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

UNITED STATES ATOMIC ENERGY COMMISSION
CONTRACT NO. AT-(40-1)-3237
QUARTERLY PROGRESS REPORT NO. 2
JULY 1, 1964 TO SEPTEMBER 30, 1964

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

July 1, 1964 to September 30, 1964

Principal Investigator

J. T. Meers

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October, 1964

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, 1964 to September 30, 1964.

The Project is under the general supervision of J. T. Meers. The graphite single crystal work has been carried out by R. Sprague and R. Bacon. The pyrolytic graphite work has been carried out by O. L. Blakslee, D. G. Proctor, E. J. Seldin, G. B. Spence, and G. Wagoner. The X-ray analysis was carried out by C. E. Lowell. This report was prepared by J. T. Meers with the assistance of the participants in the program.

ABSTRACT

Small single crystals of graphite have been grown in an iron-carbon melt at temperatures near 2700° C. Work has been continued on the production and evaluation of compression annealed pyrolytic graphite. The average orientation of crystallites and some of the structural characteristics of these materials have been determined by X-ray, optical, and electron microscope techniques. Preliminary values of Young's modulus, $1/s_{11}$, and the basal plane shear strength for compression annealed pyrolytic graphite are reported. Consistent results have been obtained for the shear modulus, c_{44} , by both sonic and ultrasonic methods.

The experimental equipment (the single crystal tensile testing apparatus, the simple shear wedges, and the resonant bar apparatus) is described.

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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.

A major part of the pre-irradiation program is the production of and characterization of compression annealed pyrolytic graphite and artificial graphite crystals. Work has been concentrated upon these areas; as materials are produced which approach the structural perfection of the graphite single crystal, the problems involved with making measurements are increased. A considerable effort is being spent on developing techniques of sample preparation and methods of measurement which will be useful in determining the high in-plane modulus and tensile strength and the low between-plane modulus and shear strength of compression annealed pyrolytic graphite.

Where possible, the elastic constants have been cross checked by comparing the results of two or more different measuring methods. This cross checking lends confidence to the validity of property values which are being determined for these materials, since standard values of elastic constants have not yet been established.

2. SUMMARY OF RESULTS

Work for this quarter has been concentrated on the production and evaluation of the mechanical properties of graphite single crystals and compression annealed pyrolytic graphite. Good progress has been made, even though the evaluation of single crystals awaits construction of apparatus.

Very thin single crystals of graphite up to 6 mm in diameter have been

grown in a carbon supersaturated iron melt. The best crystals were formed by melting pure iron in a graphite boat at 2700° C: the melt becomes saturated with carbon, the iron then slowly evaporates, and the graphite crystals grow in the supersaturated melt.

The pyrolytic graphite used in this work has been compression annealed by applying a compressive stress perpendicular to the average basal plane while the sample is heated to 2800° C. During this quarter, attempts have been made to anneal fine grain, substrate nucleated material made from methane. These runs produced materials good enough for initial measurements, but side slippage was noted in each case. Additional annealing experiments have shown that pyrolytic graphite specially made from propane had a still greater tendency to side-slip during annealing. The conclusion reached from these experiments is that highly columnar, essentially soot-free pyrolytic graphite requires more refined annealing procedures than ordinary commercial pyrolytic graphite. Optical, X-ray, and electron microscope techniques have been used to evaluate the graphites. Optical and X-ray methods were used to determine the half widths of the crystallite distribution at or near the surface of a sample. An interferometric method of studying the surface of pyrolytic graphite samples has been devised. The interference fringes form contour maps of the surface, indicating the effects of inclusions. An electron microscope study of cathodically etched, replicated c- and a-faces of both single crystals and pyrolytic graphite reveal multiple cracks in the a-surface and a continuum of etch pits on the c-surface. In some instances, a row of etch pits has been identified as the base line of a cleavage step on the crystal surface.

The final design of an apparatus for measuring tensile stress-strain curves for single crystals of graphite has been completed. The construction should be completed by the end of the third quarter.

Tensile tests have been made on specimens of compression annealed pyrolytic graphite mounted in epoxy end pieces. Because there are consistent differences in Young's modulus as measured by strain gages on the c-faces compared with values obtained from strain gages on the a-faces, the values for Young's modulus quoted in the text are to be taken as preliminary. In other static tests, the basal plane shear strength of pyrolytic graphite has been measured using simple wedge-shaped sample holders. Shear strengths of 129 psi and 152 psi were obtained for bright compression annealed graphite and wavy dull compression annealed graphite, respectively.

The resonant bar apparatus has been improved so that a minimum of manual manipulation is necessary when searching for resonances.

The exact frequency equations for the compound torsion oscillator have been derived, and the results have been applied respectively to steel-aluminum-steel and steel-graphite-steel oscillators for both as-deposited and compression annealed pyrolytic graphite. For aluminum, the compound torsion oscillator value was 3 per cent below that obtained by the resonant bar test; for graphite (much lower modulus), the value obtained by the compound oscillator technique was within 1/2 per cent of the value obtained by ultrasonic measurements.

3. MATERIALS PREPARATION

The work during this quarter has been directed toward the understanding and control of two processes: (1) the growth of graphite single crystals and (2) the compression annealing of pyrolytic graphite. In the former case, control of crystal nucleation has been of primary concern. For pyrolytic samples, work to relate the as-deposited structure to the final annealed structure has been continued, but the main emphasis has been placed upon the control of temperature, pressure, and the uniaxial alignment of the load train.

3.1. Growth of Graphite Single Crystals

G. Wagoner

In the first quarterly report,⁽¹⁾ it was pointed out that efforts to grow single crystals from a solution of carbon in molten iron were hampered by too much nucleation. Experiments designed to reduce the nucleation by reducing the rate at which iron is evaporated from the iron-carbon melt are in progress and have resulted in the production of crystals up to 6 mm across, which, to our knowledge, are the largest synthetic graphite crystals which have been grown.

The best crystals have been produced by the following method. Pure iron in a graphite crucible is heated in a tube furnace to approximately 2700° C. The iron melt becomes saturated with the dissolving graphite; then, the iron slowly evaporates, leaving a supersaturated solution in which the graphite

crystals grow. By lowering the temperature, the vapor pressure of iron over the melt is reduced and the rate of crystal growth (and nucleation) is decreased. The degree of supersaturation required to produce crystal growth is less than that required to nucleate new crystals; thus, lower evaporation rates produce larger crystals. Hold-temperatures below about 2650° C., however, require impractically long furnace runs. Several runs at 2675° C. produced fairly good crystals.

The method of increasing crystal size by slowing the evaporation has the disadvantage of remaining a haphazard process: the crystals produced in such a homogeneous manner nucleate at random positions and the entire process is unsatisfactory. As yet, no satisfactory seeding or crystal pulling operation has been devised.

3.2 Compression Annealed Pyrolytic Graphite

G. B. Spence

The pyrolytic graphite used in this work has been compression annealed by applying a compressive stress perpendicular to the average basal plane while the sample is heated to a temperature above 2800° C. For reasons of economy, the original compression annealing was done on commercial quality pyrolytic graphite. During the third month of the contract period, attempts were made to compression anneal some fine grain, substrate nucleated material specially made from methane by High Temperature Materials, Inc. Although all but one of these runs produced material sufficiently good for the initial measurement program, some side slippage was noted in each case. Subsequent work has shown that the pyrolytic graphite specially made from propane has a still greater tendency to slip during compression annealing. In order to obtain high structural perfection by compression annealing the fine grain,

substrate nucleated pyrolytic graphite, more refined annealing procedures must be found than were necessary for commercial quality material.

Work at the Advanced Materials Laboratory has been concerned with devising methods of compression annealing pyrolytic graphite without a prior thermal anneal and on measuring techniques for more accurately determining the sample temperature during thermal and compression anneals. The first compression anneals were made on material that had not been given a prior thermal anneal, and the large basal plane expansion broke the mold. The pyrolytic graphite was then thermally annealed prior to compression annealing, since part of the basal plane expansion occurs during the thermal anneal, thereby reducing the amount which occurs during the compression anneal. However, in pressing only one plate at a time, as is now being done with the special methane and propane material, there is no problem of mold breakage; and it is possible to eliminate the prior thermal anneal which might degrade the structural quality. In special thermal and compression anneals, holes were bored through the susceptor or mold in order to measure the temperature of the pyrolytic graphite as well as the temperature of the outside surface of the susceptor or mold. In the temperature range of interest, the pyrolytic graphite was about 150° C. hotter than the outside of the susceptor or mold. This difference of 150° C. should be added to previously reported susceptor and mold temperatures to obtain the sample temperature.

The compression annealing apparatus at the Research Laboratory has been modified by enlarging the diameter of the load train from 1-1/2 to 3 inches. The larger size facilitates an accurate alignment of the load train. Several compression anneals of 1 inch diameter disks were made at widely different conditions of time, temperature, and pressure. So far, no combination has produced ideal material. It is felt that the resistance heating method used at the

Research Laboratory is probably not capable of producing results as good as the induction heating method used at the Advanced Materials Laboratory.

4. MATERIAL CHARACTERIZATION

In Quarterly Progress Report No. 1⁽¹⁾, work was described in which the preferred orientation and surface condition of compression annealed pyrolytic graphite was examined by optical and X-ray techniques. Improvements of and additions to these methods include: a modification of the X-ray technique which reduces the instrumental broadening; an interferometric method of examining surface roughness; and an electron microscope examination of the c- and a-faces of single crystal and compression annealed pyrolytic graphite.

All these techniques can now be applied directly to properly prepared graphite surfaces, and the results of these surface examinations will be correlated with the observed mechanical properties.

4.1. X-Ray Evaluation of Preferred Orientation in Pyrolytic Graphite

C. E. Lowell

The standard method of measuring preferred orientation is plagued by instrumental broadening. By changing the sample geometry and axis of rotation as shown in Figure 1, a significant reduction in the instrumental broadening has been achieved. The new specimens are 0.125 inch diameter rods, one inch long, which are cut with the rod axis parallel to the plane of deposition; they are rotated about this axis. Fabrication problems made necessary the use of square cross section rods in the case of the compression annealed pyrolytic graphite. Experimental evaluation of corrections for this geometry showed the effect to be negligible over the small angular range of the crystallite distributions in these samples.

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FIGURE 1. Method for Measuring Surface Orientation by X-Rays

The results of this work are shown in Table I. The MgO single crystal results show a thirtyfold decrease in experimental broadening over the disk method. There is, however, one note of caution to be observed: the calculated depth at which the reflected intensity of the diffracted X-ray beam falls to one per cent of the surface layer is 0.031 inch, indicating that this value is essentially a surface measurement. Further evidence that the measurement is surface is shown by the difference in the peak half-width at half maximum of the 002 line for the two sides of the sample and also by the fact that the peak positions are not 180° apart. These differences account for part of the discrepancy between the disk and rod technique which cannot be accounted for solely by instrument broadening.

TABLE I.

ORIENTATION OF MgO AND PYROLYTIC GRAPHITE AS MEASURED
BY X-RAYS

Material	1/2 Width at 1/2 Max. ⁽¹⁾		Maximum Position	
	Side 1	Side 2	Side 1	Side 2
MgO	0.05°	0.05°	0.00°	180.00°
Hot Pressed Commercial	0.56°	0.60°	0.00°	181.15°
Hot Pressed Methane	1.68°	1.95°	0.00°	183.94°

(1) Precision is estimated to be $\pm 0.015^\circ$.

4.2. Interferometric Method of Evaluating Cleaved Graphite Surfaces

G. Wagoner

Highly oriented graphites, such as single crystals and compression annealed pyrolytic graphites, are very easy to cleave. Visual examination of the resulting surfaces often indicates the degree of preferred orientation, the presence of twins, growth cones, and other structural characteristics; and it is desirable to substantiate the qualitative impression one gets by such visual or microscopic examination by more quantitative evidence. An optical reflectivity method was discussed in the first quarterly report⁽¹⁾; here, preliminary observations of interference patterns with the specimen forming one mirror of an interferometer are presented.

The 5461 Å line from a mercury lamp is used to set up the interference pattern between the sample and an optically flat mirror. The interference pattern can be magnified with a microscope and either scanned visually or photographed to obtain a topographical map of the surface. The vertical displacement between fringes is one-half wavelength (2730 Å). Thus, by counting the number of fringes per unit length of the sample surface, the angle between the optical flat and that part of the sample surface can be measured. The smoothness of the surface is also indicated by the character of the fringes, a smooth surface

giving dark continuous fringes and a rough one showing discontinuous crooked fringes. Figures 2. and 3. show the patterns of two compression annealed pyrolytic graphite samples which appeared quite similar to the unaided eye. The pattern of Figure 3. was produced by a sample of pyrolytic graphite made from propane and compression annealed at 3600° C. It shows smooth contours, indicating no appreciable roughness greater than approximately one-quarter wavelength in height. The angle between the "top of the hill" and the steepest slope is 0.9°.

Figure 2. , when contrasted with Figure 3. , shows a much less well defined set of fringes. Undulations of the surface produce the fringe waviness. Most of the bumps are between 1500 Å and 3000 Å in height, as shown by a change from light to dark in the interference pattern. The distance across the bumps is about 0.01 mm. Thus, the bumps are about 100 times as wide as they are high.

Evidently, the interference technique of evaluating pyrolytic graphite surfaces is highly useful. Surface waviness, the variations in angle of the surface, cleavage steps, and other surface imperfections are clearly visible and quantitatively measurable. The resolution of height variations is a few thousand angstroms, which is somewhat better than the best resolution of a good optical microscope.

4.3. Electron Microscopy

R. Bacon, A. Silvaggi

Surface replicas suitable for electron microscopy have been obtained from both a-faces and c-faces of unetched and cathodically etched pyrolytic and single crystal graphite. Initially, a cellulose acetate replica is made using the Faxfilm technique. This replica is then shadowed at 45° with a

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FIGURE 2. Surface Undulations of Compression Annealed Pyrolytic Graphite Containing Lampblack Inclusions

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FIGURE 3. Surface Smoothness of a Compression Annealed Pyrolytic Graphite Deposited from Propane

platinum-carbon pellet, and carbon is evaporated onto the replica to make a carbon film replica containing the platinum "shadows". The last step is to dissolve the cellulose acetate.

The a-face of compression annealed unetched pyrolytic graphite sample DD-65 is shown in Figure 4. No detail of the structure is detectable; the line is a scratch on the sample surface.

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Figure 4. A-Face of Compression Annealed Unetched Pyrolytic Graphite

The a-face of compression annealed etched pyrolytic graphite sample C-101 is shown in Figure 5.

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FIGURE 5. A-Face of Compression Annealed Etched Pyrolytic Graphite, Electronic Micrograph 5200 X

The etch has revealed a series of parallel lines upon which occasional etch pits are located. It is not possible to determine if these lines are cracks or if they represent disordered planes which etch preferentially.

The replicated a-face of an unpurified single crystal is shown in Figure 6. Although this crystal was of good quality, the cathodic etch brings out a structure which appears to be microcracks over the entire sample. There are many circular etch pits located in or along the cracks: the average etch pit diameter is about $800 \text{ \AA}$; and the minimum distance between cracks is about $400 \text{ \AA}$, which is even closer than that found in compression annealed pyrolytic graphite.

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FIGURE 6. A-Face of an Unpurified Etched Single Crystal Graphite,
Electron Micrograph 7300 X

The c-face of compression annealed pyrolytic graphite sample C-4 as determined by an electron micrograph is shown in Figure 7. It should be compared with the optical micrograph of the same sample, Figure 8, which was shown in Quarterly Progress Report No. 1,⁽¹⁾ page 18 (erroneously labelled C-5 in that report). The heavy lines seen in the optical micrograph appear in the electron micrograph as well-defined rows of etch pits. The heavier rows appear to cross over or dominate the lighter rows, but no consistent pattern can be observed. The entire background is covered with etch pits, some apparently random, others showing short-range order.

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FIGURE 7. C - Face of Compression Annealed Etched Pyrolytic Graphite
Sample No. C-4, Electron Micrograph 5200 X

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FIGURE 8. C-Face of Compression Annealed Etched Pyrolytic Graphite
Sample No. C-4, Optical Micrograph 1000 X, Oblique Lighting

Figure 9 shows the c-face of another and similar sample of compression annealed pyrolytic graphite.

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FIGURE 9. C-Face of Compression Annealed Etched Pyrolytic Graphite
Sample No. C-5, Electron Micrograph 7300 X

Figure 10 is the c-face of an unpurified single crystal. The appearance of this etched surface is very similar to that of the pyrolytic graphite c-faces. Inspection of the crystal by optical microscopy permitted the identification of some large cleavage steps and well defined rows of etch pits frequently occurring at the base of such steps (marked "AA" in Figure 10). Since it is expected that many smaller cleavage steps, essentially unobservable by normal microscopy methods, must occur in all samples, it is a good assump-

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FIGURE 10. C-Face of an Unpurified Etched Single Crystal Graphite,
Electron Micrograph 5200 X

tion that many of the etch pit rows are caused by cleavage steps. However, it is difficult to interpret the complicated patterns of intersecting and crossing lines entirely on this basis.

5. STATIC TESTS ON SINGLE CRYSTALS

R. Bacon and R. Sprague

5.1. Preparation of Tensile Specimens

All attempts to cut out dog-bone shaped tensile specimens from a cleaved crystal or a thin sheet of pyrolytic graphite have resulted in a peening-over of the edges when an air abrasive cutting technique was used. A variety of substrates (copper sheet, wax, and bare glass slide) and a

variety of masks (cellulose acetate, steel, and glass) have been used.

Attempts to remove the peened-over edges by etching the sample in a gas-oxygen flame have not yet proven satisfactory, but further development of such techniques may result in an acceptable preparation procedure.

5.2. Tensile Test Apparatus

An apparatus for stress-strain tensile testing of single crystals of graphite has been designed, and final drawings have been nearly completed. The force and displacement transducers and associated electronic equipment have been purchased.

The machine itself is compact enough to be contained inside a vacuum bell jar. A line drawing of the apparatus is given in Figure 11 ; its basic components consist of:

- a. A tension load cell of the resistance strain gage type,
- b. An upper specimen grip attached rigidly to the tension element of the load cell,
- c. A lower grip attached rigidly to the crosshead,
- d. A crosshead (motion guided vertically), and
- e. A displacement transducer of the LVDT-type (linear variable differential transformer) to measure directly the motion of the crosshead.

The output from the load cell, measuring the tension in the specimen, will be recorded on the y-axis of an X-Y recorder. The output from the displacement transducer will go to the x-axis. This output (measuring the crosshead displacement) will represent the specimen elongation, except for small corrections due to machine (largely load-cell) compliance.

In order to assure perfect alignment of the machine, the load cell is mounted so that it can be translated and rotated (about any horizontal axis)

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FIGURE 11. Tensile Test Apparatus for Single Crystal Graphite

until the flat surface ground faces of the two specimen grips are in perfect alignment.

In order to permit mounting of the specimen onto the two sample grips, the latter will be removed from the testing machine to the laboratory bench and fixed in proper relative position in a special jig. The thin specimen can then be cemented on the two grip faces; the assembly will then be inserted into the machine and the jig removed, leaving the delicate specimen as the only connection between the two grips.

Because the rigidity or stability of the upper grip assembly is limited by a quarter-inch threaded connection into the load cell, there is a possibility of some unavoidable bending of the upper grip assembly relative to the lower grip during removal of the specimen mounting jig. Since the slightest motion would damage the specimen, provision has been made for the upper grip assembly to be held in place by a keeper which can slide up and down on the crosshead ways. The keeper will be moved down free of the upper grip assembly just prior to testing, so that only the specimen itself can exert a force on the load cell.

6. STATIC TESTS ON PYROLYTIC GRAPHITE

E. J. Seldin

Tensile tests have been made on specimens of compression annealed pyrolytic graphite mounted in epoxy end pieces. The testing was performed on an Instron machine, using the load cell on the Instron to detect the stress; pairs of strain gages on the specimen measure the longitudinal and transverse strains. Experimental values of the compliances s_{11} , s_{12} , and s_{13} have been obtained as a result of measurements on several specimens. Considerably higher values of $1/s_{11}$ were obtained in tests where the strain gages were

attached to the a-faces of the specimen than when the strain gages were attached to the c-faces. Several measurements have been made of compliance s_{12} ; only one measurement has been made of s_{13} . These initial results indicate that there may be some variation in the properties of individual specimens and that further refinement of the experimental techniques may be necessary for the study of this highly anisotropic material.

Shear tests were performed on thin specimens of compression annealed pyrolytic graphite which were mounted in small wedge-shaped fixtures. The fixtures were compressed on the Instron machine so that the specimens were subjected to a combination of normal and shear stresses. Sets of shear fixtures with four different angles were used in order to obtain the shear strength as a function of normal compressive stress. The shear strength at zero normal stress was obtained from an extrapolation of the data on a plot of shear stress at failure versus normal stress.

6.1. Tensile Tests on Compression Annealed Pyrolytic Graphite

Figure 12 is a drawing of a compression annealed pyrolytic graphite tensile specimen with epoxy end pieces. The graphite specimen was typically $2 \frac{3}{4}$ inches long and $\frac{1}{2}$ inch wide at the ends, with a gage section $1 \frac{1}{2}$ inches long and 0.250 inch wide. The radius of curvature at the end of the gage section was $\frac{1}{4}$ inch for some of the specimens and $\frac{1}{2}$ inch for others. The gage section was formed on a milling machine for a large thickness of the material, and the material was then cleaved to its final thickness. The broad face of the specimen shown in the lower part of the drawing is the c-face, and the narrow face of the specimen shown in the upper part of the drawing is the a-face. The epoxy end pieces were potted in a special molding fixture which held the vertical axis of the specimen centrally aligned with the molds for the end pieces. The epoxy was

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FIGURE 12. Graphite Specimen with Potted Epoxy End Pieces for Static Tensile Testing

BAKELITE ERL-2795 resin with ERL-2793 hardener. One end piece was poured and allowed to polymerize; this end was then released from the mold, replaced in the mold, and the second end piece poured. After the entire tensile specimen was released from the molding fixture, the specimen was held at 100° C. for at least one hour in order to cure the epoxy end pieces. The holes were then drilled on each end; in this operation, the specimen was held in a drilling jig which allowed the holes to be placed exactly on the center line of the specimen.

The strain gages which were used were paper-backed foil gages manufactured by the Baldwin-Lima-Hamilton Corp.; they were applied with either Duco cement or SR-4 cement. The gages were accurately positioned on the

specimen with the gage axis parallel to the longitudinal axis of the specimen by holding the specimen on a special fixture and positioning the fiducial marks on the gages under the cross-hairs of a microscope. Pairs of gages on opposite faces of the specimen were used for the measurement of the longitudinal strain and the transverse strain. Each pair of gages formed the active elements of a full strain gage bridge. A Sanborn amplifier-indicator provided excitation for each strain gage bridge, amplified the output signal of the bridge, and provided a direct voltage output proportional to the strain which was fed to an X-Y recorder. The stress was detected by the load cell of the Instron machine. The longitudinal and transverse stress-strain curves were plotted simultaneously on two X-Y recorders while the specimen was being stressed. The elastic constants were then determined from the slopes of the recorder plots.

6.2. Shear Strength Tests on Compression Annealed Pyrolytic Graphite

The shear tests were performed with fixtures of the type shown in Figure 13. Four sets of these fixtures were made, with the angle θ equal to 15° , 30° , 45° , and 60° , respectively. The specimens were thin slabs of compression annealed pyrolytic graphite which were cemented to the shear faces of the fixtures with a sealing cement. After the specimen was inserted between a pair of the fixtures, the edges of the specimen were sanded with emery cloth to reduce the area of the specimen to that of the shear faces.

A pair of fixtures containing the specimen was compressed on the Instron machine, and the Instron recorder gave a plot of the force as a function of the crosshead motion. Figure 14. shows some typical recorder traces. The deformation of the sample was small compared with the deformation of the load train and load cell; therefore, the slope of the curve is not the

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FIGURE 13. Stainless Steel Shear Fixture

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FIGURE 14. Typical Recorder Chart Showing Shear of Compression Annealed Pyrolytic Graphite

shear modulus of the specimen. There is a region at the end of the curves which indicates that slip was occurring. The shear strength was determined in all cases from the value of force at the proportional limit as shown in Figure 14. The shape of the top of the curve varied from test to test because the specimen cleaved more cleanly in some tests than in others.

The shear stress and the normal stress were computed for each test. Assuming F to be the force applied by the Instron machine at the proportional limit, $F_n = F \cos \theta$ is the force normal to the specimen and $F_s = F \sin \theta$ is the shear force on the specimen. The area of the specimen is $A = WD/\cos \theta$, where W and D are the dimensions of the base of the fixture as shown in Figure 13. The normal stress is

$$\sigma_n = F_n / A = F \cos^2 \theta / WD,$$

and the shear strength is

$$\sigma_s = F_s / A = F \sin \theta \cos \theta / WD.$$

Starting with a specimen approximately 0.04 in. thick, it was possible to run a dozen or more shear tests as the specimen was successively cleaved into thinner and thinner pieces. The cleaved surface was cleaned after each test and a single new fixture cemented to the specimen in preparation for the next test. In this manner, the specimen thickness was gradually reduced with successive tests, and specimens as thin as 0.002 in. were cleaved successfully. There was no apparent variation in the shear strength with specimen thickness; the experimentally obtained values of shear strength showed a random scatter from test to test.

6.3. Results of Tensile and Shear Tests

Tensile Tests

The results of static tensile tests on twelve specimens of compression annealed pyrolytic graphite are summarized in Table II. Some of the speci-

TABLE II.

PRELIMINARY RESULTS OF STATIC TENSILE TESTS ON COMPRESSION
ANNEALED PYROLYTIC GRAPHITE

Specimen	Thickness (inch)	Test No.	Young's Modulus*		Compliances (10^{-12} cm ² /dyne)		
			$1/s_{11}$		s_{11}	s_{12}	s_{13}
			(10^8 lb/in. ²)				
C5-4	.087	1	82	c	.177		
C5-5	.113	1	120	c	.121		
C5-7	.075	1	83	c	.175		
C5-8	.101	1	89	c	.163		
C5-10	.062	1	112	c	.130		
C5-11	.035	1	102	c	.142	-.019	
C5-12	.073	1	103	c	.141	-.024	
		2	104	c	.139	-.027	
		3	105	c	.138	-.031	
C5-13	.088	1	85	c	.171	-.032	
C5-14	.121	1	168	a	.086		
		2	158	a	.092		
		3	163	a	.089		
			81	c	.179		
C5-15	.109	1	84	c	.173	-.028	
		2	85	c	.171	-.029	
M 103-321	.141	1	82	c	.177	-.025	
		2	77	c	.188	-.034	
		3	80	c	.181	-.030	
M 103-322	.166	1	175	a	.083		-.056
		2	142	a	.102		-.054
		3	141	a	.103		
		4	118	a	.123		
			84	c	.173		
		5	120	a	.121		
			114	c	.126		

* Letter denotes faces of specimen on which gages were mounted.

mens were tested only once and others tested two or more times; in all cases of multiple tests shown in Table II, the tests were run on separate days. The stress-strain curves which were obtained from the average of pairs of strain gages were linear or very nearly linear for both the longitudinal and transverse strains, and the values of Young's modulus and the compliances given in Table II are based on the average slopes of the curves.

Most of the measurements of the longitudinal strain were made by strain gages which were cemented to the c-faces of the specimen after giving these faces a light sandblasting. The values of Young's modulus which were obtained from these tests ranged from 77 to 175×10^6 lb/in.². On two of the thicker specimens, strain gages were mounted on the a-faces for the measurement of longitudinal strain, and these measurements yielded considerably higher values for the Young's modulus.

The Young's modulus results are puzzling. From the tests in which the gages were mounted on the c-faces of the specimen, there is a spread of values which indicates that the properties of the individual specimens may not be identical. From the results of the two tests in which the gages were mounted on the a-faces of the specimens, one is led to believe that the stress distribution within the specimens was not uniform. In specimen C5-14, the strain measured on the a-face was approximately half that measured on the c-face. In specimen M 103-322, the strain was also lower on the a-face than on the c-face, but the modulus measurement varied with successive tests until it appeared to be approximately the same on both faces of the specimen. This variation with successive tests was found only with specimen M 103-322; multiple tests on four of the other specimens yielded consistent results from test to test.

A possible explanation of the difference between the strains measured on the a-faces and c-faces of a specimen is that the c-faces of the specimen

within the epoxy may be constrained by contact with the epoxy so that the outer layers of the specimen are subjected to a higher stress than the inner layers. Because of the low shear strength of the material, it is possible that the stress may fall off rapidly from the outer layers to the inner layers. If this condition applies, the outer layers will have higher than average strain and the inner layers will have lower than average strain. On the basis of this hypothesis, one would expect a more uniform stress distribution in a thin specimen than in a thick specimen, so that the modulus as measured on the c-faces should increase and approach its "true" value as the thickness of the specimen decreases. However, no such pattern emerges from the results in Table II. Further tests will be made to test the hypothesis. The difficulty must be resolved in order to obtain reliable results.

The values for the compliance s_{12} which are given in Table II. fall within a fairly narrow range. However, if the stress is higher than average in the outer layers of the specimen, the transverse strain on the c-faces is also higher than average and the absolute value of the compliance s_{12} is too high. The only measurement of s_{13} was made on specimen M 103-322, for which the longitudinal measurements are very questionable. The gages for the measurement of the transverse strain in this test were on the a-faces, and they sampled mainly the inner layers of the material; if the stress in these inner layers was lower than average, the transverse strain was also lower than average and the absolute value of the compliance s_{13} is too low. All of the numerical results given here must be considered to be tentative values until questions concerning the stress distribution within the tensile specimens are answered.

Shear Tests

The results of a series of shear tests on commercial quality compression annealed pyrolytic graphite are shown in Figure 15. The material which was used appeared to be as flat and shiny and as free from defects as any we have seen. Specimens were prepared for each set of shear fixtures, and ten to twelve tests were made with each set of fixtures. In Figure 15, the shear strength, σ_s , is plotted as a function of the normal stress, σ_n . The average values and the spread of values obtained for each set of fixtures are shown, and the plot is extrapolated back to zero normal stress. The shear strength in the limit of zero normal stress was found to be equal to 129 lb/in.², with a spread of about 10 per cent, for this particular material.

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FIGURE 15. Shear Strength Versus Normal Stress for Some Compression Annealed Pyrolytic Graphite

Some other shear tests were made on compression annealed pyrolytic graphite material which had a somewhat wavy surface. Using the shear fixtures with $\theta = 15^\circ$, an average shear strength of 205 lb/in.² was obtained; the shear fixtures with $\theta = 30^\circ$ yielded an average shear strength of 172 lb/in.². The extrapolated value of the shear strength in the limit of zero normal stress was 152 lb/in.² for this material.

The shear test is simple to perform, and it may prove to be an effective test for characterizing different materials. Additional fixtures are being prepared with different cross-sections, and further tests will be performed on other specimens of compression annealed graphite.

7. ULTRASONIC AND RESONANT BAR TESTS ON PYROLYTIC GRAPHITE

G. B. Spence, D. G. Proctor and O. L. Blakslee

7.1. Sample Preparation

The problem of machining samples of compression annealed pyrolytic graphite for the compound torsion oscillator experiment has been solved. The plate of compression annealed material is placed between the ends of two pieces of drill rod which are the same diameter as the final sample. One rod is held by a collet chuck in the head stock of a lathe and the other is placed against a live center in the tail stock and pressure applied. Nothing is placed between the sample and the ends of the drill rod; the drive is by friction only. The actual machining is done by a tool post grinder.

7.2. Resonant Bar Equipment: Sample Holder

Construction of the new resonant bar equipment, described in the First Quarterly Report⁽¹⁾, has been completed. Figure 16. shows the entire apparatus, and Figure 17. shows the details of the rack which holds the sample. The apparatus has greatly facilitated the handling of small bars

FIGURE 16. Sonic Modulus Resonant Bar Apparatus

N-6774

FIGURE 17. Details of Sample Holder for Sonic Measurements

N-6775

of compression annealed pyrolytic graphite and the precise positioning of the tone arms on the sample.

The sample rack has three supports. The two side supports slide in a track and are attached to a pair of arms that act as proportional dividers. The nodal points* for any particular resonance are proportional to the length of the bar. When the pins on the divider arms are placed at the ends of a bar, the supports are automatically set for the fundamental flexural or the second torsional - second longitudinal nodal points depending upon where along the arms the pins are set. A tee-square slide is used to assure correct positioning of the sample on the supports. For all odd harmonics (fundamental = 1) of the longitudinal and torsional resonances and all even flexural resonances, there is a nodal point at the center. When one of these modes is measured, a knob on the left is turned which raises the center support above those on either side by means of a cam.

The transducers (Weathers stereo cartridges C-501 wired for either vertical or horizontal drive and receive) are mounted so that they can be quickly positioned for either transverse or longitudinal vibrations without affecting the balance of the tone arms. Either of the tone arms or both can be raised from the sample by foot-switch-operated electromagnets, leaving the operator's hands free to manipulate the sample or transducers.

One of the features of this apparatus is the versatility and precision of adjustment. The three carriages can be roughly positioned by the hand traverse, and the two pickup arms can be precisely moved horizontally by the crossfeed in one direction and the compound rest in the other. The sample can be positioned horizontally by the crossfeed and vertically by the compound rest.

* It is important to suspend a resonant bar at its nodal points so that the resonant frequency is not affected by the support.

7.3. Compound Torsion Oscillator

One of the problems of this project has been to find c-axis samples long enough to measure as resonant bars. Resonant bar measurements are possible on very short samples if the ends of the short pieces of graphite are weighted, lowering the resonant frequency to a measurable range. The configuration currently being used is a short cylinder about $\frac{1}{4}$ in. long and $\frac{1}{2}$ in. diameter, weighted on both ends by pieces of $\frac{3}{4}$ in. long steel drill rod of the same diameter. An actual sample is shown in Figure 17.

The exact frequency equations for the torsion oscillator have been derived:

for odd harmonics $1T, 3T, 5T \dots$

$$X \tan X = \frac{\rho \ell}{2\rho' \ell'} \frac{Y}{\tan Y},$$

and for even harmonics $2T, 4T, 6T \dots$

$$\frac{X}{\tan X} = - \frac{\rho \ell}{2\rho' \ell'} \frac{Y}{\tan Y},$$

where $X = \pi f \ell \sqrt{\rho/c_{44}},$

$$Y = 2\pi f \ell' \sqrt{\rho'/c_{44}'},$$

f = resonant frequency,

ℓ = sample length,

ℓ' = length of individual inertia mass,

ρ = sample density,

ρ' = density of inertia masses,

c_{44} = sample shear constant, and

c_{44}' = shear constant of inertia masses.

For this solution: both inertia masses must be of length ℓ' ; the diameters of both masses and of the sample must be equal; and both the sample and inertia masses must be isotropic or, if anisotropic, must be of hexagonal symmetry with their c-axes parallel to the mass - sample - mass

axis. Since the hand solution of the transcendental equation is a lengthy process, the problem has been programmed for an electronic computer.

Measurements have been made on samples as short as 1 mm. One of the important factors of this test is the quality of the bond between the graphite and the steel. Thermoplastic cement and salol have both been used successfully. The drawbacks are that the former gives a thick joint and the latter a joint which is very fragile and sensitive to shock.

The results of this technique have been checked against the results of the conventional resonant bar test for aluminum and against the results of the ultrasonic method for un-annealed and compression annealed pyrolytic graphite. For aluminum, the compound torsion oscillator result is 3 per cent low; but for pyrolytic graphite, which has a much lower modulus, the different techniques are within 1 per cent or better on the same sample.

7.4. Results of Ultrasonic and Resonant Bar Tests

Several samples of compression annealed pyrolytic graphite were measured ultrasonically at one megacycle. The results are given in Table III.

TABLE III.

ELASTIC CONSTANTS OF COMPRESSION ANNEALED PYROLYTIC GRAPHITE
FROM ULTRASONIC TESTS

Sample	C_{33}	C_{44}	
	10^{11} d/cm^2	10^{11} d/cm^2	10^6 psi
P208-113		0.0213	0.0309
M 103-10A		0.0185	0.0268
M 103-10B		0.0184	0.0267
M 103-10C		0.0186	0.0270
M 103-10D		0.0189	0.0274
M 103-10E	1.6	0.0184	0.0267
C 101	2.9	0.0183	0.0265
C 105		0.0200	0.0290

The M 103-10 series of samples denote measurements made at different positions

on the same plate of methane material. Dolling and Brockhouse² obtained a value for c_{33} of $3.9 \pm 0.4 \times 10^{11}$ d/cm² on a sample of pyrolytic graphite which had been thermally annealed at $\sim 3600^\circ$ C. but which was probably not so well aligned as those measured here. However, it is doubtful that variations in the quality of the material can account for all of the differences among the three values of c_{33} . On the other hand, the very low value of c_{44} is thought to be due to dislocation contributions to the strain; thus, variations in material quality could easily account for the observed differences in c_{44} .

Thinner sections of M 103-10A, 10B, 10E, and a fourth sample from the same disk were measured by the compound torsion oscillator technique. These results, given in Table IV., are in good agreement with the ultrasonic results. (Although c_{44} and $1/s_{44}$ are theoretically equal for this material, it is convenient to denote values derived by high frequency ultrasonic methods by c_{44} and values derived by low frequency resonant bar or compound torsion oscillator methods by $1/s_{44}$.)

TABLE IV.

SHEAR MODULUS FROM COMPOUND TORSION OSCILLATOR TESTS

Sample	$1/s_{44}$	
	10^{11} d/cm ²	10^8 psi
M 103-10A	0.0189	0.0274
M 103-10B	0.0186	0.0270
M 103-10E	0.0185	0.0268
M 103-12	0.0184	0.0267

The fundamental torsional and flexural resonant frequencies were measured on a rectangular bar of compression annealed material cut parallel to the basal plane. Since the ends of the bar were slightly crooked, the bar was shortened four times and the resonances remeasured.

The extreme anisotropy of compression annealed pyrolytic graphite causes certain simplifications in the analysis of the torsional resonant frequency and certain complications in the analysis of the flexural resonant frequencies. The torsional resonant frequency of a rectangular bar with its axis parallel to the basal planes is a complicated function⁽³⁾ of the two shear constants $1/s_{44}$ and $1/2 (s_{11}-s_{12})$ and of the bar dimensions. For ordinary materials, it is necessary to know accurately the resonant frequency and one shear constant; the other shear constant can then be computed. For compression annealed pyrolytic graphite, the torsional resonant frequency is almost independent of $1/2 (s_{11}-s_{12})$; therefore, this constant need not be known accurately in order to compute an accurate value of $1/s_{44}$. Conversely an accurate value of $1/2 (s_{11}-s_{12})$ cannot be calculated, because a slight error in the measured torsional frequency would cause a very large error in the value of $1/2 (s_{11}-s_{12})$.

As discussed in the First Quarterly Progress Report⁽⁴⁾, for flexural vibrations perpendicular to the basal plane of compression annealed pyrolytic graphite, the resonant frequency is determined primarily by the shear modulus $1/s_{44}$, instead of by the basal plane Young's modulus $1/s_{11}$. A computer program is being written which will solve Timoshenko's equation for the shear modulus as well as for Young's modulus.

For flexural vibrations parallel to the basal plane of compression annealed pyrolytic graphite, the resonant frequency is determined primarily by $1/s_{11}$. The Poisson's ratio which occurs in Timoshenko's equation is $-s_{12}/s_{11}$, and the data of Table II. indicate that for compression annealed pyrolytic graphite the value of this quantity is about 0.17. Since this number is in the normal range for Poisson's ratio, this mode of flexural vibration can be analyzed by our existing computer program.

Values of $1/s_{11}$ and $1/s_{44}$, derived from the resonant frequency of the bars mentioned above, are given in Table V. The quantity L is the length

TABLE V.

ELASTIC CONSTANTS OF COMPRESSION ANNEALED PYROLYTIC GRAPHITE
FROM RESONANT BAR TESTS

Sample	L cm	$1/s_{11}$		$1/s_{44}$	
		10^{11} d/cm^2	10^6 psi	10^{11} d/cm^2	10^6 psi
M 103-11A	13.1	67	97	0.0210	0.0305
M 103-11B	11.6	66	96	0.0206	0.0298
M 103-11C	10.7	62	91	0.0209	0.0302
M 103-11D	8.6	71	103	0.0202	0.0293

of the bar; the cross sectional dimensions are 1.0 x 0.84 cm. The values of the shear modulus were influenced very little by the misalignment of the ends of the original bar, M 103-11A. However, the values are all approximately 10 per cent higher than those obtained from adjacent samples of the same material by ultrasonic and compound torsion oscillator methods. No reason for this discrepancy is known, but a 10 per cent error in the shear modulus is trivial compared with the expected 10-fold increase on neutron irradiation. Evidently, the value of Young's modulus is influenced by the crooked ends, and only the value for the shortest bar is reliable. This value is within the range of values (given in Table II) obtained from static tensile tests and is within the range $60 \pm 20 \times 10^{11} \text{ d/cm}^2$ measured by Baker and Kelly⁽⁵⁾ on graphite single crystals.

8. PLANS FOR FUTURE WORK

The primary objective of the next quarter will be the completion of all experimental apparatus necessary for the measurement of stress-strain

diagrams of pyrolytic graphite in torsion and single crystals in tension.

The evaluation of the methane and propane material will be carried out with the objective of deciding what type of material should be ordered for the third furnace load. The production of single crystals of graphite and further optimization of the compression annealing process will receive considerable effort.

The preliminary plans for the irradiations, which are very important to the second phase of this program, will be initiated during this period. It is expected that this irradiation schedule will be subject to redefinition as the program progresses.

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