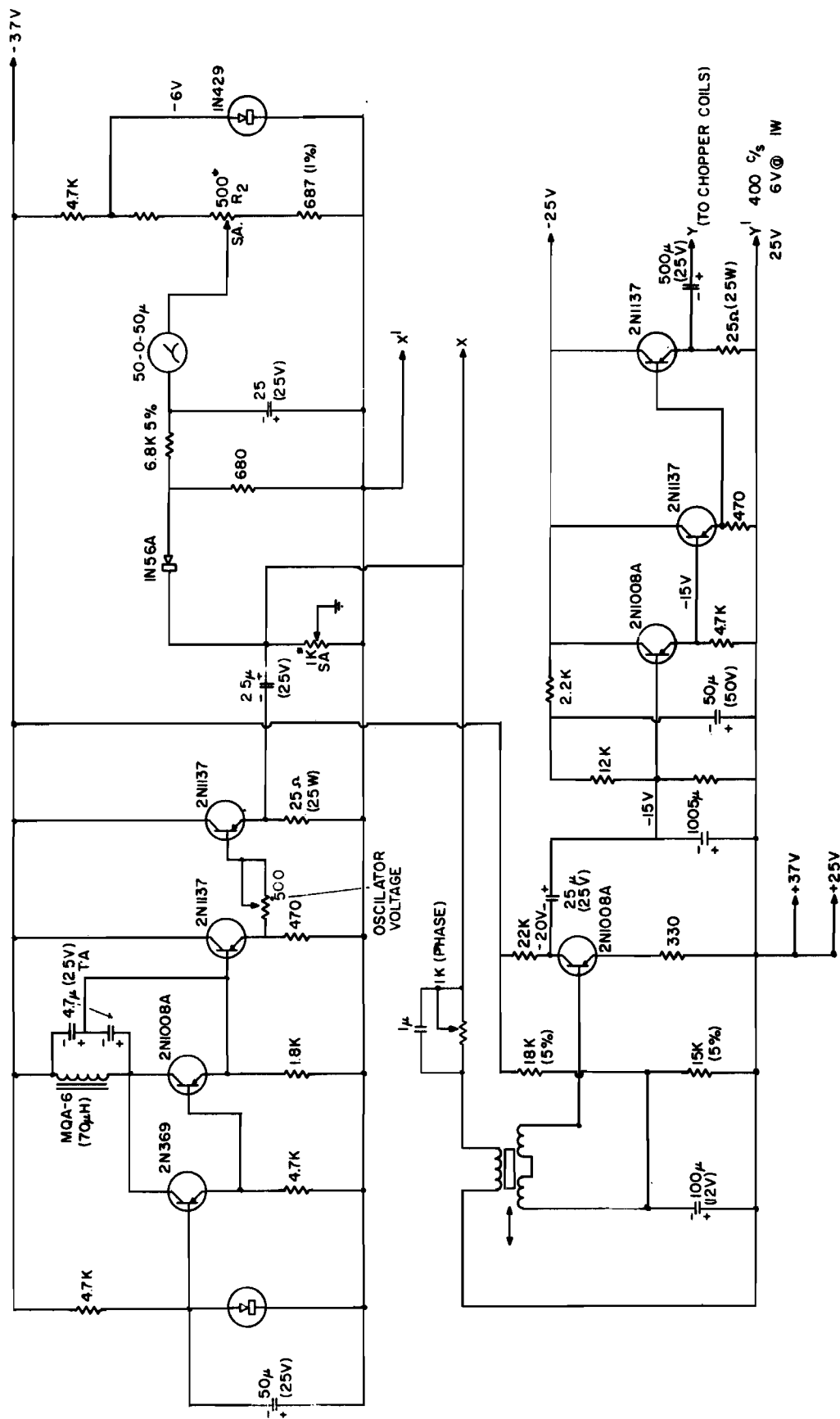


Figure 8 Electronic Circuit of 400 c/s Oscillator and Chopper Driver Amplifier. Associated with the High Temperature Elasticity Apparatus

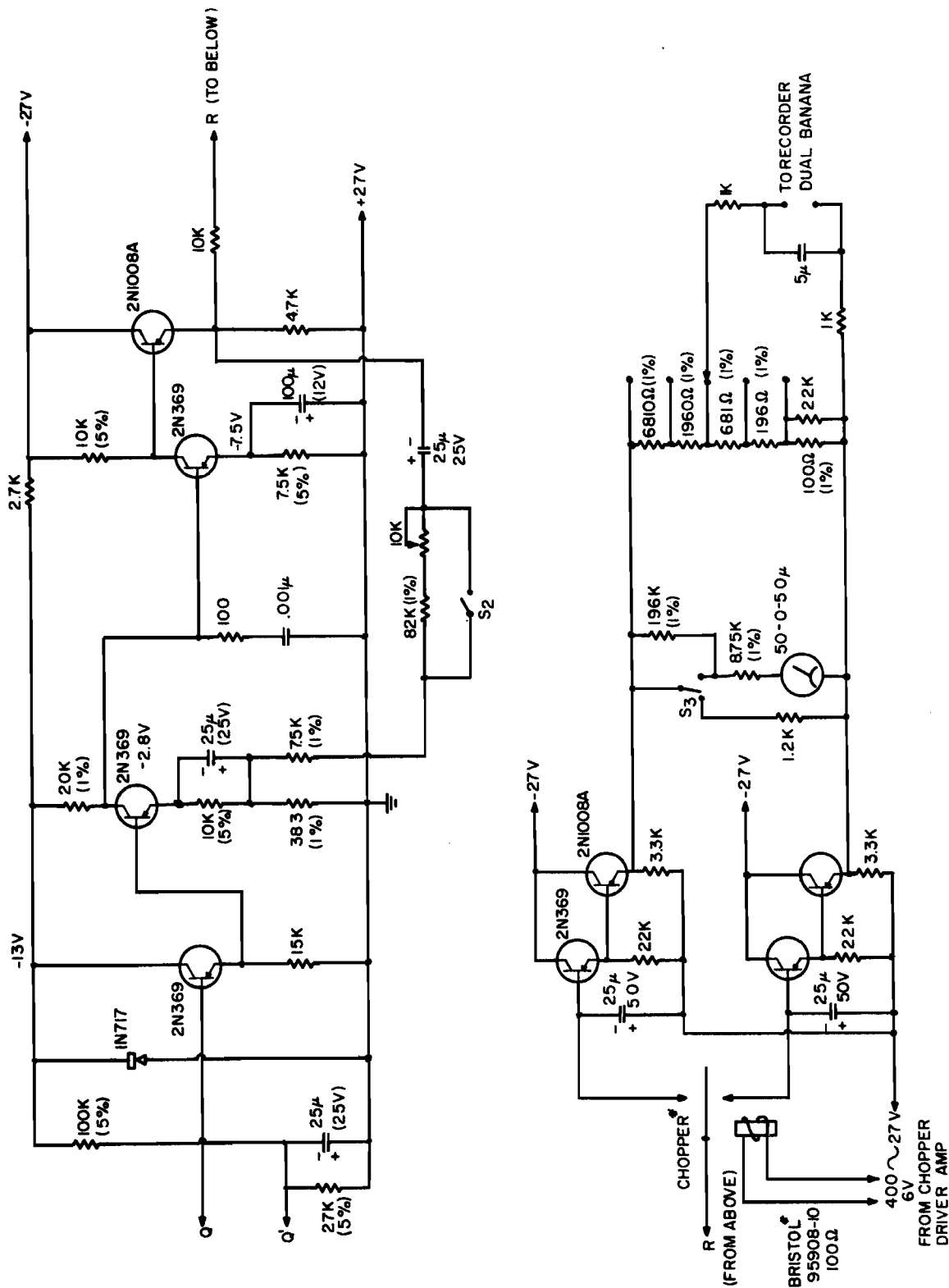


Figure 9 Amplifier and Phase Sensitive Detector Associated with the High Temperature Elasticity Apparatus

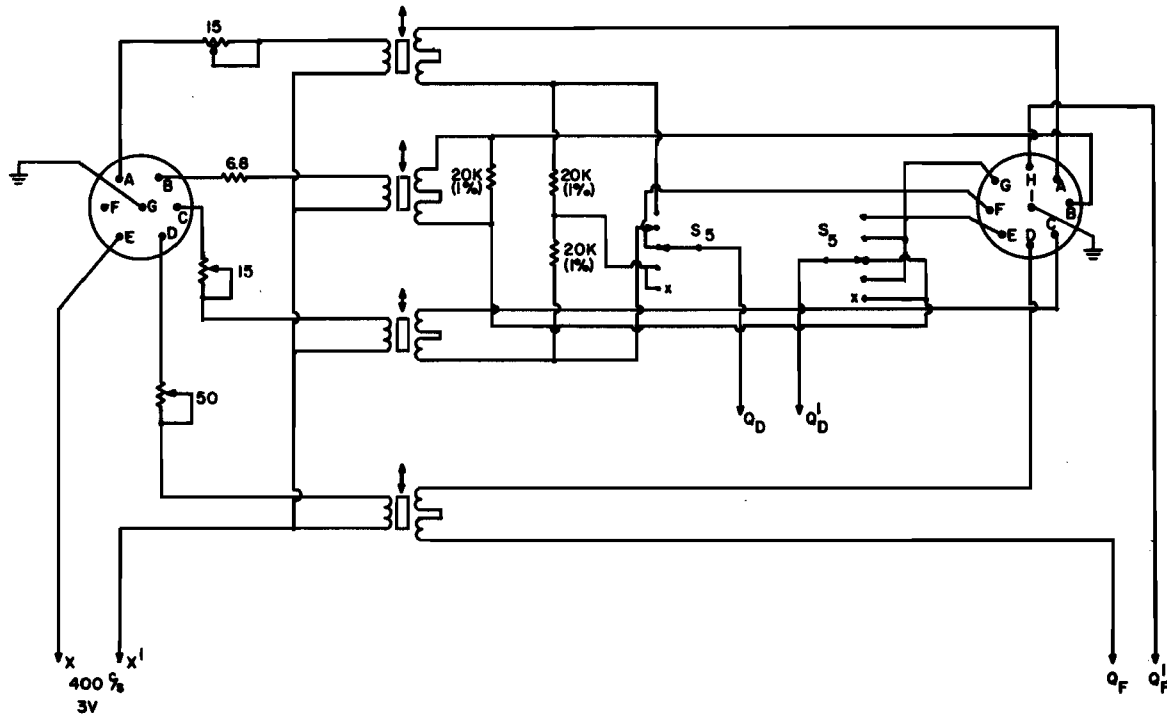


Figure 10 Diagram showing the Zeroing LVDT's and the Deflection Averaging Circuit

IV. MECHANICAL ENERGY LOSSES

As the first step in the study of mechanical energy dissipation in carbon materials, the internal friction and the dynamic modulus of some specimens in baked and graphitized state have been preliminarily investigated as functions of the strain amplitude.

A. Experimental Method

The electric block diagram of the apparatus is given in Fig. 11. The experiments were wholly executed in the audio-frequency range between 260-650 c/s, because as shown by previous work performed under this contract, in the case of carbon, short elastic waves are scattered strongly by pores and such scattering is the predominant factor in determining the amount of internal energy dissipation².

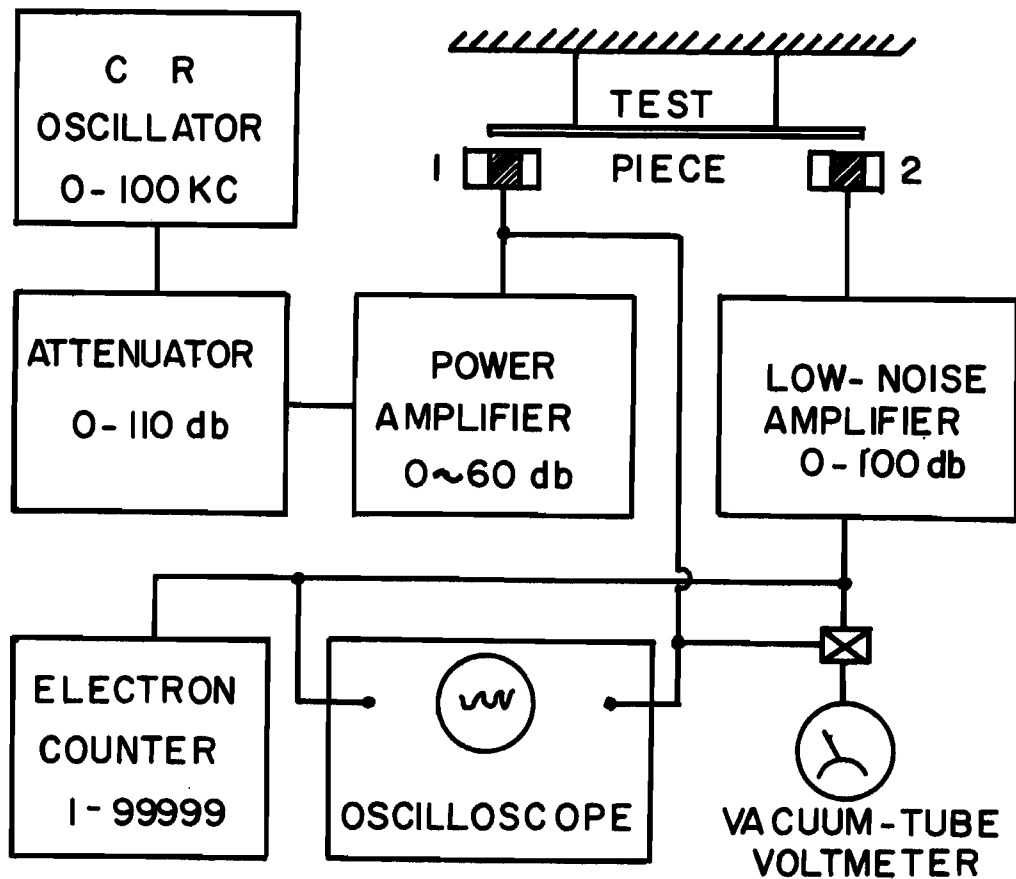


Figure 11 Electric Block Diagram of Apparatus for Measurement of Internal Friction and Dynamic Modulus

A test piece in form of a long slab supported by nylon strings at the nodal points of the transverse vibrational mode is electromagnetically driven by means of the transducer 1 and the induced vibrations are detected by means of the transducer 2. The electrical signal from the transducer 2 is amplified by a known amount and then set as the input to an electron counter, vacuum-tube voltmeter, or oscilloscope respectively. By following through the amplitude vs. frequency relationship, one can obtain the so-called resonance curve for the test piece. The sharpness of this curve is expressed by the factor

$$Q = (1/\sqrt{3})(\Delta f/f) \quad (1)$$

which gives a measure of the internal friction. Here f is the resonance frequency and Δf the half-value width. The dynamic modulus E can be calculated from the resonance frequency ($E \propto f^2$).

The driving force applied to the test piece could be varied in wide limits by controlling the electrical input to transducer 1; the strain amplitude was directly determined using a microscope.

B. Specimens

Specimens examined were taken from the three kinds of commercial carbons as follows: reactor grade graphite, a baked carbon such as manufactured for crucible use, and the so-called glassy carbon, a special hard carbon made in Japan⁷. The reactor grade graphite was formed by extrusion and then heat treated at 2800°C. in an atmosphere containing fluorine for purification. Its density ranges between 1.67 - 1.69 and its impurity content is only 2×10^{-3} per cent. The baked carbon was a block molded and then heat treated to 1200°C. Its density is quite high and ranging between 1.82 - 1.86. Finally, the hard, so-called glassy carbon was prepared from a highly cross-linked resin without using any binder and heat treated to 1000°C. This is a quite hard, brittle and gas-impermeable material which can be cut only by using diamond tools. It has been checked by means of X-ray diffraction that even after heat treatment to 2000°C. this carbon is still completely amorphous. This glassy carbon is a perfect example of a very hard carbon in which no crystalline phase is present.

Slabs about 6" long were prepared from the reactor grade graphite in such a way that the specimen axis was parallel to

the direction of extrusion or perpendicular to it pointing in the radial or tangential direction. Similar slabs were cut out from the baked carbon along the axial and the radial directions of molding. In the case of the glassy carbon a 4" long and very thin (0.06") slab was prepared.

C. Results

The experimental results are presented in Figs. 12, 13 and 14. One can see that the internal friction given by the quantity Q^{-1} in all cases gradually decreases with decrease of the strain amplitude towards a limiting value. On the contrary, the dynamic Young's modulus E is found to increase asymptotically towards a constant value E_0 with decrease of the strain amplitude. It is worthwhile to remark that both curves of Q^{-1} and of E/E_0 vs. strain amplitude flatten out in about the same range

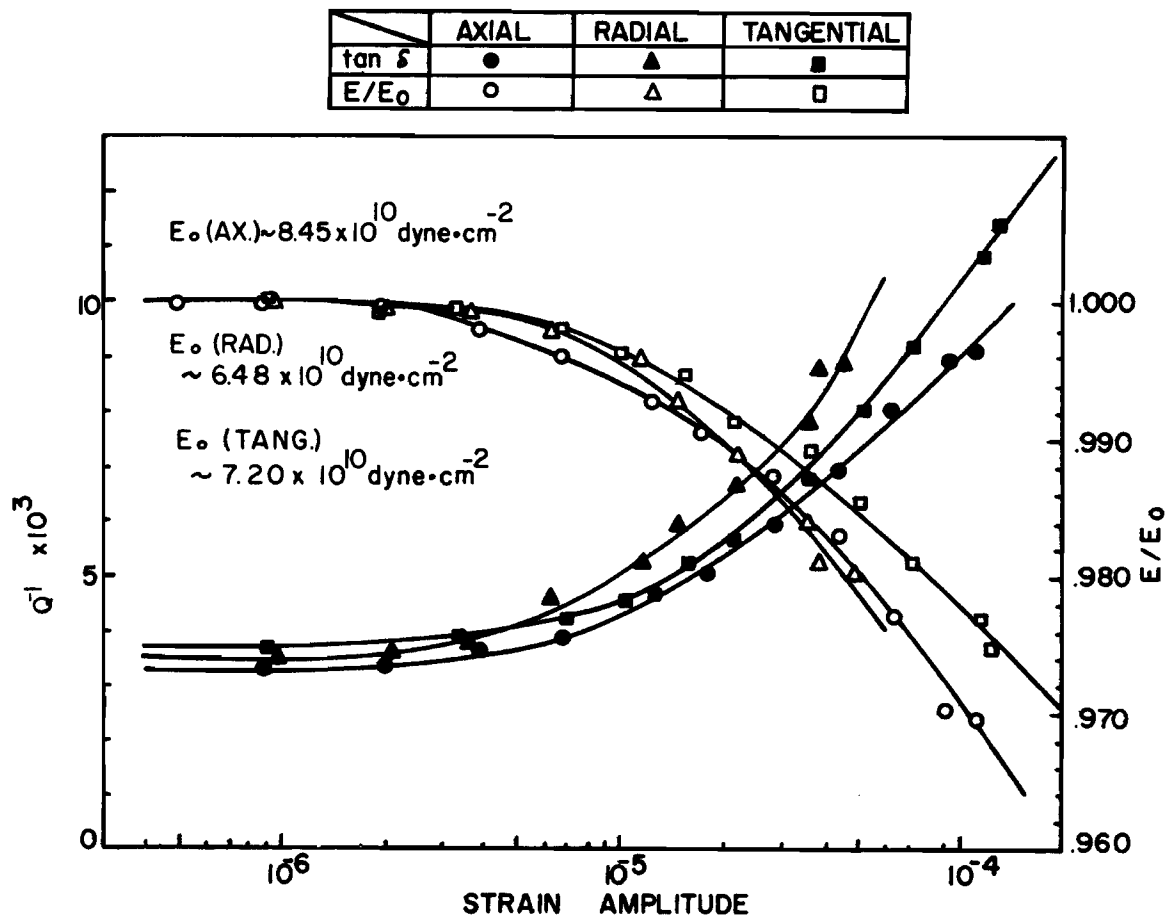


Figure 12 Strain Amplitude Dependence of Mechanical Energy Loss in Graphite Specimens

	AXIAL	RADIAL
$\tan \delta$	●	▲
E/E_0	○	△

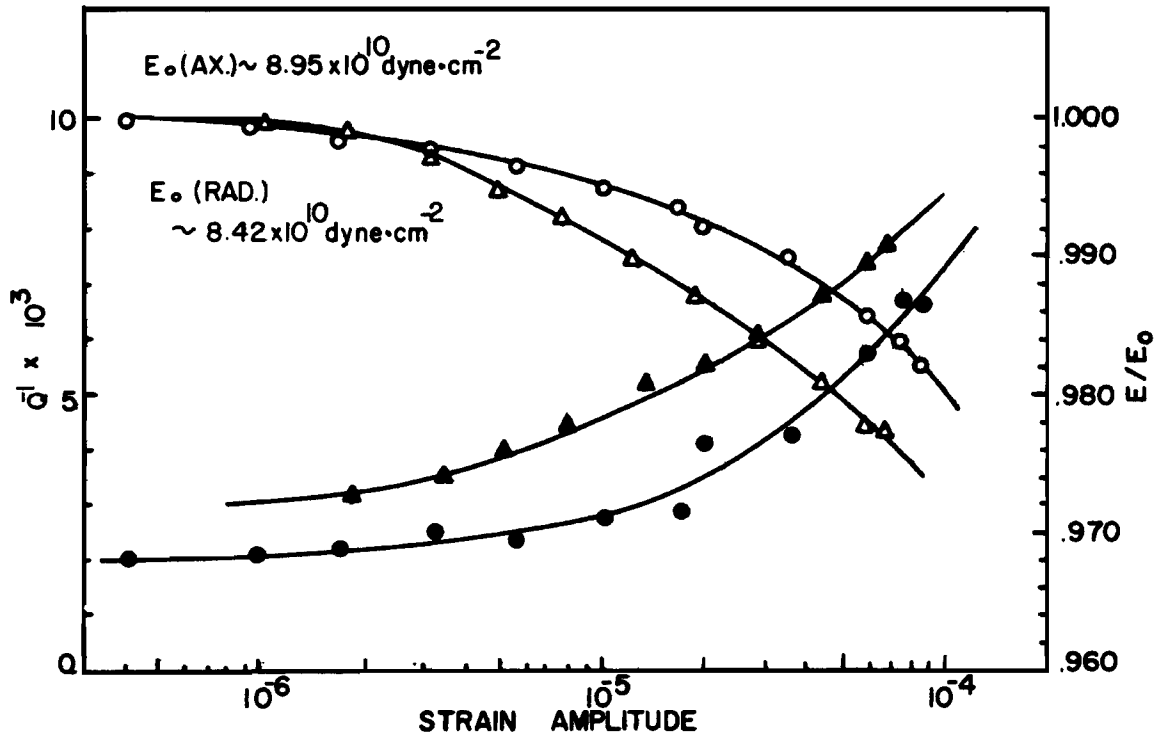


Figure 13 Strain Amplitude Dependence of Mechanical Energy Loss in Baked Carbons

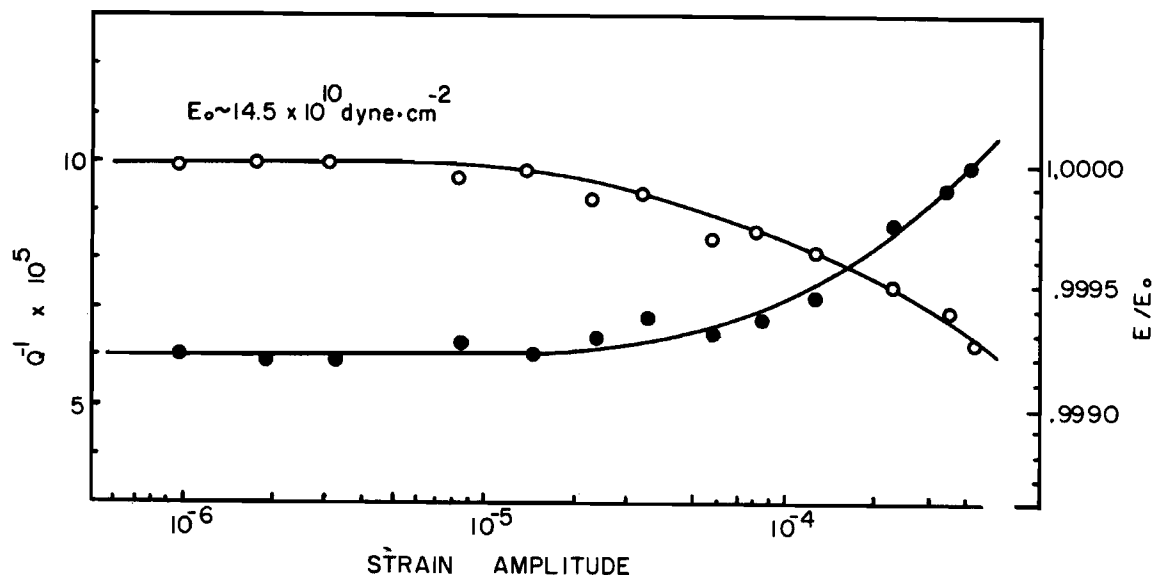


Figure 14 Strain Amplitude Dependence of Mechanical Energy Loss in Glassy Carbons

of strain amplitudes. In other words, the behavior of one of the quantities gives information as to the behavior of the other. Although in experiments of this kind any variation of Young's modulus, i.e., the change in resonance frequency, can be detected much more precisely than that of Q^{-1} (by a factor of 10^2 in accuracy), the total change in E is percentagewise much smaller and as a result both curves show approximately the same scatter of experimental data. Such a close correlation between Q^{-1} and E as established here might turn out to be quite useful in the later stages of this study when the temperature dependence at low temperatures will be investigated.

It seems that the internal friction, as well as the dynamic modulus, consists of two parts, one amplitude-dependent and the other amplitude-independent. The amplitude-dependent energy loss is probably connected with the plastic (viscous) flow in the specimen and the amplitude-independent portion represents the intrinsic anelasticity of carbon material. It is quite interesting that for such small strains as 10^{-5} (far below the usual yield point) a contribution of the plasticity is still observed.

1. Graphite

In the case of the reactor grade graphite (Fig. 12) the difference in orientation of specimens seems to have only a

minute effect on the mechanical loss characteristics, except for the amplitude-dependent part at larger strains where E is larger, Q^{-1} is smaller. The transition point from the amplitude-dependent to the independent region is about 3×10^{-6} and the same for all three test pieces.

2. Baked carbon

The baked carbon exhibits lower Q^{-1} and higher E values than graphite (Fig. 13). This corresponds to the well-known facts that baked carbon is hard and brittle while graphite is soft and ductile. A considerable difference in behavior between the two test pieces, the axial and the radial one can be noticed as far as the energy losses are concerned. The radial sample shows an amplitude dependence down to strains as low as 10^{-6} while the axial one shows a transition point about the same as that for graphite.

3. Glassy carbon

As seen in Fig. 14 the internal friction is for the glassy carbon much lower than for the more conventional carbon products mentioned above, with numerical values of the order of 10^{-5} . The amplitude-independent region extends to higher strains than for the other carbons. These facts are consistent with its extremely brittle properties mentioned before (Section B).

D. Concluding Remarks

One of the purposes of this study is to clarify the microscopic mechanism of the energy loss in carbon materials. The amplitude dependence reported above for Q^{-1} is similar to that obtained by others for metals and explained in terms of the dislocation theory⁸. In view of the outstanding dynamical character, the dislocations lying in the basal plane are expected to play a most important role in such phenomena. Efforts are being made to clarify theoretically their possible contribution to the internal friction.

V. THERMAL CONDUCTIVITY MEASUREMENTS

A description of the improved thermal conductivity test apparatus was given in detail previously³. The important features of the new design and some preliminary results were presented in that report. During the period covered by this report, further tests were conducted to prove the design features of the equipment and to extend the investigations made possible with the new apparatus.

The following investigations have been made during the period of this report:

- 1) A series of tests to establish reproducibility of results obtained on successive heating and cooling of the test specimen
- 2) A series of tests to determine the effect of heat treatment temperature on thermal conductivity at various temperatures
- 3) Measurements of conductivity on a test specimen without internal heat generation.

A. Tests to Establish Reproducibility

In the first of a series of tests, conductivity measurements were made on a test rod extruded in our laboratory from a mixture of 100 parts of soft filler coke particles (200/270 mesh size) and 50 parts of M-30 soft binder. This rod was then baked and heat treated to 3000°C. prior to testing. Its apparent density was 1.54. The rod was inserted in the conductivity test chamber and an argon atmosphere slightly above one atmosphere of pressure was maintained throughout the experiments. The rod was heated internally by passing a current through it and the radial temperature gradient was measured. The results of a series of test runs made on this rod are shown on Fig. 15. The thermal conductivity was determined during three successive runs during which the temperature did not exceed 2200°C. Good reproducibility of measured values was obtained during this first sequence of test runs. On a fourth run, the rod was heated and data obtained throughout the temperature range of 1200° - 3000°C. As can be seen the measured values below 2200°C. were again in good agreement with the results obtained on the three previous test runs. However, upon subsequent cooling and repeating the test, results were obtained indicating a noted decrease in the conductivity throughout the entire temperature range as can be readily observed on Fig. 15. The change in conductivity appears to have taken place after the rod had been heated to temperatures above 2200°C. and approaching 3000°C. The electrical

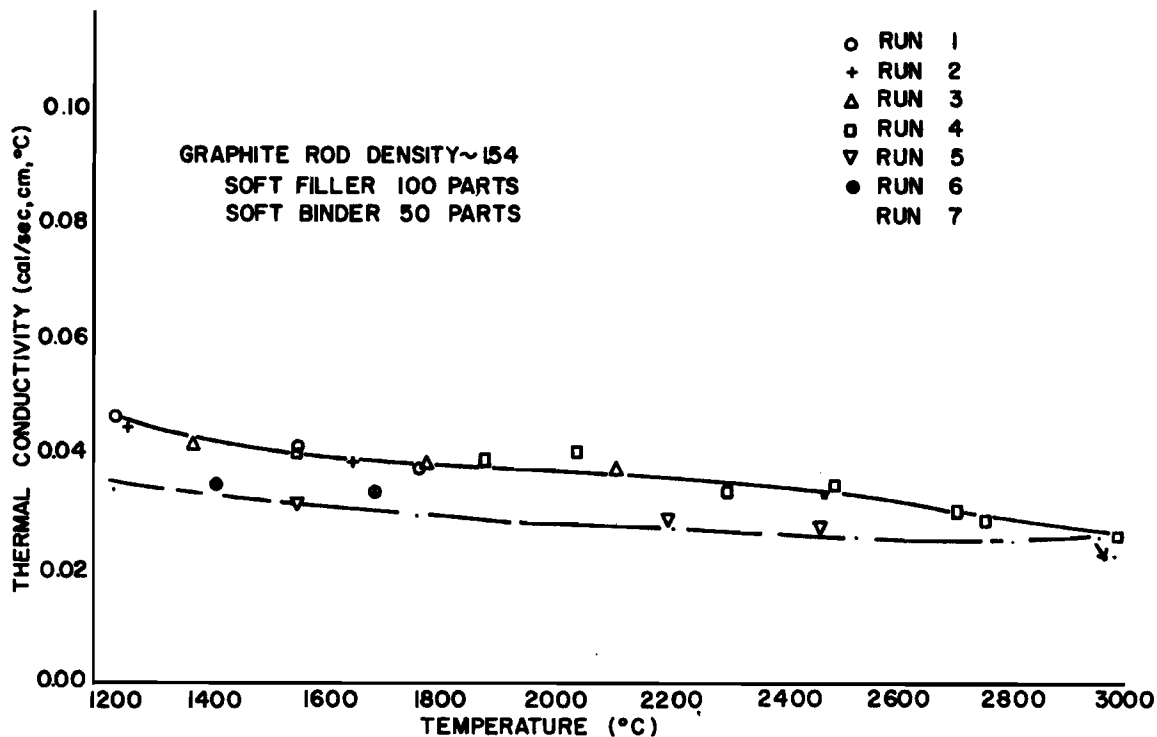


Figure 15 Thermal Conductivity vs. Temperature of Graphite made from Soft Filler and Soft Binder Carbons

resistivity also shows an increase over values obtained prior to operating the rod at elevated temperatures, thus indicating changes in the carbon material. Exposure at the 3000°C. temperature level for extended time gave evidence of a continuous decrease in thermal conductivity with time of exposure as well as of a continuous increase in the electrical resistivity. The decrease in thermal conductivity is indicated on Fig. 15 by an arrow showing the change occurring after several hours of high temperature operation.

After obtaining this evidence of changes in the carbon rod, the rod was taken out from the conductivity apparatus and broken to examine its cross section. A concentric ring of a different appearance than the original carbon, around the periphery of the rod to a depth of about 1/4 of the radius, showed that some changes took place in the carbon material. It was felt that this change is due either to sublimation of carbon or to oxidation by a gas coming out from materials within the apparatus. Since an argon atmosphere was maintained in the test chamber at all times, it was suspected that a reacting gas might be re-

leased by the insulating carbon powder (Thermax) contained in the insulating cylinder used in the test chamber. At the high temperature operation this cylinder reaches quite high temperatures and any contamination associated with the Thermax powder may have been released and attacked the test rod under prolonged high temperature operation.

In an attempt to eliminate this possible source of difficulty, the Thermax filled insulating cylinder was removed from the test chamber and a series of tests were made to see if the results obtained indicated this to be the source of trouble.

The rod used in these experiments was produced in our laboratory from a filler of soft coke (50 parts 65/100 mesh and 50 parts 200/270) and 35 parts of hard binder. This rod was baked and graphitized to a temperature of 3000°C. prior to testing. The apparent density of this rod was 1.65. The same operating techniques were used as reported above. The results of a series of first test runs are shown in Fig. 16. There

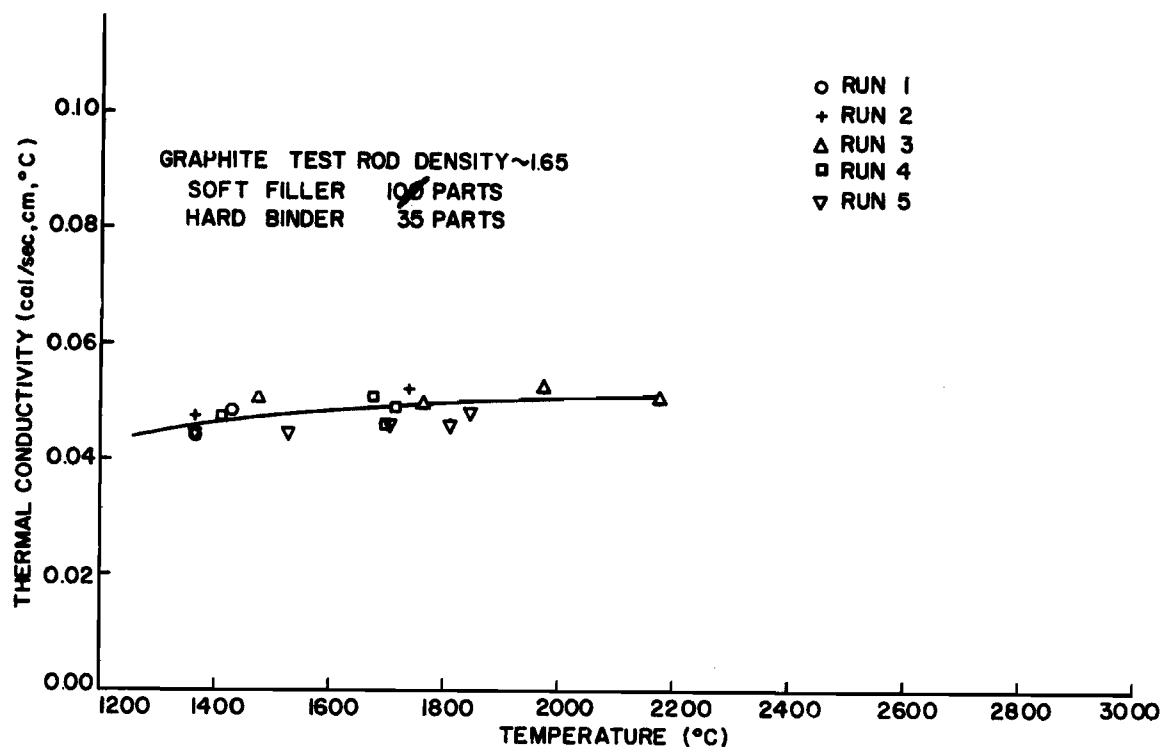


Figure 16 Thermal Conductivity vs. Temperature of Graphite made from Soft Filler and Hard Binder Carbons

is some spread in the measured conductivity values at various temperature levels. It is felt that the scatter in conductivity values in Fig. 16 is due to the inherent difficulties of measuring the temperature gradient within the test rod, especially at the lower temperature levels. This difficulty has been noted in previous reports but to date no method or instrumentation to eliminate this difficulty has been found.

Further tests were conducted with the Thermax filled cylinder shield removed in an effort to see if rod temperatures approaching 3000°C. could be obtained even with this shield removed from the apparatus and to check the reproducibility of repeat runs. A carbon rod extruded in our laboratory from a mixture of soft filler and hard binder was baked and then graphitized to a temperature of 3100°C. This test rod was mounted in the test chamber and a series of measurements were made of thermal conductivity and electrical resistivity. The test rod was heated by passing an electrical current through it and data obtained for conductivity determination at temperatures from 1000°C. to 2800°C. The rod was then cooled to room temperature and again heated throughout the same temperature range and measurements made again. The same procedure was followed for five consecutive runs and the results are shown in Fig. 17. In each of these test runs data were obtained from which the electrical resistivity was also determined. The thermal conductivity and the electrical resistivity values determined on each of these five successive runs show good agreement and indicate that reproducibility of results has been obtained. There does not appear to be any evidence of a definite change in the properties of the rod upon successive runs.

It is of interest to note that the thermal conductivity as measured for the rod made from soft filler and hard binder shows a slight increase in value with temperature throughout the temperature range investigated. This is in contrast with carbons made from soft filler-soft binder which show a decrease in conductivity with temperature from 1000°C. - 2500°C. and then a slight increase at temperatures above 2500°C.

B. Test to Determine Effect of Heat Treatment

Of particular interest was the second series of tests made to determine the effect of heat treatment temperature on the thermal conductivity and electrical resistivity at various temperature levels. In this investigation a carbon rod extruded in our laboratory from a soft filler-soft binder mixture was baked to a temperature of 1000°C. This rod was installed in the conductivity test chamber again with the Thermax filled cylinder removed. The helical carbon coil surrounding the test rod was used as a heating element to bring the rod to various heat

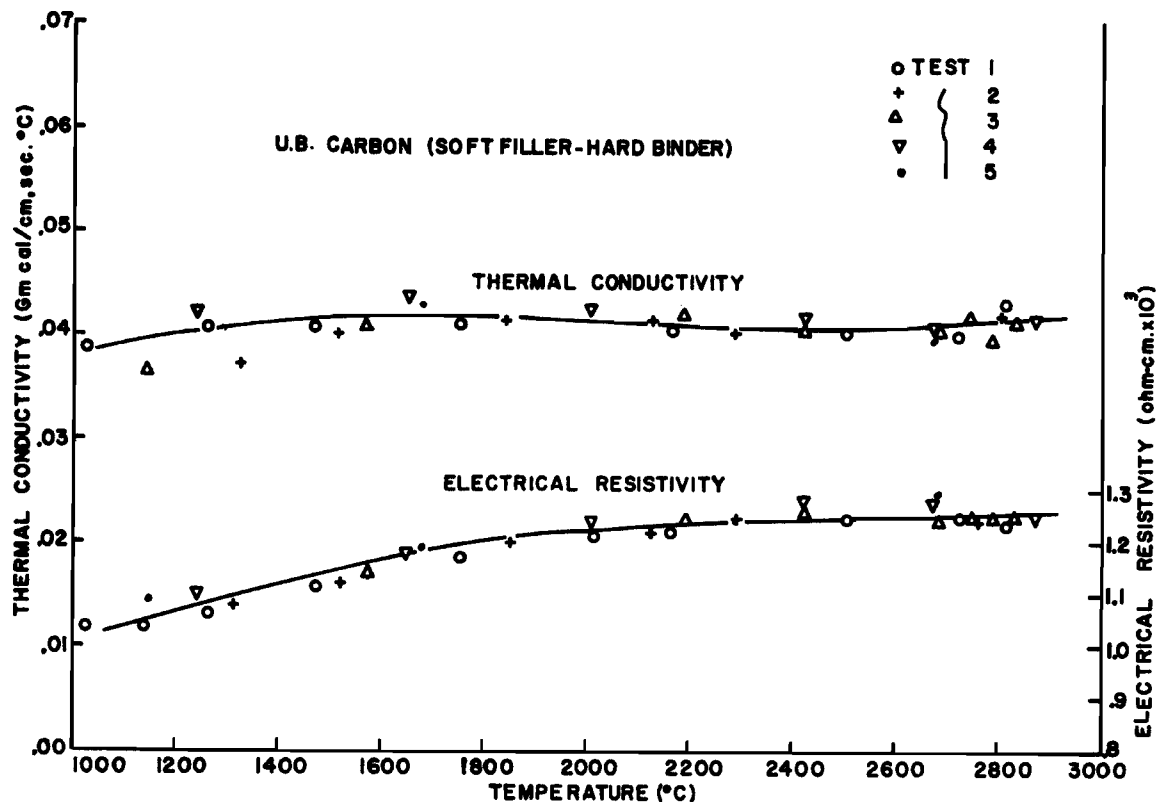


Figure 17 Thermal Conductivity and Electrical Resistivity vs. Temperature of Graphite made from Soft Filler and Hard Binder Carbons

treatment temperatures. The rod was first heated to a temperature of 1200°C. and then cooled to room temperature. It was then heated internally by applying voltage to it and data were obtained to calculate the thermal conductivity and electrical resistivity at various temperatures but limited to a maximum of 1200°C, the heat treatment temperature. The rod was then heated externally to a heat treatment temperature of 1500°C. and then the above procedure repeated.

The heat treatment was further increased in steps to temperatures of 1800°C, 2100°C, 2400°C. and 2800°C. and after each heat treatment temperature, the thermal conductivity and electrical resistivity were measured for various temperatures up to the heat treatment temperature. The results of these measurements are given in Figs. 18 and 19.

These two sets of curves show the changes that occur in the baked carbon as it is heat treated up and through the graphi-

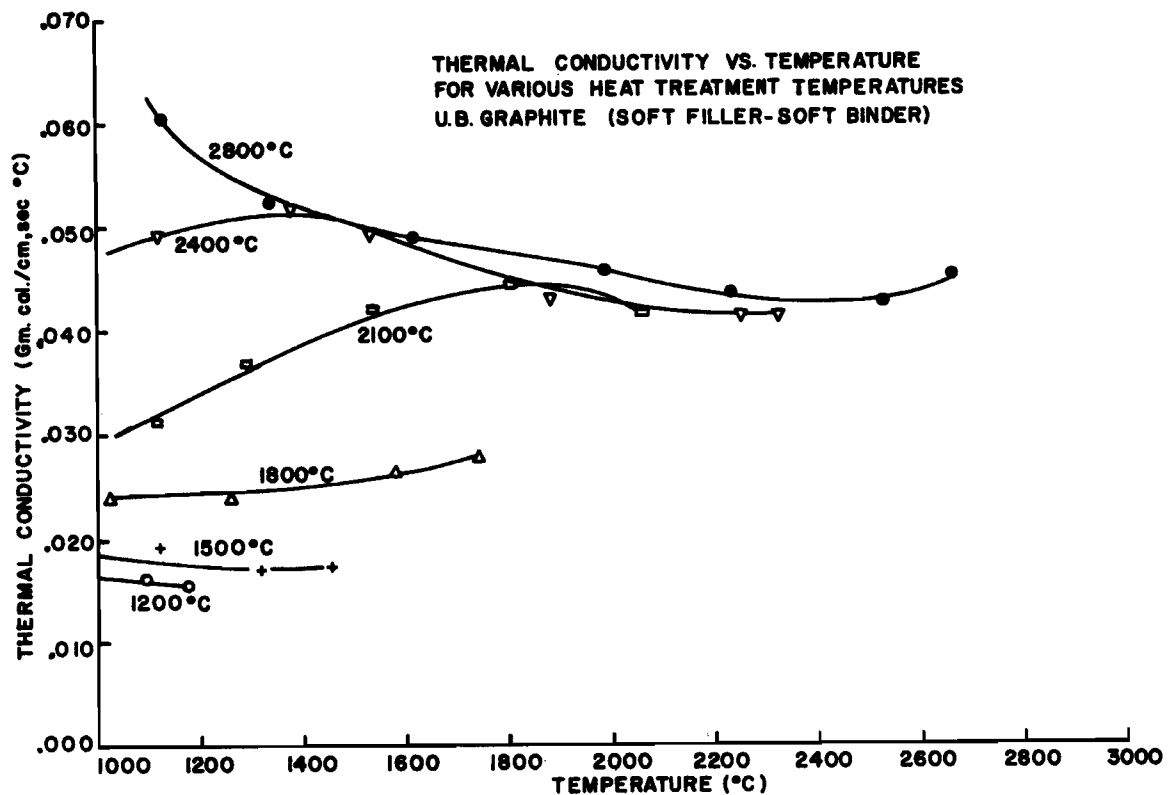


Figure 18 Thermal Conductivity vs. Temperature for Various Heat Treatments of Carbons made from Soft Filler and Soft Binder Carbon

tizing temperature range. Of special interest is the maximum of heat conductivity values observed at heat treatments of 2100°C. and greater. The position of this maximum shifts to lower temperatures as the heat treatment temperature is increased above 2100°C. and apparently shifts to temperature below 1000°C. for heat treatment of 2800°C. The observed system of curves is in good agreement with the expected temperature dependence of thermal conductivity as a function of crystallite size as predicted by Mrozowski⁹ (see also Figure 6 of the review by Castle¹⁰).

The variation of electrical resistivity (Fig. 19) shows a noted decrease in resistivity with increased heat treatment temperature. For a given heat treatment the temperature coefficient of the resistivity is negative until heat treatment temperatures above 2400°C. have been reached. After this the resistivity shows a positive temperature coefficient. It is well to note that the electrical resistivity varies linearly with

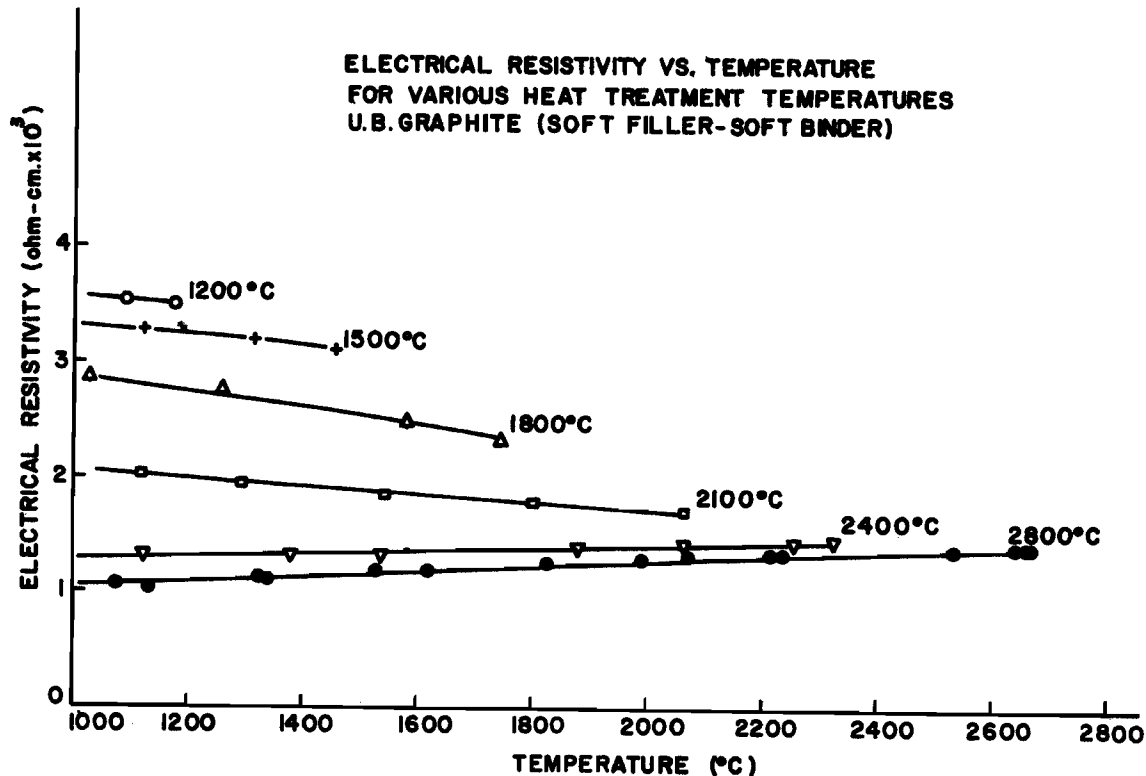


Figure 19 Electrical Resistivity vs. Temperature for Various Heat Treatments of Carbon made from Soft Filler and Soft Binder Carbons

temperature and does not exhibit maximum or minimum values as does thermal conductivity. The intermediate stage of a curve with a minimum located above 1000°C. was probably missed between 2100°C. and 2400°C. heat treatments. One can see that there is no direct relation between the thermal and the electrical conduction in carbons.

C. Measurements without Internal Heat Generation

In an attempt to measure thermal conductivity of a carbon without internally heating it by an electric current flowing through the specimen, the conductivity test chamber was modified as shown in Fig. 20. The test specimen was made in the form of a thick walled cylinder heated from the inside by means of a carbon rod heating element, replacing the test rod in the original test apparatus. The test sample was insulated at each end by providing cylindrical shields so that longitudinal heat losses were negligible. The test chamber was evacuated so that radiation heating of the test cylinder resulted in a uniform

radial heat flow. The radial heat flow was equated to that generated per unit length in the heating element by measuring the current flow and the voltage drop along a given length. The radial temperature gradient was determined by measuring the temperature at various depths of holes drilled into the thick cylinder.

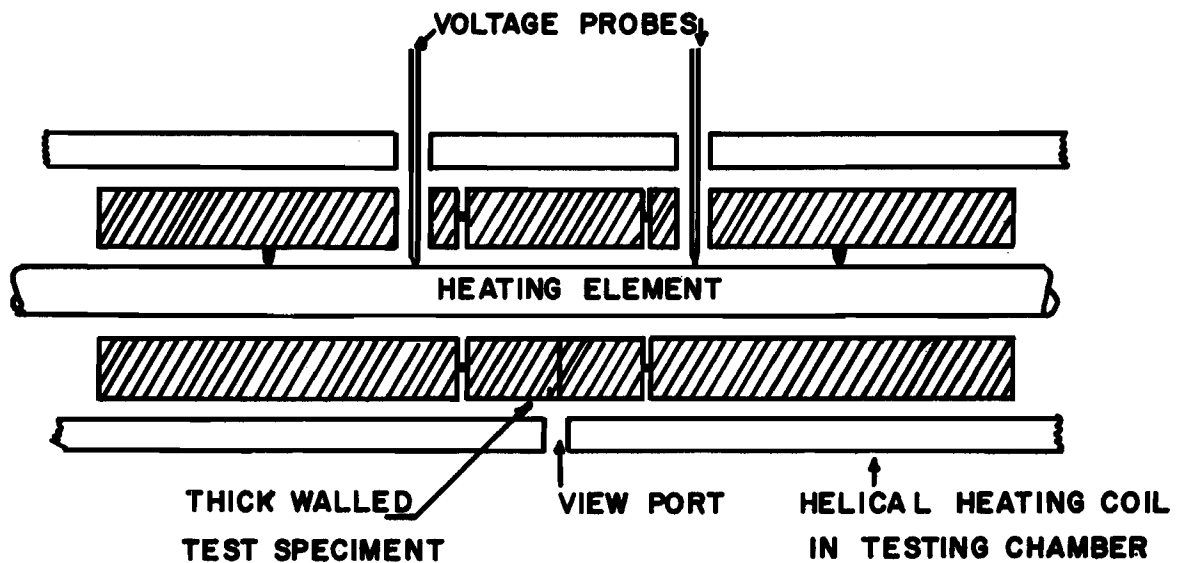


Figure 20 Arrangement for Conductivity Measurement using Thick-Walled Cylinder Method

Preliminary results of the thermal conductivity measured by this technique are shown in Fig. 21 for several repeat test runs. The values obtained are in agreement with results obtained previously by using the internal electrical heating

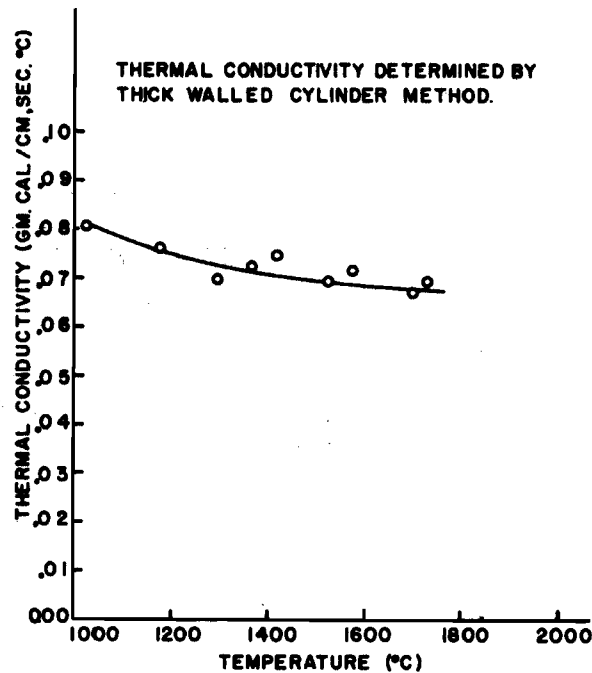


Figure 21 Thermal Conductivity vs. Temperature as Determined by Thick-Walled Cylinder Method

method. One disadvantage of this technique is the limited temperature range for which conductivity can be determined, namely only up to about 1800°C. The main advantage of this method is that it allows specimens with different crystal orientation to be investigated to determine the anisotropic variation in conductivity. The difficulties in aligning the test specimen within the test chamber, in addition to limited temperature range that can be investigated, limit the usefulness of this technique.

VI. THERMAL DIFFUSIVITY

A. Introduction

The purpose of this investigation is to measure the thermal diffusivity of different kinds of graphite at high temperatures.

Two methods, the transient heat flow and the "steady" sinusoidal heat flow, were chosen. The applied techniques and the obtained results were presented in the previous reports^{2,3}. However, the previously reported values of the thermal diffusivity³ are inaccurate due to surface oxidation of the specimen and to low response time of the measuring equipment.

The work performed since was directed to improve the experimental conditions; substantial progress has been achieved. The problem of oxidation appears solved. In the transient heat flow method the decay of the surface temperature was measured by a radiamatic pyrometer. It was discovered that the speed of response of the radiamatic pyrometer caused a time lag between the actual and measured temperatures, thus introducing a serious error which reduced the value of the measured thermal diffusivity. Experiments performed by using a new infrared radiation pyrometer, the Thermodot (model TD-6) with response times of 0.3 sec., 0.03 sec., and < 0.03 sec. showed an increase in the values of the thermal diffusivity with decreasing response time. No change in the measured thermal diffusivity was found when the response time was decreased below 0.03 sec. The new values of the measured thermal diffusivity are at maximum only 25 per cent lower than the corresponding calculated values of K/dc as compared to a maximum of 50 per cent difference obtained previously.

A furnace and the apparatus required to measure the thermal diffusivity by the "steady" sinusoidal heat wave were built and completed. To check the operation some preliminary measurements were performed on copper.

B. Transient Heat Flow Method

The thermal diffusivity was measured on five different types of graphites initially heat treated to about 3000°C. The Thermodot Model TD-6 with the response time of 0.03 sec. was used to measure the decay of temperature at the surface. Prior to each series of tests the furnace was flushed three times with Argon and an Argon pressure exceeding atmospheric by 3 cm Hg was maintained during the tests.

The curves of the temperature dependence of the thermal dif-

fusivity in direction perpendicular to the extrusion axis are presented in Fig. 22 for University of Buffalo graphite "R" $d = 1.55 \text{ g/cm}^3$, Graphite Specialties Company graphite "W" $d = 1.86 \text{ g/cm}^3$, and University of Buffalo graphite "Z" $d = 1.32 \text{ g/cm}^3$ and in Fig. 23 for U.B. graphite "G" $d = 1.55 \text{ g/cm}^3$ and U.B. graphite "A" $d = 1.33 \text{ g/cm}^3$ (see Table 2). The reproducibility of the results in Fig. 22 appear to be reasonably good but in the case of Fig. 23 the points appear more scattered.

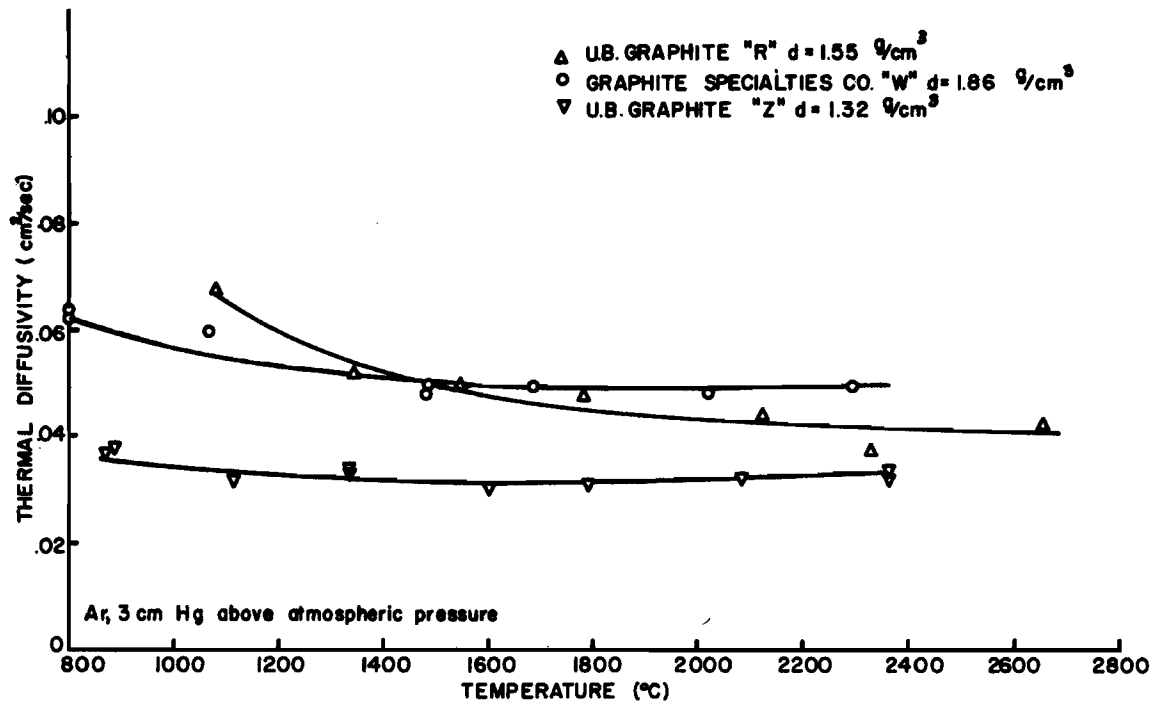


Figure 22 Thermal Diffusivity vs. Temperature for Various Graphites in the Direction Perpendicular to Extrusion Axis. Transient Heat Flow Method

Table 2
Composition of the "Green" Extruded Rods used for Thermal Diffusivity Measurements

Type	UB Graphite "R" (SF-SB)	Graphite Specialties Co. "W"	UB Graphite "Z" (HF-HB)	UB Graphite "G" (SF-SB)	UB Graphite "A" (SF-SB)
Filler Type	Texas Coke	Thermax	Phenol Formaldehyde	Texas Coke	Texas Coke
Binder Type	M-30 Coal Tar Pitch		Phenol Benzaldehyde	M-30 Coal Tar Pitch	M-30 Coal Tar Pitch
Filler Size	65/100 50 parts		100/150 50 parts	200/270 100 parts	28/35 100 parts
Binder Content	200/270 50 parts		270 50 parts	50 parts	44 parts
Density (g/cm ³) HT to 300°C.	1.55	1.86	1.32	1.55	1.33

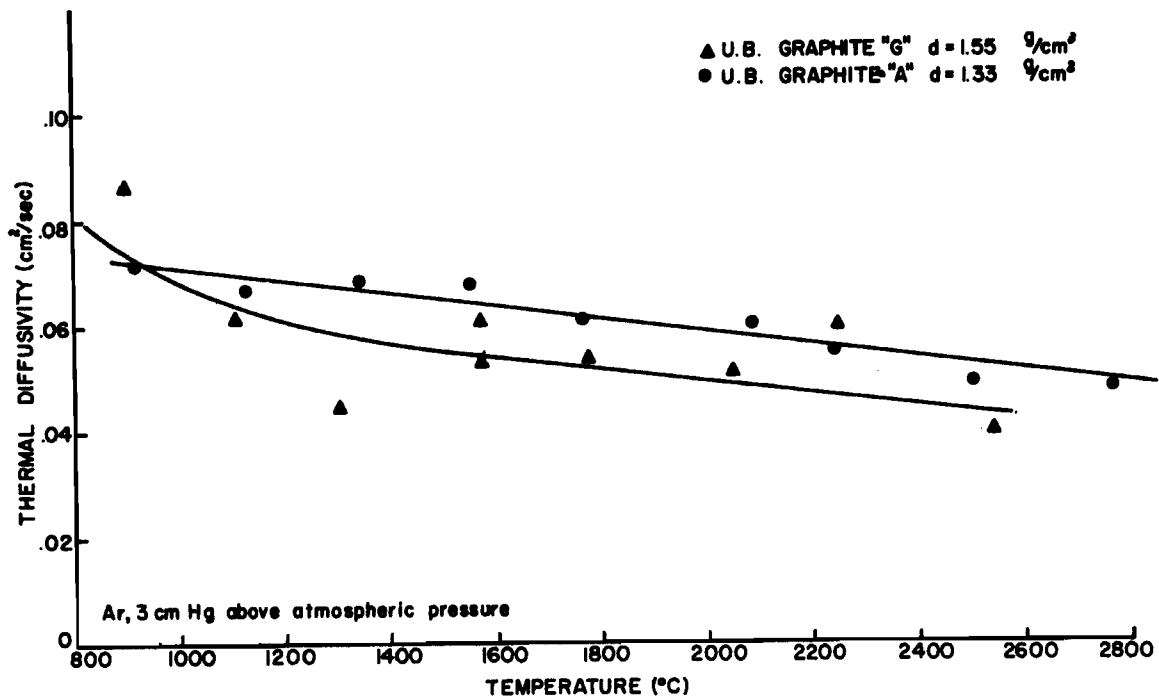


Figure 23 Thermal Diffusivity vs. Temperature for Various Graphites in the Direction Perpendicular to Extrusion Axis. Transient Heat Flow Method

Corresponding values of the thermal conductivity were obtained independently of the thermal diffusivity on the same samples, using the same experimental arrangement as previously described³. Fig. 24 shows the measured thermal conductivity vs. temperature for the various samples of graphites.

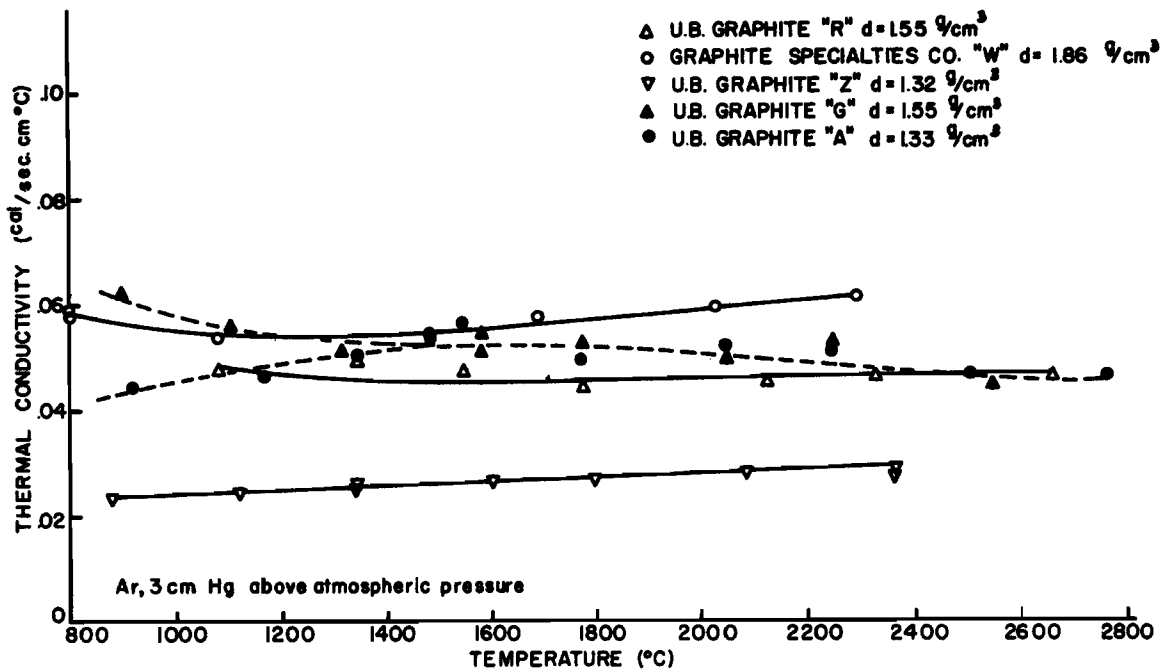


Figure 24 Thermal Conductivity vs. Temperature for Various Graphites in the Direction Perpendicular to the Extrusion Axis

The temperature dependence of the specific heat is shown in Fig. 25. The specific heat is obtained from references 12 and 13.

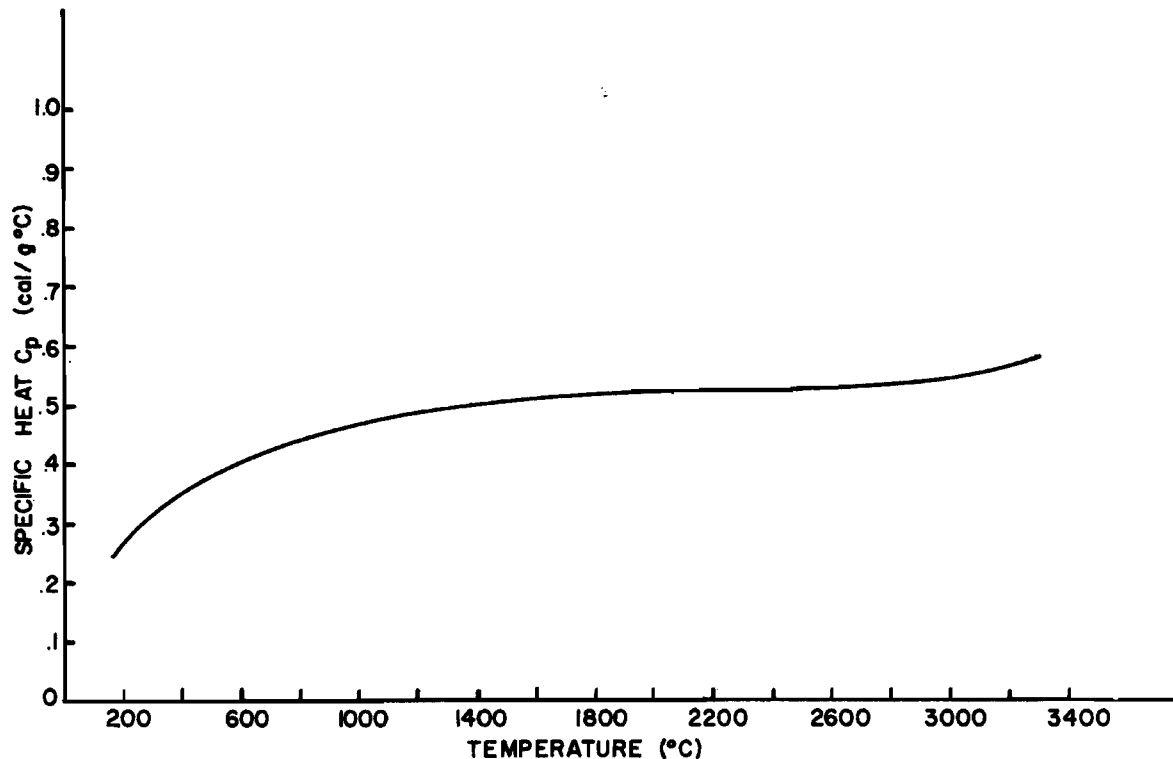


Figure 25 Specific Heat vs. Temperature plotted according to data from ref. 12 and 13

K/dc was calculated using the K , c , and d values given in Figs. 24, 25. The curves of K/dc vs. temperature for the various samples of graphite are shown in Fig. 26.

From the data Fig. 24 and 26, the percentage deviation $(a - K/dc) / K/dc \times 100$ was calculated and the values obtained for the various samples of graphites plotted vs. temperature are presented in Fig. 27. Some scattering in the percentage deviation is observed; however, a single curve can be drawn which represents the deviation for the various graphites, thus indicating that the deviation is due to some systematic factor. The curve shows that the deviation ranges from about -5 per cent at lower temperatures up to -25 per cent at high temperatures.

The influence of response time on the thermal diffusivity data appears to have been eliminated. However, the remaining discrepancy cannot be explained at the present time.

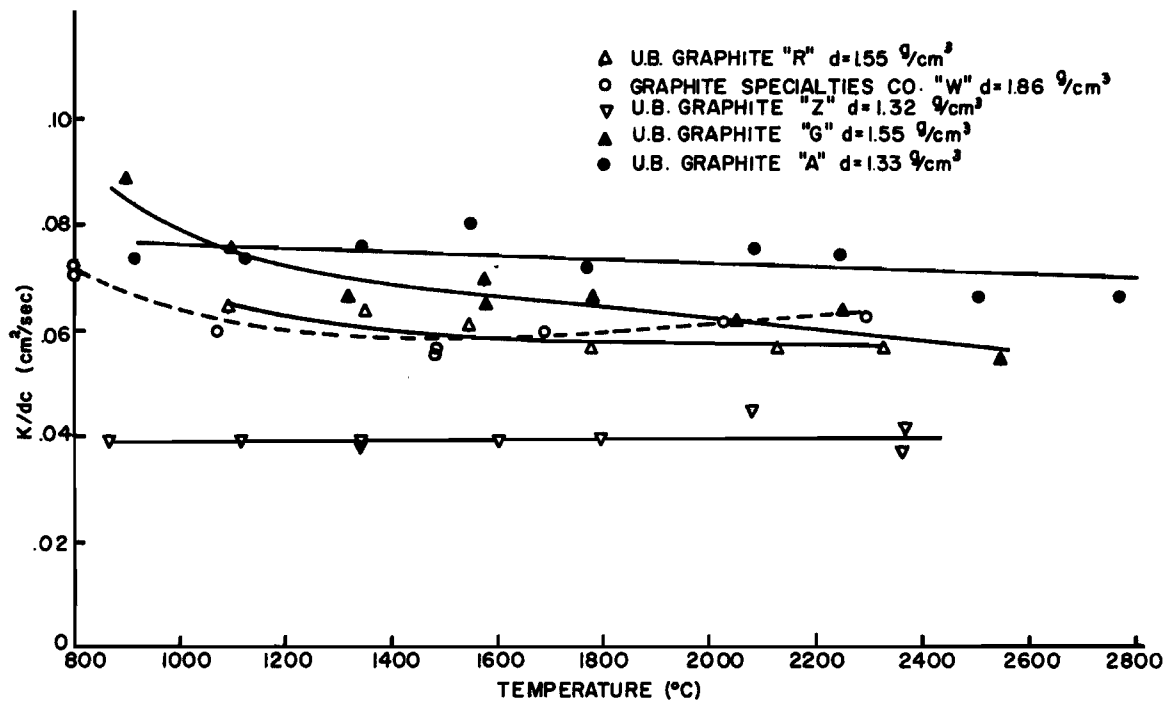


Figure 26 Ratio of the Thermal Conductivity to Density and Specific Heat vs. Temperature for Various Graphites

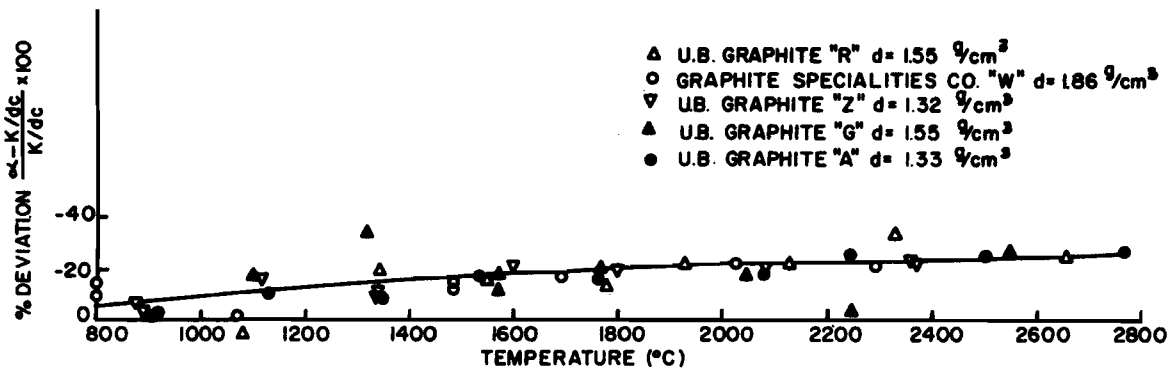


Figure 27 Percentage Deviation $(\alpha - K/dc) / K/dc$ vs. Temperature for Various Graphites

C. "Steady" Sinusoidal Heat Flow Method

The schematic drawings of the new furnace and the apparatus are shown respectively in Fig. 28 and 29. The apparatus is made of copper and can be used for measurements up to about 900°C. The specimen and the shield can be easily exchanged so that the thermal diffusivity can be measured on different kinds of materials. The experimental arrangement and procedure are similar to the ones previously described².

Calculations of the amplitude ratio A_{01} and the corresponding phase shift angle $(\Delta\phi)_{01}$ for two fixed points longitudinally located, respectively 3" and 4" from the end of the specimen, were performed by using the equations published in reference 14. The calculations were done for various values of $\sqrt{\omega/2\alpha}$ and of the heat loss factor $b = hp/ACd\omega$, where ω is the angular velocity of the sinusoidal temperature wave, h is surface coefficient of heat transfer, b is outside parameter of cross section of specimen, A is cross sectional area of specimen, C is specific heat and d is density. Considerable time was spent on performing these calculations by hand. However, later, the computations were expanded and checked by using an IBM 1620 computer. The values obtained by hand and computer agreed closely.

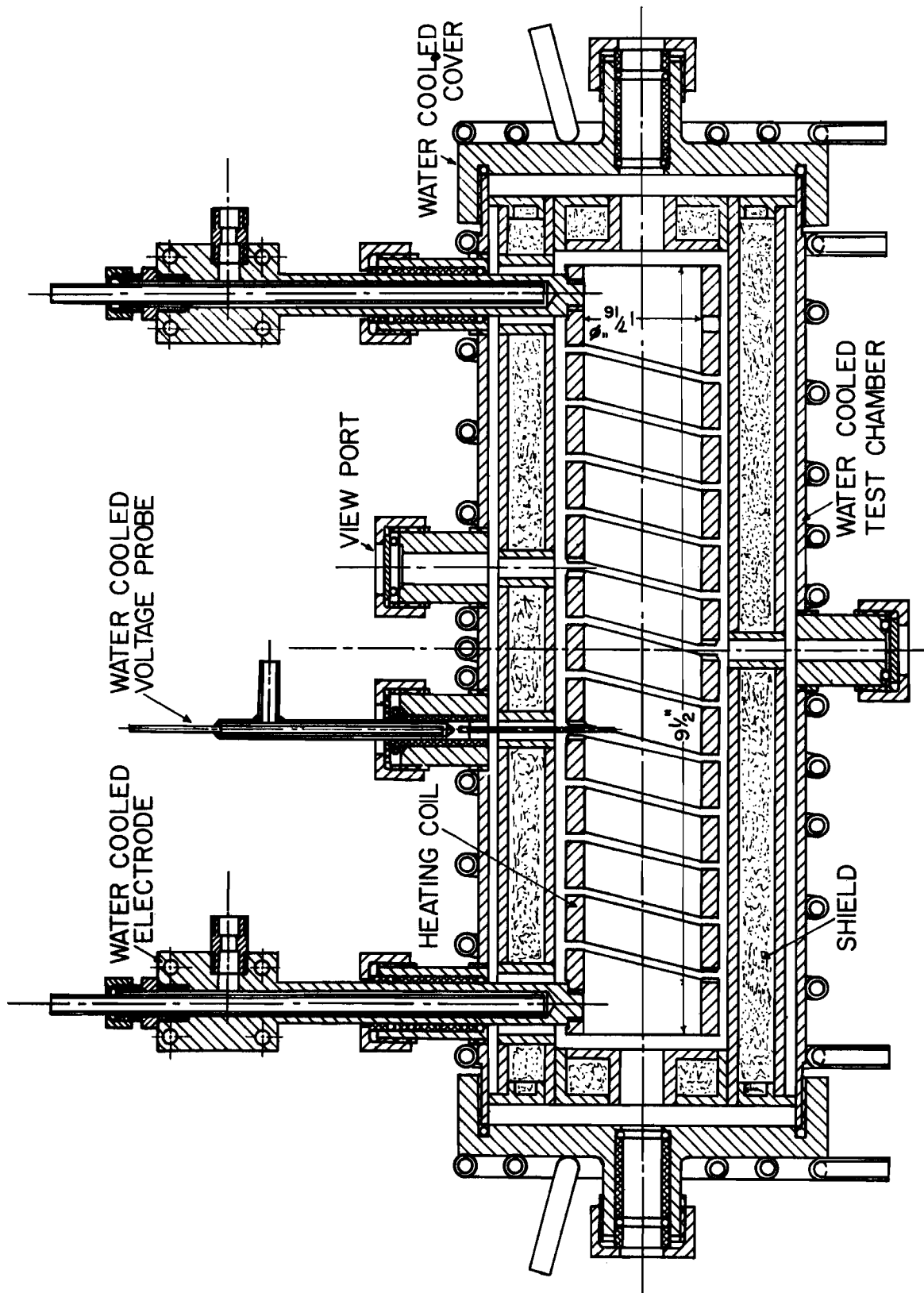


Figure 28 Schematic of the Furnace for the "Steady" Sinusoidal Heat Flow Method

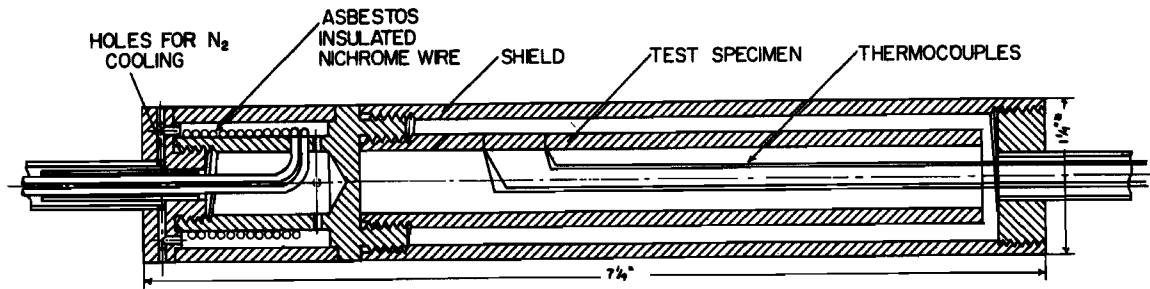


Figure 29 Schematic of the Apparatus for Thermal Diffusivity Measurements

Two charts (Fig. 30 and 31) were prepared where $\sqrt{\omega/2\alpha}$ is respectively plotted vs. the amplitude ratio A_{01} and vs. the phase shift angle $(\Delta\phi)_{01}$ for various values of the heat loss factor. The charts were then combined into one universal chart (Fig. 32) where $\sqrt{\omega/2\alpha}$ is plotted vs. the heat loss factor b for various values of the amplitude ratio A_{01} and the phase shift angle $(\Delta\phi)_{01}$. Fig. 32 shows that experiments leading to values of A and $\Delta\phi$ located in certain regions of the chart should be avoided because α obtained from A_{01} and $(\Delta\phi)_{01}$ is not single valued when the heat loss factor b is unknown.

The evaluation of the thermal diffusivity α for an independently selected angular velocity ω is now a simple matter when the amplitude ratio A_{01} and the phase shift angle $(\Delta\phi)_{01}$ are determined from the recorded temperature waves.

The previously reported diffusivities^{2,11} measured on National Carbon Company graphite type (CS) were checked by using this chart. Unfortunately, the data showed that b was negative in this case and the exact values of the diffusivity could not be obtained because the chart was only prepared for positive values of b . Nevertheless, using $b = 0$, the values agreed fairly closely with the previously reported values, however, the latter ones determined by an approximate method appear to be more accurate than the value obtained for the chart by assuming $b = 0$.

Preliminary measurements were performed on copper in the temperature range $100^\circ - 700^\circ\text{C}$. in order to check the performance of the equipment. However, due to the very high diffusivity of copper and to variations in the power supply used for the external heater, erratic results were obtained. An effort is being made to eliminate these difficulties in order to obtain reasonably good results.

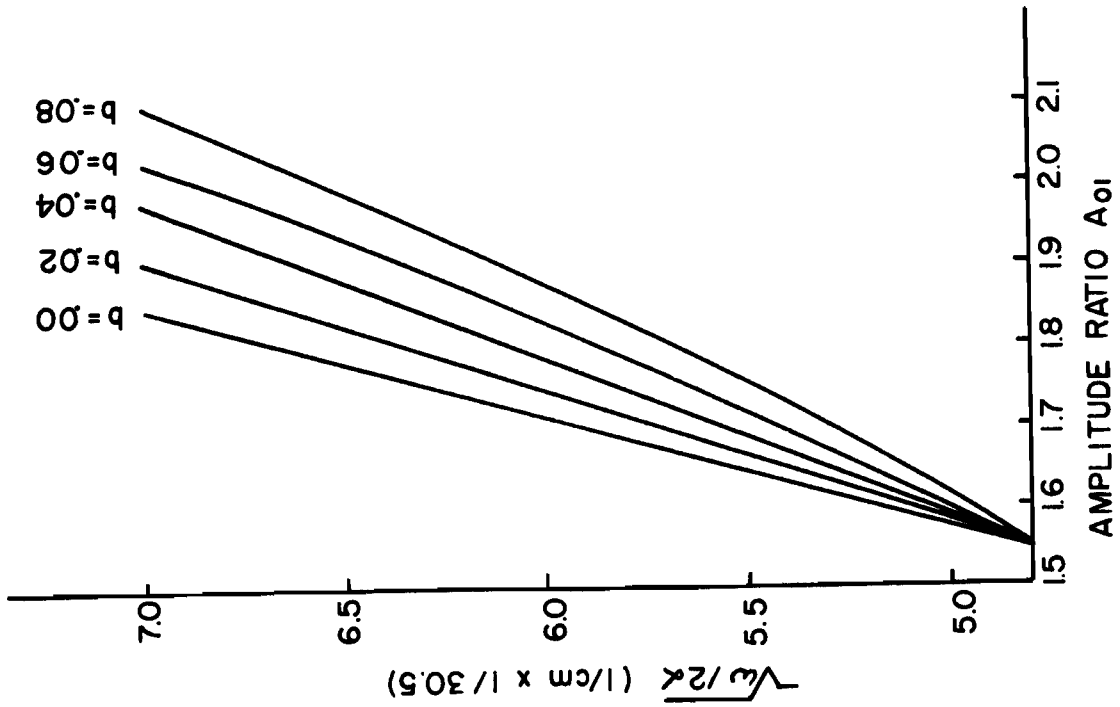


Figure 30 $\sqrt{\omega/2\alpha}$ vs. the Heat Loss Factor b

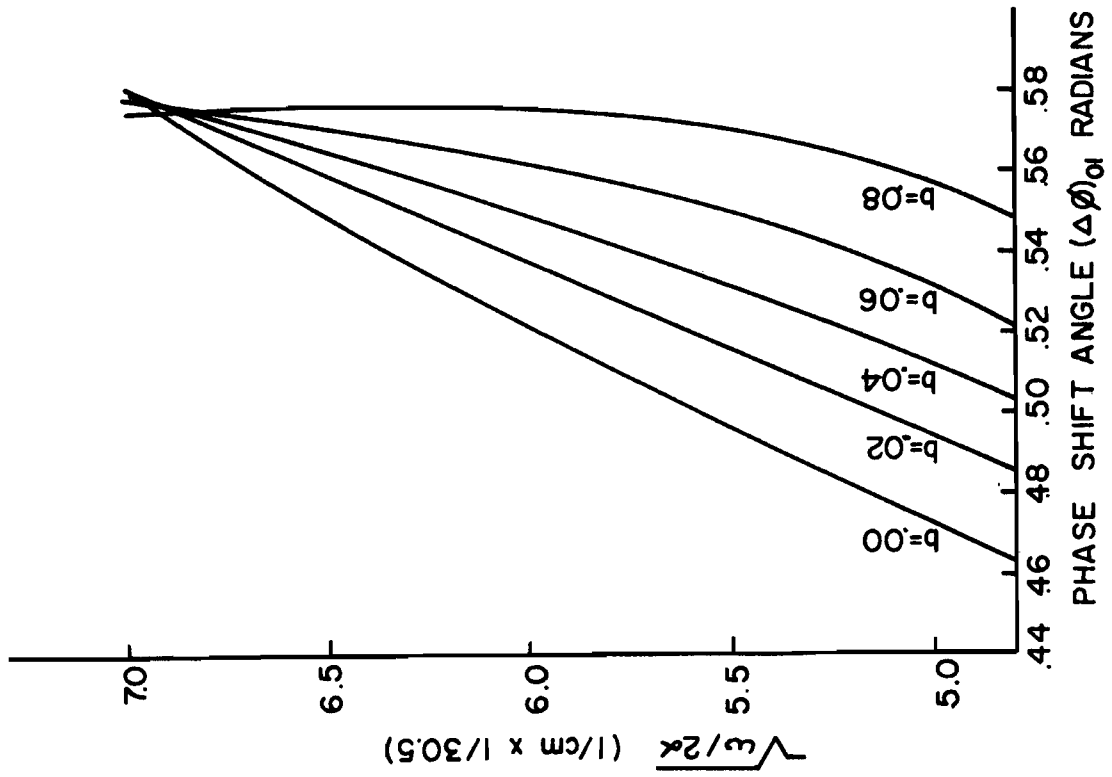


Figure 31 $\sqrt{\omega/2\alpha}$ vs. the Heat Loss Factor b

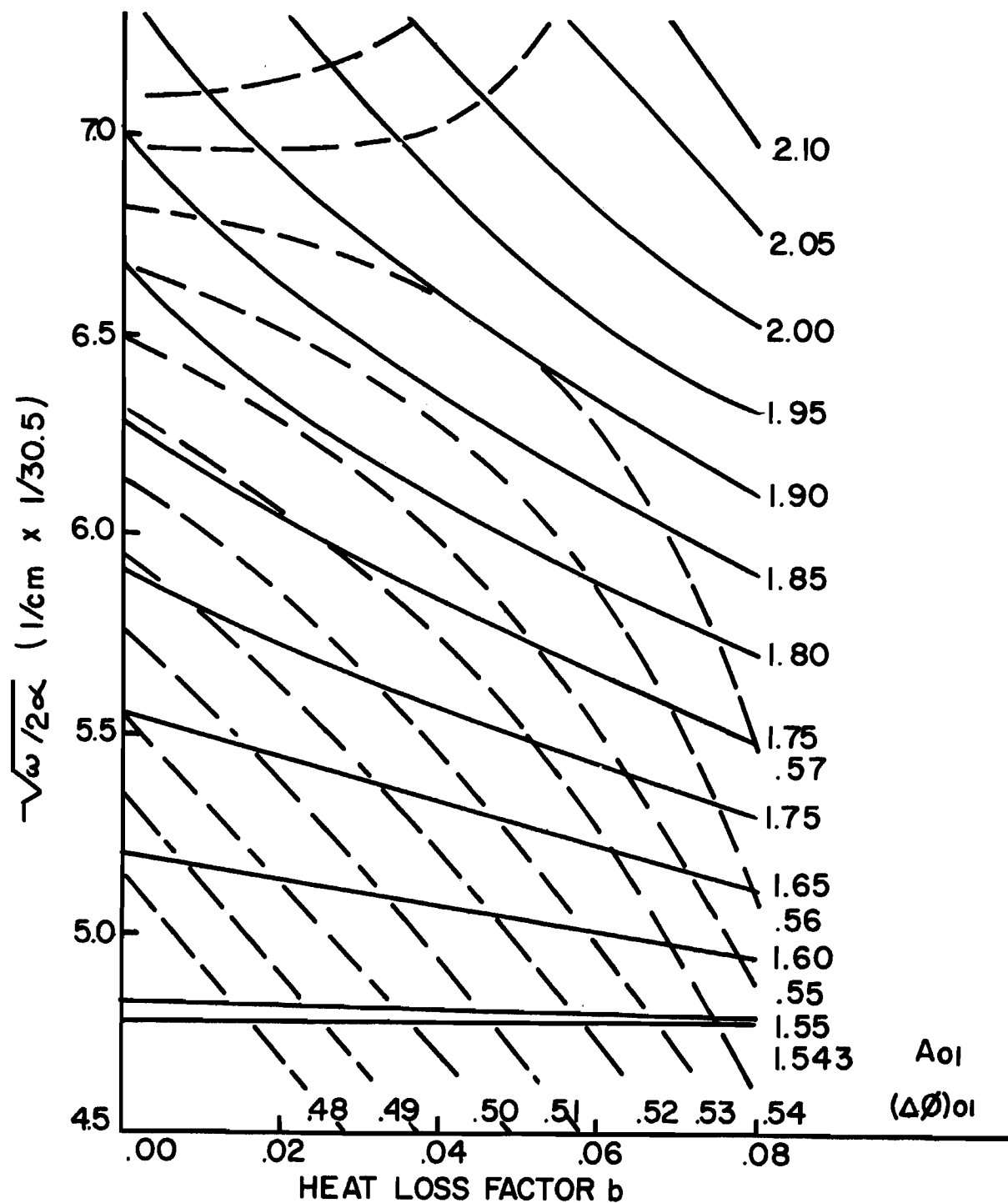


Figure 32 $-\sqrt{\omega/2a}$ vs. the Heat Loss Factor b for values of A_{01} and $(\Delta\phi)_{01}$

VII. REFERENCES

1. WADC Technical Report 58-360, Part I, "Investigation of Elastic and Thermal Properties of Carbon-Base Bodies," University of Buffalo, December 1958.
2. WADC Technical Report 58-360, Part II, "Investigation of Elastic and Thermal Properties of Carbon-Base Bodies," University of Buffalo, March 1960.
3. WADC Technical Report 58-360, Part III, "Investigation of Elastic and Thermal Properties of Carbon-Base Bodies," University of Buffalo, February 1961.
4. H. W. Davidson, H. H. W. Losty and A. M. Ross, Industrial Carbon and Graphite, Society of Chemical Industry, London (1959), p. 551.
5. S. Mizushima, Proceedings of the Fifth Carbon Conference, Pergamon Press (in press).
6. F. V. Fair and F. M. Collins, Proceedings of the Fifth Carbon Conference, Pergamon Press (in press).
7. S. Yamada and H. Sato, to be published in Nature No. 9, 743 (1962).
8. See for example, A. Granato and K. Lucke, J. Appl. Phys. 27, p. 583 and 789 (1956).
9. S. Mrozowski, Phys. Rev. 86, 251 (1952).
10. J. G. Castle, Proceedings of the First and Second Carbon Conference, University of Buffalo (1956) p. 13.
11. N. H. Juul, Proceedings of the Fifth Carbon Conference, Pergamon Press (in press).
12. "The Production and Properties of Graphite for Reactors" National Carbon Company (1955) p. 28.
13. WADC Technical Report 56-400 Part I, "Thermal Properties of Materials" 21 (1957).
14. M. A. El-Mifni and B. T. Choo, ASME Vol. 78, 813 (1956) Part II.