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EFFECTS OF NEUTRON DAMAGE ON THE MECHANICAL
PROPERTIES OF PYROLYTIC AND SINGLE CRYSTAL GRAPHITE

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Principal Investigator
J. T. Meers ✓

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FOREWORD

This report summarizes the progress of an investigation of the effects of neutron damage on the mechanical properties of both graphite single crystals and pyrolytic graphite which has been compression annealed. The work is jointly supported by the United States Atomic Energy Commission under Contract No. AT-(40-1)-3237 and Union Carbide Corporation, Carbon Products Division. The contract is administered by Dr. D. F. Cope, Director, Reactor Division, Oak Ridge Operations. The report covers the period July 1, 1965 to June 30, 1966.

The Project is under the general supervision of J. T. Meers. The graphite single crystal work has been carried out by D. E. Soule, O. L. Blakslee, D. G. Proctor, and C. W. Nezbeda. The pyrolytic graphite work has been carried out by O. L. Blakslee, N. F. Graves, E. J. Seldin and G. B. Spence. This report was prepared by J. T. Meers and E. J. Seldin.

ABSTRACT

A careful analysis has been made of the optical reflectivity method of measuring the c-axis misorientation of the pyrolytic graphite. The static shear apparatus has been revised to allow the measurement of both shear modulus and shear strength of single crystals. The ultrasonic method has also been used successfully for the determination of the shear modulus of single crystals. The static torsion apparatus for the testing of pyrolytic graphite has been automated to make the testing more convenient. Calculations have been made to determine the feasibility of measuring c_{13} on compression-annealed pyrolytic graphite; with present measuring techniques, a reliable value of c_{13} probably cannot be obtained directly from experiment.

All of the specimens for the irradiation program have been prepared; the preirradiation tests on the specimens have been performed, and the specimens have been sent to Battelle Northwest for neutron irradiation. The results of the preirradiation tests are reported here.

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1. INTRODUCTION

The general objective of the program is to determine the effects of neutron irradiation upon the mechanical properties of graphite single crystals and of highly ordered pyrolytic graphite. The specific objective for the work during the first year was the production and preirradiation evaluation of these materials. This work has been completed and is described in the first annual summary report.⁽¹⁾

Work during this period has been directed mainly toward the production of material and the fabrication and preirradiation evaluation of the test specimens for the irradiation program. This work has been completed. In addition, some of the experimental methods and techniques have been improved, and measurements of the shear modulus of the single crystal have been made. These measurements of the shear modulus had been considered impossible during the first year of this program.

2. SUMMARY

A sufficient amount of good quality compression-annealed pyrolytic graphite has been prepared for the irradiation program from the M-300 series of pyrolytic graphite disks. Specimens have been prepared for a total of nine capsules, which represent nine different irradiation conditions. All of the preirradiation tests on the specimens have been performed, and the results are summarized in this report. The specimens have all been sent to Battelle Northwest for irradiation.

The optical reflectivity apparatus, which is used to measure the surface flatness of compression-annealed pyrolytic graphite, was subjected to a careful analysis in order to correct for the inherent broadening effect of the apparatus. The measured half-width at half maximum, $\phi_{\frac{1}{2}}$, is related to the half-width which is a function of the graphite surface, $\theta_{\frac{1}{2}}$, and to the inherent half-width of the apparatus, $\psi_{\frac{1}{2}}$, by the expression:

$$\theta_{\frac{1}{2}} = \frac{1}{2} \sqrt{\phi_{\frac{1}{2}}^2 - \psi_{\frac{1}{2}}^2} .$$

This expression was used to correct the measured half-widths of the compression-annealed pyrolytic graphites. A representative half-width has been obtained from at least one specimen of each of the compression-annealed pyrolytic graphite disks which are being used in the irradiation program.

The apparatus which was made for the measurement of the shear strength of single crystal graphite was modified to measure the shear modulus of very thin specimens. The reliability of the apparatus was proved by measuring specimens of compression-annealed pyrolytic graphite, the shear moduli of which had been measured previously by the ultrasonic and static torsion methods. The agreement between the different measurements was excellent.

The ultrasonic test method has also been used successfully to determine the shear modulus c_{44} of small single crystal specimens. The range of values obtained for the shear modulus has widened, and the latest values approach more closely those calculated from specific heat data and from neutron diffraction tests.

A new torsion apparatus has been constructed; it is similar in principle to the apparatus used in previous work, but it has been automated to make data collection more convenient.

Calculations to determine the feasibility of measuring the stiffness constant, c_{13} , for compression-annealed pyrolytic graphite by ultrasonic techniques show that there is little hope of determining this property accurately from a measurement of the velocity of a quasi-transverse ultrasonic wave. Although the possibility exists of introducing the waves at the desired angle into the c-face of a specimen by a refraction technique, the effort required to develop this method can be more profitably expended in measuring the other four stiffness constants.

3. MATERIALS PREPARATION

The pyrolytic graphite materials which were used during the first year of the program were compression annealed at the Carbon Products Division, Advanced Materials Laboratory, Lawrenceburg, Tennessee. During the second year of the program, the work at Lawrenceburg was discontinued and two furnaces and the molds and rams required for compression annealing were fabricated for use at the Parma Technical Center. The furnace characteristics and the behavior of the load train under pressure and temperature were determined in the first half of the second year using ordinary commercial pyrolytic graphite as specimens. A multi-step annealing sequence was adopted, and most of the good quality material used for the irradiation program was fabricated at Parma from the M-300* series of pyrolytic graphite disks. Each disk was first subjected to a high temperature, low pressure anneal. The surfaces of the disk were then stripped to expose layer planes with no, or very few, cleavage steps. After stripping, the compression-annealed disk was conical shaped; this effect was a result of the original pyrolytic disk having been deposited on a conically-shaped substrate surface. At least two and usually three or more compression anneals, with intermediate stripping, were required to flatten and improve the quality of each disk. Prior to each compression anneal, the sample and tooling were machined or reshaped as required. Nine disks of the M-300 series were successfully processed by the above procedure. A typical finished compression-annealed disk is shown in Figure 1; some tensile specimens taken from the interior of this disk are shown in Figure 2.

A fourth furnace load of special methane-base pyrolytic graphite, designated the M-400 series, was ordered as backup material for the M-300 series in the event that more material would be needed for the irradiation program. The surfaces of all the disks of the M-400 series were uniformly

* M-300: M - Methane base, 3xx - Third furnace load

Figure 1
Disk of Compression-Annealed Pyrolytic Graphite M325
(Approximately 3-inch diameter x 0.25-inch thick)

N-9611

Figure 2
Two Tensile Specimens From Disk M325

N-9612

bright, and the grain structure was fine and very uniform. However, the techniques which were successful with the M-300 material did not work for the M-400 material. Several disks of the M-400 series were compression annealed, but they have so far yielded no acceptable material for the irradiation program.

4. MATERIALS CHARACTERIZATION

4.1 Criterion of Acceptable Quality

The criterion for the acceptability of material for irradiation test specimens which was given in a previous report ⁽²⁾ evolved from the several tests which have been used to characterize the compression-annealed pyrolytic graphite. During the first year's work, it was believed that compression-annealed pyrolytic graphite, the crystallite c-axes of which had an angular distribution of 1° half-width at half maximum, as obtained from X-ray or optical reflectivity measurements, would give an adequate approximation to a graphite single crystal. However, as the optical reflectivity measurement was perfected, this requirement was found to be inadequate. The next step in establishing a criterion of acceptability evolved from the static* tensile test. It was initially found that linear stress-strain curves were consistently obtained only when the material had a Young's modulus ($1/s_{11}$) of approximately 85×10^{11} dynes/cm² or greater; this value of $1/s_{11}$ was, therefore, arbitrarily taken as an acceptable lower limit. However, static tensile tests of the most recently prepared compression-annealed pyrolytic graphite have yielded linear stress-strain curves for samples having moduli ($1/s_{11}$) as low as 50×10^{11} dynes/cm². The improvement in the appearance of the static tensile test results is believed to be caused by greater sample uniformity throughout the gage section, a direct result of the more effective annealing sequence adopted for the sample preparation. These measurements have persuaded us to relax the previously adopted criterion of acceptability, and some of the tensile specimens which were included for irradiation tests had moduli below 85×10^{11} dynes/cm²; the lowest modulus was 72.8×10^{11} dynes/cm².

* The term "static" refers to tests made in which the sample was subjected to static loads which produce macroscopic strains within the sample.

4.2 Optical Reflectivity

E. J. Seldin

A need to improve the resolution of the optical reflectivity apparatus arose when some very flat compression-annealed pyrolytic graphite (M304) was produced, the half-width at half maximum of which, as measured with the apparatus, was comparable with that measured for a plane mirror. As a result, the apparatus was carefully aligned, and the experiment itself was analyzed. The resolution was greatly improved by careful adjustment of the apparatus and by the introduction of a small aperture directly in front of the detector. In the analysis of the experiment, a formula was derived which relates the measured half-width at half maximum to a half-width which is a function of the surface and to an inherent half-width of the apparatus.

Apparatus

Figure 3 shows the optical reflectivity apparatus. An incandescent light bulb source illuminates the slit of a collimator, and the beam of light from the collimator is directed at an arbitrary fixed angle of incidence on the surface of the specimen, from which it is reflected into a telescope. At the focal point of the objective lens of the telescope is a silicon diode detector the electrical resistance of which decreases as the intensity of the light which falls on it increases. The telescope scans the reflected light beam at a constant rate of rotation while the intensity of the light beam, as detected by a simple circuit containing the diode, is recorded on a strip chart recorder. The half-width at half maximum of the recorder tracing of intensity vs. the angle of rotation is then taken as an index of the flatness of the reflecting surface.

The optical reflectivity apparatus is basically a spectrometer. The resolution of the apparatus was maximized by replacing the silicon diode with an eyepiece lens with cross-hairs. The standard adjustment techniques for aligning a spectrometer were employed to focus the collimator, and the central axis of the collimator was adjusted so that it was in the plane swept out by the central axis of the telescope during rotation. The silicon diode was then inserted in the end of the telescope, and the direct beam from the collimator was scanned. The position of the silicon diode and the width of

Figure 3
Optical Reflectivity Apparatus

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the collimator slit were adjusted to give the narrowest output response curve on the recorder. A collimator slit width of 0.1 mm was close to the optimum size; a further reduction in slit width did not reduce the base width of the output response curve but led instead to a triple peak in the curve which is believed to be a diffraction effect. The half-width at half maximum intensity which was obtained after this procedure measured 0.05 degree, a value which was a little lower than the lowest half-width previously obtained with the apparatus. The reflected beam from an optical flat was then scanned, and the same half-width was obtained.

A further improvement was made in the resolution of the apparatus by introducing a small aperture directly in front of the silicon diode detector. The diode has an effective light collecting area with a diameter of 1 mm. Since the image of the collimator slit is, in the case of reflection from an optical flat, approximately equal to the slit width, i.e., 0.1 mm, most of the width of the output response curve was due to the effect of scanning a narrow beam with a wide detector. Circular apertures with several different diameters were tried, and an aperture of 0.2 mm diameter was finally chosen: the half-width at half maximum was reduced to 0.015 degrees. This half-width is believed to be sufficiently small for the characterization of the surfaces of compression-annealed pyrolytic graphite. Further improvement in the resolution can be made by using a smaller aperture, but the intensity of the output signal is also reduced as the size of the aperture decreases. The aperture size which was chosen represents a compromise in that it allows an acceptably small instrument half-width and requires no major modifications in the light detection system.

Analysis of the Output Response

It is the purpose of the analysis to give a correction for instrument broadening and to indicate at what point further improvement in the resolution of the apparatus may be desirable. The experiment is illustrated schematically in Figure 4. The incident light beam is directed toward the reflecting surface at a fixed angle of incidence α and is reflected toward the telescope, which rotates and scans the reflected beam. The recorder plots the output response function $T(\phi)$, which represents the intensity of the light as a function of the angle of rotation ϕ . We shall assume that $\phi = 0$ corresponds with the central maximum of $T(\phi)$.

Figure 4

Schematic of Optical Reflectivity Test

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We assume in this analysis that the inherent broadening of the apparatus is due to the fact that all of the rays in the incident beam of light are not parallel, and we assume that the angular distribution of the intensity of the incident beam is given by some function $S(\psi)$. In the absence of a reflecting surface, the telescope scanning the incident beam would then observe the function $T(\phi) = S(\phi)$; in fact, $S(\psi)$ is obtained in just this manner. We shall assume that $\psi = 0$ at the maximum of $S(\psi)$.

The degree of flatness of the reflecting surface can be described by the function $I(\theta)$, the distribution of surface normals per unit area, where θ is the angle of deviation from the axis of rotational symmetry of the specimen. We assume that $I(\theta)$ is a symmetrical function with its central maximum at $\theta = 0$ and that it is the same for all planes passing through the axis of rotational symmetry of the specimen. For the case where the reflecting surface is an optical flat, $I(\theta)$ is a delta function, being equal to unity at $\theta = 0$ and equal to zero for all other θ .

The output function $T(\phi)$ can be related to the incident beam function $S(\psi)$ and the specimen surface distribution function $I(\theta)$ by the following reasoning. Consider, in Figure 4, a ray of light incident at some arbitrary angle $\alpha + \psi$. With the telescope set to observe only those rays incident at some angle $\alpha + \phi$, the ray incident at angle $\alpha + \psi$ will be observed only if the surface normal is at an angle θ such that $\theta = (\psi - \phi)/2$.

[Proof: The total angle between the incident and reflected ray is $2\alpha + \psi + \phi$. Half of this angle is $\alpha + (\psi + \phi)/2$. From Figure 4 and the definition of θ , we obtain

$$\theta = (\alpha + \psi) - [\alpha + (\psi + \phi)/2] = (\psi - \phi)/2.$$

Note that θ is independent of the angle of incidence α . Note also that if the positive direction of either ψ or ϕ in Figure 4 is reversed, the sign of θ is changed but the magnitude remains constant; thus, the results of this analysis are not changed.]

The intensity of the light reflected at ϕ is obtained by using the product of the intensity of the incident beam $S(\psi)$ and the surface distribution function $I(\theta)$ evaluated at $\theta = (\psi - \phi)/2$ and summing over all values of ψ . This step yields

$$T(\phi) = \sum_{\text{all } \psi} S(\psi) I\left(\frac{\psi - \phi}{2}\right) \quad (1a)$$

which also can be expressed in integral form as

$$T(\phi) = \int_{-\infty}^{\infty} S(\psi) I\left(\frac{\psi - \phi}{2}\right) d\psi. \quad (1b)$$

Two special cases are of interest:

(1) For an optical flat, $I(\theta)$ is a delta function, i. e., $I(0) = 1$ and $I(\theta) = 0$ for $\theta \neq 0$. The above equations then yield $T(\phi) = S(\phi)$, the condition which is actually observed.

(2) For a perfectly collimated incident beam of light, $S(0) = 1$ and $S(\psi) = 0$ for $\psi \neq 0$. The above equations then yield $T(\phi) = I(-\phi/2) = I(\phi/2)$. In other words, when there is no instrument broadening, the observed response $T(\phi)$ gives $I(\theta)$ directly, except that now $\theta = \phi/2$.

In the integral equation (1b), $I(\theta)$ can be considered in a mathematical sense to be an operator function which operates on the function $S(\psi)$ to produce the function $T(\phi)$. The equation then gives us a means of obtaining $I(\theta)$ from the observed $T(\phi)$ and $S(\psi)$. In practice, one can tabulate the observed input function $S(\psi)$ and the measured function $T(\phi)$ and use a standard computer program for a set of linear equations to calculate and plot the function $I(\theta)$.

Since we are not particularly interested in obtaining the exact function $I(\theta)$ but, rather, are interested in using the half-width at half maximum intensity as an index of the surface, we may use a simpler method of analyzing the data. One may assume that the input and response functions are both Gaussian; computations based on the assumption offer a fairly good approximation to the actual shapes of the input and response curves and allow us to solve the integral equation (1b) analytically for $I(\theta)$. The solution is easy to obtain because $I(\theta)$ must also be Gaussian.

Let us assume that $S(\psi) = e^{-a^2\psi^2}$ and $I(\theta) = e^{-b^2\theta^2}$. The appropriate half-widths for these functions are:

$$\psi_{\frac{1}{2}} = \frac{0.834}{a} \quad \text{and} \quad \theta_{\frac{1}{2}} = \frac{0.834}{b} \quad (2)$$

From the integral equation (1b), we obtain

$$T(\phi) = \int_{-\infty}^{\infty} e^{-a^2\psi^2} e^{-b^2(\frac{\psi - \phi}{2})^2} d\psi, \quad (3)$$

which can be written, by removing all terms which do not contain ψ outside of the integral, as

$$T(\phi) = e^{-[a^2b^2/(4a^2 + b^2)]\phi^2} \int_{-\infty}^{\infty} e^{-(a^2 + b^2/4)[\psi - b^2\phi/(4a^2 + b^2)]^2} d\psi \quad (4)$$

The integral in Equation (4) is a constant. Therefore, we can write

$$T(\phi) = \text{constant} \cdot e^{-c^2 \phi^2}, \quad (5)$$

where $c^2 = a^2 b^2 / (4a^2 + b^2).$ (6)

Since $\phi_{\frac{1}{2}} = \frac{0.834}{c}$, Equations (2) and (6) can be combined to yield

$$\theta_{\frac{1}{2}} = \frac{1}{2} \sqrt{\phi_{\frac{1}{2}}^2 - \psi_{\frac{1}{2}}^2}. \quad (7)$$

Equation (7) is a simple expression for obtaining the half-width $\theta_{\frac{1}{2}}$ of the surface distribution function from the observed half-width $\phi_{\frac{1}{2}}$ and the instrument half-width $\psi_{\frac{1}{2}}$.

Results

With the 2 mm aperture in front of the silicon diode, the instrument half-width $\psi_{\frac{1}{2}}$, as measured by scanning the direct beam from the collimator and also by scanning the reflection from an optical flat, was 0.015 degree.

When a specimen of compression-annealed pyrolytic graphite from disk M304 was used as the reflecting surface, a half-width $\phi_{\frac{1}{2}}$ of 0.10 degree was measured from the output response curve on the recorder. [Note that in the previous reports this value would correspond to $\theta_{\frac{1}{2}} = 0.05$ degree.] Using the correction formula, Equation (7), we obtain $\theta_{\frac{1}{2}} = 0.049$ degree. This measurement represents the lowest half-width which has thus far been obtained for compression-annealed pyrolytic graphite.

Past data, taken at times when the instrument half-width was not accurately known, still offer reliable qualitative characterizations of the materials tested. The analysis presented here indicates that the correction for instrument broadening becomes greater as the half-width $\theta_{\frac{1}{2}}$ of the surface distribution function approaches the instrument half-width $\psi_{\frac{1}{2}}$. The half-width $\phi_{\frac{1}{2}}$ of the output response function, which is the measured half-width, will always be equal to or greater than the instrument half-width. The resolution of the apparatus is now sufficiently high to determine accurately the half-widths for the best materials we have made. If some of the graphite surfaces to be tested in the future are appreciably more nearly flat than the best material made thus far, it may be desirable to reduce the instrument half-width still further. Optical reflectivity measurements were made on representative specimens from each disk of the M-300 series which was used for the irradiation program. The results are summarized in Table I.

TABLE I
Representative Optical Reflectivity
Half-Widths

Disk	$\theta_{\frac{1}{2}}$ (Degrees)
M-304	0.05
M-305	.95
M-306	.21
M-307	.15
M-308	.31
M-310	.73
M-311	.33
M-313	.41
M-314	.09
M-316	.13
M-317	.16
M-318	.17
M-319	.18
M-321	.17
M-323	.18
M-324	.11
M-325	.08

5. STATIC SHEAR TESTS ON SINGLE CRYSTALS AND COMPRESSION-ANNEALED PYROLYTIC GRAPHITE

5.1 Apparatus Modification

D. E. Soule, C. W. Nezbeda, and D. G. Proctor

The apparatus which was used for measuring the shear strength of single crystal graphite^(1,2) was modified to measure the shear modulus of very thin graphite specimens. The following changes were made:

1. The force couple was shifted directly to the shear heads and the

separation between the stainless and/or nylon cables was minimized.

This change reduced the effective torque on the shear heads and the compliance of the apparatus. A close view of a specimen between the shear heads is shown in Figure 5.

2. The transducer coil was shifted directly to one shear head to reduce the error in the actually measured strain.

3. Since bowing of the aluminum base plate had been detected when the load was applied, the pulleys were shifted to a separate plate positioned below the original one.

4. A Daytronic type 6 transducer exciter-demodulator was used in place of the Daytronic 201 AH to improve the sensitivity. The transducer circuitry and amplifier were impedance-matched by the Daytronic Corporation at Dayton, Ohio; this checkout, which involved minor circuit changes, reduced noise and drift and increased the sensitivity by a factor of two.

5. For the measurement of the shear modulus of a single crystal, the transverse shear carriage still had too much compliance. Therefore, a new carriage with extremely low compliance was designed which utilized a 1-inch diameter, Thompson 60-Case shaft riding in special adjustable split-ball bushing mounts. For a 10 kilogram load, the displacement of the stationary shear head was calculated to be approximately 2×10^{-5} cm. For the same load, the displacement of the moving or longitudinal shear head was calculated to be 1×10^{-6} cm. These displacements are smaller than those produced in a typical single crystal graphite sample by factors of 200 to 4000, respectively.

6. A new 3-inch diameter micrometer head replaced the old one so that the transducer circuit could be calibrated directly in divisions to 0.0001 inch.

The revised apparatus is shown in Figure 6. In order to check the capability of the apparatus, tests were made on a specimen of compression-annealed pyrolytic graphite M-311 which had previously been tested by the ultrasonic and static torsion test methods. The same value for the shear modulus was obtained as had been previously reported (0.32×10^{11} dynes/cm²). The sensitivity of the pure shear apparatus for the measurement of the shear modulus is believed to be comparable with that of the other test methods used in this study.

Figure 5
Close View of Single Crystal Specimen and Shear Heads

10 X

N-9613

5.2 Shear Modulus for Pyrolytic and Single Crystal Graphite

D. E. Soule, C. W. Nezbeda

Testing the Shear Apparatus

Two cylindrical samples, designated as F and G, were cut from a block of M-311 (D & E) compression-annealed pyrolytic graphite (shown in Figure 7) in a region previously measured nondestructively by sonic methods. Each sample was cycled a number of times by loading and unloading to progressively higher loads. The first sample, F, was cycled seven times by

Figure 6
Revised Shear Apparatus

N-9614

Figure 7

Sample Location Scheme

adding and removing a set of weights. Measurements on the second sample, G, were made by using a continuous stream of water as the load in seven tests and weights as the load in four tests. The results are given in Table II. There is excellent agreement between the static and sonic measurements; furthermore, there was no systematic change in shear modulus with the repetitive loading for either sample F or G.

TABLE II

Comparison of Static and Sonic Measurements of Shear Modulus $1/s_{44}$

Method of Measurement	Sample	$G(1/s_{44})$ (10^{11} dynes/cm ²)	Remarks
Sonic	M-311 D & E	0.0323	
Pure Shear (Static)	M-311 (D & E)F	0.0327 ± 0.0012	Ave. of 7 meas.
	M-311 (D & E)G	0.0323 ± 0.0018	Ave. of 11 meas.

Dependence of Shear Modulus on Thickness

When the accuracy of the shear technique with relatively thick pyrolytic samples was established, the task of establishing the validity of measurements made on much thinner samples, notably single crystals, was attempted. One factor, apart from a dependence on thickness itself, was the quantitative effect of light sandblasting of the sample surfaces. A sample H (3mm diameter) was cut from an area adjoining G (6mm diameter) in the M-311 (D & E) slab and cleaved to thinner pieces as shown by the "sample location scheme" in Figure 7. These samples were prepared for shear measurements in the same way that the single crystals were prepared. The relative sizes of the thickest 6mm diameter sample and one of the thin 3mm diameter samples are shown in Figure 8. Repetitive shear measurements were then made for the thickness range from 2.35mm to 0.103mm, a thickness below that usually encountered with single crystals. The results, shown in Figure 9, indicate that within the ~10 percent scatter of the data, the measured modulus is effectively independent of thickness.

The depth of the surface roughness produced by the light sandblasting was also checked under a microscope and found to average -4μ . This value represents approximately an 8 percent uncertainty in thickness for the 0.103mm case. Consequently, such a surface treatment probably does not produce a very extensive deformed region below the observed geometrical surface. This result is expected because of the resilience of graphite in the c-direction and the virtual absence of dislocation propagation in that direction. It is believed that these tests established the validity of the single crystal shear modulus measurements.

Stress-Strain Curves of Single Crystals and Pyrolytic Graphite

Stress-strain curves have been obtained both for single crystal and compression-annealed pyrolytic graphite specimens. Figure 10 shows two successive stress-strain curves obtained for the single crystal specimen EPC-3. The initial modulus was greater in the second test: this result may be an indication of strain hardening in the crystal. A residual strain was left in the specimen after the first test, but this strain was not measured. The

Figure 8
Pyrolytic Samples Used for the Thickness Dependence Measurements

11 X

N-8230

Figure 9

Shear Modulus versus Thickness for Pyrolytic Graphite

N-8460

Figure 10
Two Successive Stress-Strain Curves for a Single Crystal

N-8463

initial linear elastic portion of the single crystal stress-strain curve exists only over a very limited region of strain $\lesssim 0.0005$. If it were assumed that the strain is taken up uniformly by the entire intrinsic lattice, this strain corresponds to a shift of a carbon atom by only 0.0017A, a value representing 0.12 percent of the nearest neighbor interatomic distance (1.42A). Table III lists the values for the shear modulus $1/s_{44}$ which were obtained in static shear tests on four single crystals (EP-85, EP-86, EPC-3, EPC-4). The values are presented in the order in which they were obtained; the first two values are very preliminary and do not fairly represent the accuracy that was obtained with EPC-3 and EPC-4. The moduli determined for EPC-3 are close to the average value of 0.01×10^{11} dynes/cm² obtained by Baker and Kelly⁽³⁾ from indirect cantilever beam measurements of natural single crystals. However, specimen EPC-4 had a much higher modulus, and more recent ultrasonic test results on single crystals have yielded even higher values for the shear modulus (see Section 8). The data indicate at this time that the shear modulus of a perfect graphite crystal may be an order of magnitude or more greater than the value given by Baker and Kelly.

Two specimens of compression-annealed pyrolytic graphite were stressed a number of times in succession. Specimen M311-K was subjected to ten successive tests, and no change was observed in the value of the shear modulus as calculated from the initial slopes of the stress-strain curves. Specimen M304-C2 was subjected to four successive tests at increasingly higher stress levels; the initial modulus remained constant for the first three tests and increased on the fourth test. There is little or no evidence of strain hardening affecting the initial modulus of the compression-annealed pyrolytic graphites.

Figure 11 offers a comparison of the stress-strain curves to failure for the single crystal EPC-3 and the compression-annealed pyrolytic graphite specimen M311-K. The single crystal curve is the same as curve II in Figure 10, and the curve for M311-K is the tenth and last curve obtained for that specimen. The shear strength of the single crystal was much lower than that of the compression-annealed pyrolytic graphite, and a relatively high shear strain was sustained by the single crystal before failure occurred.

TABLE III
Basal-Plane Shear Results

<u>Single Crystal Graphite</u>				
Sample	Area (cm ²)	Thickness (mm)	$1/s_{44}$ (10 ¹¹ dynes/cm ²)	Shear Strength (10 ⁶ dynes/cm ²)
EP-85	0.203	0.070	0.011	4.2±0.8
EP-86	0.160	0.263	0.026	
EPC-3	0.200	0.381	{(I) 0.0132±0.0020 (II) 0.0176±0.0025}	
EPC-4	0.0651	0.198	0.042	
Baker and Kelly (3) (Cantilever Beam)			(0.01)	
<u>Compression-Annealed Pyrolytic Graphite</u>				
C-5			---	8.3±1.5
M-305			---	12.4±2.6
M-311-F			0.0327	12.2±1.2
-G			0.0323	
(ultrasonic)			(0.0323)	
M-304-C2			0.0244	9.2±2.6
(resonant bar)			(0.0246)	
(ultrasonic)			(0.0242)	

Figure 11
Comparison of Single Crystal
and Pyrolytic Stress-Strain Curves

N-8462

Since stress-strain curves to failure on crystals with higher shear moduli have not yet been obtained, there is some uncertainty at this time as to whether the values of shear strength and maximum strain obtained with crystal EPC-3 are truly representative of the single crystal.

5.3 Ultrasonic Tests on Single Crystals

O. L. Blakslee, C. W. Nezbeda, and D. E. Soule

Values of the shear modulus c_{44} of single crystals have been determined by measurements of the time of transit of shear waves through crystals of known thickness. The experimental technique was identical to that described in a previous report.⁽¹⁾ The specimen was cemented with Salol to one end of an aluminum buffer rod 1 x 1 1/4-inch diameter, and a transducer receiver 2 mm in diameter was cemented to the top of the specimen. Figure 12 is a photograph of the specimen and transducer receiver on top of the buffer rod. The times of transit of a 1 mc pulse through the buffer rod and specimen and through the buffer rod alone were measured. Figure 13 shows the pulse shapes observed in a typical test. The apparatus measures the time from the start of the pulse to any point on the observed transmitted signal. The time of flight through the crystal was measured by using the three peaks on the first one and one-half waves in the signals as points of reference and by taking the time differences between the positions of the three corresponding parts of the wave patterns. The two wave patterns tended to differ slightly after the second wave, probably because of reflected waves in the specimen. For single crystal specimen EPC-4, the value of c_{44} which was determined by the ultrasonic test method was $c_{44} = 0.0464 \pm 0.0077 \times 10^{11}$ dynes/cm²; in the static test which followed, a value of 0.0420×10^{11} dynes/cm² was obtained for the initial shear modulus. Other ultrasonic test results on single crystals are given in Section 8.

5.4 Comparison of Moduli Determined in Static, Sonic, and Ultrasonic Tests with Those Calculated from Other Measurements

Prior to the present series of measurements made on graphite, the only reasonably reliable c_{44} values which existed were those which were deduced from cantilever beam, low-temperature specific heat and neutron diffraction measurements. Now that reasonably accurate c_{44} values have been determined

Figure 12
Single Crystal Specimen with Receiver-Transducer
and Aluminum Buffer Rod

N-9615

for single crystals and compression-annealed pyrolytic graphite, a comparison can be made with values calculated from other types of measurement.

The data in Table IV have been compiled from the literature in order to demonstrate several significant points about both c_{44} and c_{33} as they relate to different measurements made on several types of graphite. Included in Table IV are those characteristics of the lattice, plus its imperfections, which may affect these elastic constants. The data cover a range of graphites extending from the Essex County single crystals down to the very fine grain and practically isotropic lampblack-based graphite, SA-25. The physical

Figure 13
Two Wave Forms of Transmitted Signals
With and Without Graphite Specimen

Transmitted
pulse through
buffer rod alone

Transmitted
pulse through buffer
rod and crystal EPC-4

N-9616

TABLE IV

Values of c_{33} and c_{44} as Determined by Different Methods for Various Graphites

Material	Crystallite Size	Crystallite Tilt	Layer Spacing (Å)	Stacking Fault Degree	c_{33} (10^{11} d/cm ²)	T (°K)	Measurement	c_{44} (10^{11} d/cm ²)	T (°K)	Measurement
Single Crystal Essex County	≥1 mm		3.35	Almost negligible	---	---	---	0.011-0.042 0.028-0.166	300 300	Pure shear Ultrasonic
Madagascar	>100μ		3.35	Very low	3.56	0.4-2	Spec. heat ^(7,8) (Compressibility)	0.452	0.4-2	Spec. heat ^(7,8) (4,5)
Canada	~100μ		3.35	Low	{ 3.56 ≥1.8	1.3-4.2	Spec. heat ^(4,5) " " (4,6)	0.405 0.23	1.3-4.2 "	Spec. heat ^(4,5) " " (4,6)
Ceylon			3.35	Low	3.50	300	Compressibility ⁽⁹⁾ with X-ray			
Compression-annealed pyrolytic	~30-60μ	<1°	3.35		3.44±0.10 3.65	300 300	Ultrasonic ⁽¹⁾ Static compression ⁽¹⁾	0.018-0.035	300	Pure shear ⁽¹⁾ Diagonal shear ⁽¹⁾ Torsional shear ⁽¹⁾ Sonic resonance ⁽¹⁾ ultrasonic ⁽¹⁾
Thermally-annealed pyrolytic	~100μ	~5°	3.35		3.9±0.4	300	Neutron diffraction ⁽¹⁰⁾	0.42±0.2	300	Neutron diffraction ⁽¹⁰⁾
Pile graphite (H-CS-11)	~240Å		3.35	Low	3.56	1.3-4.2 0.4-2	Spec. heat ^(4,5) " " (7,8)	0.292 0.312	1.3-4.2 0.4-2	Spec. heat ^(4,5) " " (7,8)
As-received pyrolytic	~230Å	≤40°	≤3.44	Very high	2.03	0.4-2	Spec. heat ^(7,8)	0.0702	0.4-2	Spec. heat ^(7,8)
Lampblack-based (SA-25)	~100Å	Almost random	3.40	High	2.88	1.3-4.2 0.4-2	Spec. heat ^(4,5) " " (7,8)	0.0702	1.3-4.2 0.4-2	Spec. heat ^(4,5) " " (7,8)

The measurement temperatures and methods are listed after their respective elastic constant values.

characteristics presented in Table IV are:

1. Crystallite size as determined from the broadening of the (00 ℓ) X-ray lines,
2. Average crystallite basal plane tilt,
3. Layer spacing, and
4. Degree of stacking faults as qualitatively inferred from the resolution of the (h0 ℓ) lines.

If one considers the low-temperature specific heat results, the only vibrational modes which may be excited are those with low frequencies (approximately 10^{11} cps), where $\hbar\omega \leq kT$. Because of the weak van der Waals interlayer bonds, the only moduli which must be considered are c_{33} , which is related to longitudinal vibration of the planes along the c-axis, and c_{44} , which is related to a shearing type transverse vibration of the planes with respect to each other. The values shown in Table IV indicate that:

- (1) The c_{44} values determined from the specific heat are larger than those determined statically by factors of 1.4 to 41.
- (2) The c_{33} values determined from the specific heat values agree quite well with those determined statically,
- (3) c_{44} is very sensitive to the degree of stacking faults, or degree of turbostraticity, whereas c_{33} is only slightly affected, and
- (4) The crystallite size does not appear to be a major factor by itself.

The room temperature neutron diffraction results of Dolling and Brockhouse⁽¹⁰⁾ for a good thermally-annealed pyrolytic graphite gives $c_{44} = 0.42 \times 10^{11}$ dyne/cm², a value which agrees very well with the values of 0.452 and 0.405×10^{11} dyne/cm² obtained by Komatsu⁽⁸⁾ from the specific heat of Madagascar and Canadian natural graphites. The agreement is considered to be significant, because the neutron diffraction experiment looks directly at the lattice vibrational mode involved.

The values of c_{33} calculated from the specific heat data agree well with those values determined by entirely independent measurements such as

neutron diffraction, ultrasonic, sonic resonance, static compression, and compressibility (determined by direct X-ray measurements of the lattice spacing change) tests. The average value of c_{33} obtained from the data given in Table II, omitting the rough estimate of reference (6) and the values for those graphites with interlayer spacing not equal to 3.35 Å, is $3.61 \pm 0.13 \times 10^{11}$ dynes/cm². This value appears to be valid for both the single crystal and coke-base polycrystalline graphites.

The values of c_{44} calculated from the specific heat data, which are close to the value calculated from the neutron diffraction data, are higher than those obtained experimentally in our tests on single crystals and compression-annealed pyrolytic graphite. The most recent ultrasonic test data on single crystals reveal, however, that the shear modulus covers a wider range than had previously been observed. These recent results indicate that the shear modulus may increase as the degree of perfection of the crystal increases; thus, the specific heat value may represent an appropriate upper limiting value for the modulus. The values of the shear modulus obtained by mechanical testing are believed to be accurate: five different testing techniques yielded consistent results. One might speculate that a set of mechanisms is operative in the mechanical testing of the graphite which does not influence either the specific heat or neutron diffraction results. These mechanisms most likely involve dislocations and larger scale structural defects such as twins, kinks, tilt boundaries, etc. Unfortunately, no good model exists at the present time to explain the large range in the values of c_{44} .

6. STATIC TESTS ON PYROLYTIC GRAPHITE

E. J. Seldin

6.1 Torsion Test Apparatus

A new torsion apparatus, shown in Figure 14, has been constructed. It is similar, in principle, to the apparatus described in previous reports but has been automated to make collection of data more convenient. The specimen, which can be 1/4, 3/8, or 1/2 inch in diameter, is cemented with Eastman 910 cement between steel specimen holders. The upper specimen holder is fixed to the frame of the apparatus by a set screw, and the lower specimen holder fits on top of the rotating torsion shaft. The lower end of the torsion shaft is connected through a clutch to a gear box, which is, in turn, connected to a variable speed, reversible motor. In the center of the

Figure 14
Automated Torsion Apparatus

N-8599

torsion shaft is a torque transducer; with associated electronic equipment, the torque transducer produces a voltage proportional to the torque over the range ± 200 oz. in. Calibration of the torque transducer is achieved by fixing the upper end of the torsion shaft, disengaging the clutch, and applying dead weight loads through strings and pulleys to the 2-inch diameter wheel below the torque transducer. The rotation of the lower specimen holder is communicated to the core of a linear variable differential transformer (LVDT) by a bar attached to a ring around the lower specimen holder. The distance between the center of the specimen and the axis of the LVDT core is 4 inches. Since the rotation of the specimen is usually extremely small, the linear motion of the LVDT core is directly proportional to the angular twist of the specimen. The LVDT is calibrated by means of a micrometer screw. Two microswitches mounted at the rear of the apparatus act as safety devices; when activated by the bar which rotates with the lower specimen holder, the microswitch turns off the motor which drives the torsion shaft.

In a torsion test, the outputs of the LVDT and torque transducers are applied to the X and Y axes, respectively, of an X-Y recorder, and the curve of torque-vs.-angle of twist is recorded as the test is performed. Cyclic tests in which the torque is reversed are easy to perform. The compliance of the apparatus has been determined: it is a small, easily applied, correction factor.

7. ULTRASONIC TESTS ON PYROLYTIC GRAPHITE

O. L. Blakslee

Determination of c_{13} of Compression-Annealed Pyrolytic Graphite

The accuracy of the value for stiffness c_{13} of compression-annealed pyrolytic graphite is less than that of any of the other elastic constants; it has not yet been directly measured experimentally. We discuss here the feasibility of an experimental determination of c_{13} by ultrasonics. Garland and Silverman⁽¹¹⁾ have shown that acoustical wave velocities for propagation at an angle θ to the c-axis are given by:

$$\rho U_t^2 = c_{66} \sin^2 \theta + c_{44} \cos^2 \theta, \text{ and} \quad (1)$$

$$\begin{aligned} \rho U^2 = & c_{11} \sin^2 \theta + c_{33} \cos^2 \theta + c_{44} \\ & \pm \{ (c_{11} - c_{44})^2 \sin^4 \theta + (c_{33} - c_{44})^2 \cos^4 \theta \\ & + 2 \sin^2 \theta \cos^2 \theta [(c_{11} - c_{44})(c_{44} - c_{33}) + 2 (c_{13} + c_{44})^2] \}^{\frac{1}{2}}. \end{aligned} \quad (2)$$

The velocity of a pure transverse wave polarized perpendicular to the c-axis is denoted by U_t . The other two waves are of mixed character; the plus sign in Equation (2) gives a quasi-longitudinal wave velocity, U_{ql} , and the minus sign gives a quasi-transverse velocity, U_{qt} . Since Equation (2) contains c_{13} , calculations were made to determine the feasibility of determining c_{13} by measuring the velocity of a wave propagating at some angle θ to the c-axis with $\theta \neq 0$ or 90° .

Figure 15 shows the percent error in c_{13} per degree of error in θ as a function of θ for both the quasi-transverse and quasi-longitudinal waves. The plots were obtained by solving Equation (2) for c_{13} , differentiating with respect to θ , and calculating the derivative as a function of θ . It is impractical to use the quasi-longitudinal wave near $\theta = 0$, where the error is zero, because the wave is almost longitudinal. However, Figure 15 indicates that the quasi-transverse wave at $\theta = 13^\circ$ may possibly be used.

Calculations were performed to evaluate the error introduced in the determination of c_{13} from the velocity of the quasi-transverse wave at $\theta = 13^\circ$ by errors in each of the independent variables: c_{11} , c_{33} , c_{44} , θ , and $2\rho U_{qt}^2$. In these calculations, the following elastic constants were used:

$$c_{11} = 106 \pm 1 \times 10^{11} \text{ dynes/cm}^2,$$

$$c_{33} = 3.6 \pm 0.1,$$

$$c_{44} = 0.025 \pm .005, \text{ and}$$

$$c_{13} = 2.72 \times 2^{\pm 1}.$$

The average and extreme values were used, and it was determined that a one percent error in c_{44} causes negligible error in the calculated c_{13} , that a one percent error in θ causes -0.7 to -2.5 percent error in the calculated c_{13} , and that a one percent error in c_{11} causes 1.0 to 1.3 percent error in the calculated c_{13} .

Figure 15

Percent Error in c_{13} Per Degree Error in θ vs. θ

N-8721

The calculated value of c_{13} is much more sensitive to errors in c_{33} and $2\rho U_{qt}^2$, and the magnitude of the possible error in c_{13} strongly depends on the actual value of c_{13} (see Figure 16). Figure 16 indicates that, if c_{13} is less than approximately 2×10^{11} dynes/cm², there is little hope of determining it accurately from a measurement of the velocity of a quasi-transverse ultrasonic wave.

One additional complication is presented by the fact that the laminar structure and very weak interplanar bonds of compression-annealed pyrolytic graphite make the machining of a 13° face extremely difficult. Although the possibility exists of introducing the waves at the desired angle into the c-face of a specimen by means of a refraction technique, the effort required to develop this method can be more profitably expended in measuring the other stiffness constants. We conclude that no attempt should be made to determine c_{13} directly from experiment at the present time.

8. IRRADIATION TEST PROGRAM

The research program was reviewed in August, 1965, at the United States Atomic Energy Headquarters, Germantown, Maryland. The suggested irradiation schedule contained in the Research Proposal was amended and reduced to the schedule given in Table V. The irradiations will be performed by members of the Battelle Northwest Laboratories under the direction of Dr. H. H. Yoshikawa, Manager, Graphite Research and Development.

TABLE V
CPD Irradiation Program

A. Low Temperature

Irradiation Facility: Hanford Production Reactor, Cold Snout Facility
Irradiation Temperature: 40 to 60° ± 5°C
Nominal Exposures: 2, 5, 20, 50 MWD/AT
Number of Capsules: 4 plus 2 for experiment checkout
Dimensions of Capsule: Inside 2-3/4" diameter x 14-1/2" long
Start Irradiation: As soon as samples are ready

B. Intermediate Temperature

Irradiation Facility: Hanford Production Reactor, Hot Test Hole
Irradiation Temperature: Nominal 650°C
Nominal Exposures: 300, 650, and 1000 MWD/AT
Number of Capsules: 3
Dimensions of Capsule: Inside 4" x 4" x 24"
Start of Irradiation: As soon as samples are ready

C. High Temperature

Irradiation Facility: ETR or GETR
Irradiation Temperature: 1000°C ± 50°C (approx.)
Nominal Exposures: 800 (1/2 cycle), 1600-3200 (1-2 cycles) MWD/AT
Number of Capsules: 2
Dimensions of Capsules: Inside 3/4" diameter x 4" long per module;
4 to 5 modules
Start Irradiation: When samples are available

Figure 16
 Percent Error in c_{13} Due to a One Percent Error
 in Either ρU_{qt}^2 or c_{33}

N-8722

The preirradiation program is now completed. Specimens for nine capsules have been prepared and sent to Battelle Northwest Laboratories. Figure 17 shows the actual contents of a typical capsule. Table VI, which is a revision of a similar table given in a previous report,⁽²⁾ contains a list of the specimens; the total number of pyrolytic graphite specimens was reduced because less material was available than had been anticipated, but the number of single crystal specimens was increased.

The preirradiation test results are summarized in the following four tables. Table VII, listing the resonant bar specimens by capsule, contains the values of $1/s_{11}$ obtained in the sonic resonance tests and the values of c_{11} and c_{66} obtained by ultrasonic tests.* There are no ultrasonic data for the resonant bar specimens contained in the first two capsules. Table VIII lists the tensile specimens by capsule and the values of $1/s_{11}$ obtained in the static tensile tests. The tensile stress-strain curves were all linear and showed no hysteresis; the variation in values of the modulus is believed to be due to differences in the quality of the material from one specimen to the next, the modulus increasing as the quality of the material improves. Some of the values which were obtained are unaccountably high, higher than our previous estimate of the maximum possible modulus. Table IX lists the 1/2-inch diameter disks by capsule and the values of c_{33} and c_{44} obtained from ultrasonic tests.

Values of c_{44} were also obtained by means of the ultrasonic test method for many of the natural single crystal specimens which were included in the capsules for irradiation. Table X lists the results for the single crystals, and Figure 18 shows some typical cut single crystals. There are no results for the single crystals which were included in the first two capsules because the experimental method was developed only after the first two capsules were sent to Battelle Northwest. The application of this test to single crystals had not been anticipated at the start of the program; this test and the static pure shear tests for single crystals provide an additional amount of welcome and unexpected information.

* The values of c_{11} and c_{66} in Table VII are outside the range of values previously determined on whole disks.⁽¹⁾ The present values were obtained with 1 mc waves on thin bars. Preliminary measurements at 5 mc yield values of c_{11} approaching those previously reported for whole disks. The dependence of c_{11} and c_{66} upon size and frequency is being investigated.

Figure 17
Contents of Radiation Test Capsule

1 X

N-9188

Figure 18
Cut Single Crystal Disks

N-9649

TABLE VI
Samples for Each Irradiation Capsule

Sample Use	No. Per Capsule	Maximum Sample Dimensions, in.
Sonic Resonant Bar Test	2	1/4 x 1/4 x 2-5/8
Ultrasonic, Sonic (CTO), Torsion Test	4	1/2 diam. x 5/16
Tensile Test	3	5/16 x 5/8 x 2-1/4
Compression Test	3	1/4 diam. x 5/16
Shear 60° (Static) Test	1	1/4 x 1/4 x 3/4
Shear 45° (Static) Test	1	1/4 x 1/4 x 3/4
Shear 30° (Static) Test	1	1/4 x 1/4 x 3/4
Shear 15° (Static) Test	1	1/4 x 1/4 x 3/4
Single Crystals	2 (minimum)	3/4 diam. x 1*

* Dimensions refer to size of capsule containing single crystals.

TABLE VII
Resonant Bar Test Results, Sonic ($1/s_{11}$) and Ultrasonic (c_{11} and c_{66})

Capsule No.	Specimen	c_{33} (10^{11} dynes/cm ²)	c_{44} (10^{11} dynes/cm ²)	Capsule No.	Specimen	c_{33} (10^{11} dynes/cm ²)	c_{44} (10^{11} dynes/cm ²)
1	M308-S1	3.27	.0260	6	M318-S1	3.56	.0241
	M323-S1A	3.34	.0217		M321-S3	3.47	.0256
	M323-S2	3.52	.0220		M321-S5A	3.56	.0269
	M325-S3	3.45	.0246		M324-S3B	3.60	.0214
2	M323-S1B	3.39	.0214	7	M318-S3B	3.63	.0257
	M323-S3	3.48	.0213		M318-S4A	3.53	.0247
	M325-S1	3.46	.0249		M321-S5B	3.50	.0268
	M325-S2	3.48	.0246		M324-S5	3.32	.0234
3	M310-S2	3.53	.0303	8	M313-S1	3.50	.0262
	M321-S1	3.49	.0258		M318-S3A	3.54	.0247
	M324-S1A	3.65	.0210		M318-S5B	3.55	.0248
	M324-S3A	3.55	.0214		M324-S4B	3.62	.0233
4	M310-S1	3.51	.0294	9	M313-S2	3.41	.0265
	M321-S4	3.49	.0262		M316-S1	3.50	.0274
	M324-S1B	3.63	.0207		M318-S2	3.57	.0235
	M324-S4A	3.45	.0241		M318-S5A	3.59	.0236
5	M310-S4	3.59	.0290				
	M318-S4B	3.47	.0252				
	M321-S2	3.47	.0260				
	M324-S2A	3.47	.0210				

TABLE VIII
Tensile Test Results

Capsule No.	Specimen	$1/s_{11}$ (10^{11} dynes/cm ²)	Capsule No.	Specimen	$1/s_{11}$ (10^{11} dynes/cm ²)
1	M313-2-2	84.0	6	M314-T2-2	95.0
	M323-1	81.8		M316-T2-2	88.7
	M325-1	72.8		M321-T1-2	90.1
2	M306-T1	84.5	7	M306-T4-3	110.7
	M310-T1	83.0		M316-T2-3	96.1
	M325-2	104.1		M323-T1-1	89.8
3	M306-T3-1	88.3	8	M316-T2-4	92.2
	M313-1-2	95.5		M317-T1-3	90.7
	M316-T1-2	96.1		M321-T1-1	87.9
4	M306-T2	83.0	9	M314-T2-1	93.7
	M314-T1-1	95.9		M316-T2-5	88.1
	M317-T2-1	80.0		M317-T2-2	82.8
5	M314-T1-2	102.7			
	M316-T1-3	86.5			
	M317-T1-2	93.3			

TABLE IX
Ultrasonic Test Results on 1/2-Inch Diameter Disks

Capsule No.	Specimen	c_{33} (10^{11} dynes/cm ²)	c_{44} (10^{11} dynes/cm ²)	Capsule No.	Specimen	c_{33} (10^{11} dynes/cm ²)	c_{44} (10^{11} dynes/cm ²)
1	M308-S1	3.27	0.0260	6	M318-S1	3.56	.0241
	M323-S1A	3.34	.0217		M321-S3	3.47	.0256
	M323-S2	3.52	.0220		M321-S5A	3.56	.0269
	M325-S3	3.45	.0246		M324-S3B	3.60	.0214
2	M323-S1B	3.39	.0214	7	M318-S3B	3.63	.0257
	M323-S3	3.48	.0213		M318-S4A	3.53	.0247
	M325-S1	3.46	.0249		M321-S5B	3.50	.0268
	M325-S2	3.48	.0246		M324-S5	3.32	.0234
3	M310-S2	3.53	.0303	8	M313-S1	3.50	.0262
	M321-S1	3.49	.0258		M318-S3A	3.54	.0247
	M324-S1A	3.65	.0210		M318-S5B	3.55	.0248
	M324-S3A	3.55	.0214		M324-S4B	3.62	.0233
4	M310-S1	3.51	.0294	9	M313-S2	3.41	.0265
	M321-S4	3.49	.0262		M316-S1	3.50	.0274
	M324-S1B	3.63	.0207		M318-S2	3.57	.0235
	M324-S4A	3.45	.0241		M318-S5A	3.59	.0236
5	M310-S4	3.59	.0290				
	M318-S4B	3.47	.0252				
	M321-S2	3.47	.0260				
	M324-S2A	3.47	.0210				

TABLE X
Graphite Single Crystals Prepared for Irradiation Tests

Capsule No.	Capsule Contents	Shear Modulus in Units of 10^{11} dynes/cm ² - c_{44} (Ultrasonic) Before Irradiation
1	2 - uncut 1 - cut	
2	2 - uncut 1 - cut	
3	EPC-9 - cut EPC-10 - cut	.061 ₃ .051 ₁
4	EPC-11 - cut EPC-12 - cut	.028 ₀ .066 ₉
5	EPC-13 - cut EPC-14 - cut 5 uncut crystals	.059 ₀ .089 ₀
6	EPC-15 - cut EPC-16 - cut 5 uncut crystals	.094 ₆ .046 ₀
7	EPC-18 - cut EPC-19 - cut 5 uncut crystals	.056 ₂ .134
8	EPC-20 - cut EPC-21 - cut 5 uncut crystals	.079 ₀ .100
9	EPC-22 - cut EPC-23 - cut 5 uncut crystals	.166 .062 ₃

9. POSTIRRADIATION PLANS

The irradiated samples will be returned from Battelle Northwest Laboratories to the Parma Technical Center for postirradiation measurements. The same schedule of measurements employed during the preirradiation evaluation will be followed so that pre- and postirradiation measurements may be directly compared. In addition, postirradiation tests will include tests for compression, static torsion, shear, and tests employing the compound torsion oscillator (CTO).

We expect that the shear modulus c_{44} will be affected more than any other elastic constant by the radiation. If c_{44} changes drastically, either of the following problems may arise: (1) if c_{44} increases greatly with irradiation, some of the thinnest samples (for example, the single crystals) may be difficult to measure with the same degree of accuracy as in the preirradiation evaluation; (2) if the value of c_{44} decreases greatly with irradiation, greater care may be needed in handling the specimens during the postirradiation measurements. The measurement of the other elastic constants should be no more difficult after the irradiation than before.

kim

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Battelle Memorial Institute
Pacific Northwest Laboratory
Attention: R. Nightingale
Post Office Box 100
Richland, Washington

Los Alamos Scientific Laboratory (2)
Attention: R. D. Baker, R. D. Fowler
Post Office Box 1663
Los Alamos, New Mexico 87544

General Atomic
Division of General Dynamics Corporation
Attention: W. Goddell
Post Office Box 608
San Diego, California 92112

Library (T.D.S.)
Jet Propulsion Laboratory
California Institute of Technology
Attention: N. E. Devereux, Library Supervisor
4800 Oak Grove Drive
Pasadena, California 91103

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
United States Air Force
Attention: R. G. Spain - MANF
Wright-Patterson AFB, Ohio 45433

Air Force Materials Laboratory
Air Force Systems Command
United States Air Force
Attention: S. Bradstreet - ASRCE-32
Wright-Patterson AFB, Ohio 45433

Union Carbide Corporation