GENERAL ATOMIC

GA-A13524
UC-77

MECHANICAL PROPERTIES OF GRAPHITE FOR HIGH-TEMPERATURE GAS-COOLED REACTORS: A REVIEW

by
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Prepared under
Contract E(04-3)-167
Project Agreement No. 17
for the

San Francisco Operations Office
U.S. Energy Research and Development Administration

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Date: *06/26/2007*

GENERAL ATOMIC PROJECT 3224

DATE PUBLISHED: SEPTEMBER 22, 1975

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$\frac{d}{dt} \left(\frac{\partial L}{\partial \dot{x}} \right) = \frac{\partial L}{\partial x}$

ABSTRACT

Experimental data on the mechanical properties of reactor graphites (excluding irradiation creep and fatigue) under High-Temperature Gas-Cooled Reactor (HTGR) conditions are reviewed.

Unirradiated graphite exhibits a nonlinear (approximately parabolic) stress-strain curve and a "permanent set" when the load is reduced to zero. Poisson's ratio decreases with increasing strain. The tensile strength tends to decrease with increasing specimen size. The flexural strength is higher than the tensile strength by 30% to 100%. Both Young's modulus and strength increase with increasing measurement temperature, fractional increases being greater for the strength. The strength is generally lower under biaxial tension-tension or tension-compression than under uniaxial tension. Failure under multiaxial loading agrees best with Ely's constant strain energy criterion. A limited amount of work on notched test samples indicates that fracture mechanics analysis may be applicable to graphite. Some of the characteristics of graphite failure are compatible with the Weibull statistical theory of strength, but this theory does not provide a reliably self-consistent model. No single theory has been found to predict a failure criterion applicable under all conditions of loading.

Oxidation drastically reduces both the strength and Young's modulus: a 1% burn-off reduces the strength by about 10%.

Neutron irradiation changes the mechanical properties of graphites by (1) pinning basal dislocations and (2) closing microcracks. Irradiation progressively removes the curvature from the stress-strain curve. Dynamic determinations of Young's modulus increase rapidly and then level off; the level of the plateau decreases with increasing irradiation temperature. Further irradiation causes a second increase and finally a decrease in

Young's modulus. The static Young's modulus of irradiated graphite agrees with the dynamic modulus, whereas the static modulus of unirradiated graphite is lower than the dynamic modulus. No systematic effect of irradiation on Poisson's ratio has been found. Irradiation increases the tensile and flexural strengths. At low irradiation temperatures, the strength, S , and Young's modulus, E , of unirradiated and irradiated samples are related by the condition of constant strain energy fracture; thus, $S^2/E = \text{constant}$. At higher irradiation temperatures, the strengths are often somewhat higher than predicted by the constant strain energy condition and may approach the condition $S/E = \text{constant}$. The effect of irradiation on the scatter in strength values is not clearly defined.

Most published work on the effect of oxidation in a radiation field relates to carbon dioxide. Oxidation by carbon dioxide in the presence of irradiation reduces the strength more gradually than the oxidation of unirradiated graphite.

Based on an assessment of the literature, recommendations are made for design-basis values for the elastic properties and strength of irradiated graphite, and failure criteria are suggested for particular loading conditions.

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1. INTRODUCTION

1.1. SCOPE OF REVIEW

The graphite moderator blocks in the core of an HTGR serve as primary structural components. Accurate values for the elastic properties of the blocks under service conditions are needed for stress calculations, and strength data are needed to assess the reliability of the design.

In this report the available data (including effects of neutron irradiation) on stress-strain curves, elastic constants, Poisson's ratio, strength, fracture toughness, failure criteria, and failure under complex stress systems are reviewed. The literature on the uniaxial mechanical properties of unirradiated graphites is covered only in outline because a number of published review articles (Refs. 1-4) summarize this area. Irradiation-induced creep was reviewed in a previous report (Ref. 5), as was fatigue in graphite (Ref. 6); consequently neither topic is considered here. Finally, design-basis curves for the elastic moduli and strength of HTGR graphites as functions of irradiation conditions are recommended, and suggestions are made as to appropriate mechanical failure criteria.

1.2. FAST NEUTRON FLUENCE UNITS

In many of the figures in this review, fast neutron fluences are given in terms of neutrons with energies greater than 0.18 MeV for specimens irradiated in test capsules in light-water-moderated test reactors ($E > 0.18$ MeV, LWR). Some European data are quoted in terms of the Nickel Dido Equivalent (NDE) fluence. For HTGR design purposes, usual practice utilizes the fluence of neutrons with energies greater than 0.18 MeV in a graphite-moderated HTGR spectrum ($E > 0.18$ MeV, HTGR). For comparisons between different irradiation facilities, the preferred unit is the

Equivalent Fission Fluence for Graphite Damage (EFFGD). Conversion factors between the different units can be calculated on the basis of the number of displaced carbon atoms; on this basis, 1 n/cm² (EFFGD) displaces 720×10^{-24} carbon atoms per atom. Calculated factors for converting fluences in this report expressed as n/cm² (E > 0.18 MeV, LWR) to other units are as follows:

$$\text{Fluence (NDE)} = 0.6 \times \text{fluence (E > 0.18 MeV, LWR)},$$

$$\text{Fluence (E > 0.18 MeV, HTGR)} = 0.89 \times \text{fluence (E > 0.18 MeV, LWR)},$$

$$\text{Fluence (EFFGD)} = 1.12 \times \text{fluence (E > 0.18 MeV, LWR)}.$$

2. MECHANICAL PROPERTIES OF UNIRRADIATED GRAPHITE

2.1. UNIAXIAL STRESS-STRAIN RELATIONSHIP AT ROOM TEMPERATURE

Although polycrystalline graphite is generally regarded as a brittle material, a limited amount of slip on the basal planes is possible even at room temperature, and consequently the stress-strain curves are non-linear. Figure 1 shows an example of a room-temperature stress-strain curve in both compression and tension for a coarse-grained nuclear-grade graphite (EGCR-type AGOT) (Ref. 3). Both the tension and compression portions of the curve are concave toward the strain axis. The strain at failure under compression is typically ten times higher than the strain at failure in tension (the tensile failure strain is about 0.1% to 0.2%), while the ultimate compressive strength is typically four or five times higher than the ultimate tensile strength.

The ultimate tensile strength of specimens taken from large blocks of coarse-grained nuclear-grade graphite falls in the range 1000 to 3000 psi. The strength of large extrusions and moldings is influenced by the type of filler coke and pitch binder, the grain orientation of the filler coke, and the amount of carbon derived from impregnation. For extruded stock the strength is higher in the axial direction than in the radial direction, with the strength anisotropy reflecting the degree of orientation of the filler coke; in extruded graphites made from needle cokes the axial strength may be as much as twice the radial strength. In molded stock the orientation dependence is reversed. Specimens from the center of a log are usually weaker than those from the periphery and ends of the log because of the influence of impregnant carbon.

When graphite is subjected to a tensile stress close to its ultimate strength, fracture may occur after a period of time with no increase in

Fig. 1. Uniaxial stress-strain curve for EGCR-type AGOT graphite, parallel to extrusion axis (from Ref. 3)

stress (delayed fracture or static fatigue). This effect is usually masked by scatter in the data. One series of tests on extruded RC-4 graphite tested as cantilever beams in air at room temperature (Ref. 7) was designed to allow for the spread in strengths. In these tests delayed fracture was shown to occur after 10^5 sec in specimens stressed at 97% of their expected fracture stress.

If a load is applied and then removed, the stress-strain curve does not retrace its original path; a residual strain remains when the stress is reduced to zero ("permanent set"). Examples of stress-strain curves produced during load cycling in both tension and compression are shown in Figs. 2 and 3 (Ref. 1). If the load is reapplied, the reloading curve (dashed lines in Figs. 2 and 3) deviates slightly from the unloading curve, resulting in a narrow hysteresis loop. The reloading curves rejoin the original loading curve shortly after the load-reversal point is passed.

2.2. ELASTIC MODULI AT ROOM TEMPERATURE

The non-Hookean behavior causes a problem in defining Young's modulus for graphite. Some commonly used Young's moduli are:

1. Sonic modulus (dynamic modulus) obtained from the frequency of vibration in the longitudinal stretching or flexural mode.
2. Tangent modulus at zero stress, i.e., the slope of the stress-strain curve at the origin.
3. Quasi-elastic modulus after preloading and unloading, i.e., the slope of a straight line connecting the unloading and reloading points on the curves in Fig. 2.
4. Chord modulus, i.e., the slope between two specified stress points on the original loading curve.

Fig. 2. Tensile stress-strain curve with repeated loading for unspecified nuclear graphite, parallel to extrusion axis (from Ref. 1)

Fig. 3. Compressive stress-strain curve with repeated loading for unspecified nuclear graphite, parallel to extrusion axis (from Ref. 1)

5. Secant modulus, i.e., ultimate tensile strength divided by the strain at failure.

The tangent modulus (slope at the origin) is the same for compression and tension and is usually somewhat lower than the sonic modulus (Fig. 4) (Ref. 2). In tension, the quasi-elastic modulus is close to the tangent modulus and only slightly dependent on the amount of preload (see Fig. 2) (Ref. 1), but in compression the quasi-elastic modulus decreases with increasing preload (Fig. 3) (Ref. 1). For engineering purposes, the tensile quasi-elastic modulus is the most useful Young's modulus, because it closely approximates the elastic response of a graphite component after initial loading and because it can be more accurately measured from a stress-strain curve than the tangent modulus. With repeated cycling, the width of the hysteresis loop decreases during the first few cycles but the value of the quasi-elastic modulus changes very little (Fig. 5) (Ref. 4).

The Young's modulus of nuclear graphites falls in the range 0.5 to 2×10^6 psi, depending on the preferred orientation, density, and crystallinity of the material.

Figure 3 shows that extensive preloading in compression reduces the quasi-elastic modulus. Compressive preloading also reduces the sonic modulus and the tensile moduli (Refs. 1,2,8). Annealing to the graphitization temperature restores the mechanical response of a preloaded specimen to its original condition (Refs. 2,4).

2.3. LATERAL STRAINS AND POISSON'S RATIO

Although the stress-longitudinal strain curves have a symmetrical shape in tension and compression (Figs. 1 and 4), the stress-lateral strain curves are significantly different in tension and compression (Refs. 2,4). During a tensile test the stress-lateral strain curve is convex toward the strain axis, whereas during a compressive test the curve is concave toward the strain axis. An example is shown in Fig. 6 (Ref. 4). One result is

Fig. 4. Composite tension and compression data for two specimens of ATJ graphite, in the with-grain direction (from Ref. 2)

Fig. 5. Cyclic stress-strain curves for EGCR-type AGOT graphite, parallel to the extrusion axis (12 cycles) (from Ref. 4)

Fig. 6. Schematic drawings of longitudinal and lateral stress-strain curves for nuclear graphite (from Ref. 4)

that after loading in tension and returning to zero stress, the specimen has a positive permanent set in both the longitudinal and transverse directions. Another result is that Poisson's ratio measured during a tensile test decreases with increasing strain, but during a compression test Poisson's ratio is relatively insensitive to strain level. An example is shown in Fig. 7.

For an isotropic graphite, Poisson's ratio is independent of direction and falls in the range 0.1 to 0.25 (Refs. 9-11). For a needle-coke graphite stressed along the axis of fabrication, Poisson's ratio is the same in all orthogonal directions [$\mu_{31} = \mu_{32} = -(S_{13}/S_{33})$] and is typically 0.1 to 0.15. When stress is applied in the radial direction, Poisson's ratio calculated from the strain measured in the other radial direction [$\mu_{12} = \mu_{21} = -(S_{12}/S_{11})$] is about 0.1, while Poisson's ratio for the axial strain [$\mu_{13} = \mu_{23} = -(S_{13}/S_{11})$] is somewhat lower (typically 0.05) (Ref. 4).

2.4. EQUATIONS FOR STRESS-STRAIN CURVES

Several workers have postulated models to represent the stress-strain behavior of polycrystalline graphite. The one which has received the most acceptance is that of Jenkins (Ref. 12). This model proposes a limited amount of plastic strain occurring in individual grains; the number of grains with plastic strain increases as the applied stress increases. Rheologically, this is equivalent to a series of springs and friction elements. The initial loading curve has the form

$$\epsilon = A\sigma + B\sigma^2 ,$$

where ϵ is the strain, σ is the stress, A is the reciprocal of the tangent modulus, and B is a plastic compliance parameter. The equation for unloading from a peak stress, σ_m , and peak strain, ϵ_m , is

$$\epsilon_m - \epsilon = A(\sigma_m - \sigma) + \frac{1}{2} B(\sigma_m - \sigma)^2 ,$$

Fig. 7. Transverse-to-longitudinal strain ratio curves for EGCR-type AGOT graphite (from Ref. 4)

and the equation for reloading from σ_o is

$$\epsilon - \epsilon_o = A(\sigma - \sigma_o) + \frac{1}{2} B(\sigma - \sigma_o)^2 .$$

Although based on a very simple concept, Jenkins' equations fit the mechanical behavior of nuclear graphite remarkably well. An alternative model by Hesketh (Ref. 13) visualizes an aggregate of grains with a distribution of yield stresses. Hesketh's model predicts a loading curve of the form

$$\sigma = \frac{E\epsilon}{6} \left(5 - \frac{9}{8} \frac{\epsilon}{\epsilon_y} \right) ,$$

where E is the single crystal elastic modulus and ϵ_y is the single crystal yield strain. Woolley (Ref. 14) derived a model based on the motion of basal dislocations in which grains become plastic progressively. The predicted loading curve has the form

$$\sigma = E\epsilon_o \left[1 - \exp \left(- \frac{\epsilon}{\epsilon_o} \right) \right] ,$$

where E is the tangent Young's modulus and ϵ_o is a constant. Both Hesketh's and Woolley's equations can be made to fit observed initial loading curves, but unlike Jenkins' model they cannot easily be extended to unloading or reloading curves.

2.5. EFFECT OF SPECIMEN SIZE ON TENSILE STRENGTH

The effect of specimen size on ultimate tensile strength has been the subject of several investigations which gave no consistency of results. Losty and Orchard (Ref. 1) found that tensile specimens of an unspecified nuclear graphite with cross-sectional areas of 0.5, 0.250, and 0.125 in.² had similar tensile strengths, while specimens with a 0.0625-in.² area had just significantly lower strengths. No similar size effect was found for

flexural specimens. Digesu and Pears (Ref. 15) tested specimens of ATJ graphite whose diameters ranged from 0.063 to 1 in. The ultimate tensile strengths were similar except for the 1-in.-diameter specimens, whose mean strength was 11% lower. Greenstreet et al. (Ref. 16) found no significant effect of specimen size on the strength of EGCR-type AGOT graphite for diameters in the range 0.128 to 0.75 in. In contrast, Lungagnani and Krefeld (Ref. 17) found that the mean strength of tensile specimens of a Gilso-coke graphite decreased progressively as the specimen volume increased from 0.7 to 6.2 cm³, with the smallest specimens averaging 30% stronger than the largest specimens. Experiments on near-isotropic H-451 graphite at General Atomic Company showed that tensile specimens measuring 0.5 in. in diameter by 3 in. in length average about 5% weaker than specimens measuring 0.25 in. in diameter by 0.9 in. in length (Ref. 18).

Interpretation of the effect of specimen size on strength is discussed in Section 2.10 of this report.

2.6. FLEXURAL TESTS AT ROOM TEMPERATURE

When graphite specimens are tested in bending and the bending moment at failure is used to calculate the modulus of rupture assuming linearly elastic behavior, the values obtained are between 1.5 and 2 times higher than the ultimate tensile strength. Greenstreet et al. (Ref. 16) performed flexural tests on EGCR-type AGOT graphite and analyzed the results taking nonlinearity into account. They found that the true outer fiber stress at failure in the bend tests agreed with the ultimate tensile strength after the observed ultimate tensile strength had been increased to allow for nonuniformity of strain around the circumference. In contrast, Brocklehurst and Darby (Ref. 19) found that for an isotropic nuclear graphite, the conventional modulus of rupture overestimates the true outer fiber stress by only a few percent.

A rigorous treatment of the effects of nonlinearity on the flexural strength is complex because such a treatment should take into account transverse strains and the shift in the neutral axis of the beam. A

simplified approach may be made by using one of the expressions in Section 2.4 in place of Hooke's law to calculate the relationship between bending moment and outer fiber strain of the beam. Woolley's equation (Ref. 14) relates stress, σ , strain, ϵ , Young's modulus, E , and a curvature parameter, ϵ_o :

$$\sigma = E\epsilon_o \left[1 - \exp \left(-\frac{\epsilon}{\epsilon_o} \right) \right] .$$

This equation yields the following relationships between the modulus of rupture (bend strength calculated assuming elastic behavior) and the true outer fiber strain at fracture, ϵ_f :

Rectangular cross-section beam

$$MOR = E\epsilon_o \left\{ 1.5 - \frac{3\epsilon_o^2}{\epsilon_f^2} \left[1 - \left(1 + \frac{\epsilon_f}{\epsilon_o} \right) \exp \left(-\frac{\epsilon_f}{\epsilon_o} \right) \right] \right\} ;$$

Cylindrical beam

$$MOR = E\epsilon_f \left[1 - 0.340 \left(\frac{\epsilon_f}{\epsilon_o} \right) + 8.33 \times 10^{-2} \left(\frac{\epsilon_f}{\epsilon_o} \right)^2 - 1.62 \times 10^{-2} \left(\frac{\epsilon_f}{\epsilon_o} \right)^3 + 2.60 \times 10^{-3} \left(\frac{\epsilon_f}{\epsilon_o} \right)^4 \right] .$$

Correction factors which may be applied to convert the conventionally calculated modulus of rupture to the true outer fiber stress at fracture are plotted in Fig. 8 for a graphite with $\epsilon_o = 0.004$ (appropriate for a near-isotropic petroleum-coke-based graphite such as Great Lakes Carbon Company grade H-451). These correction factors were applied to the results of four-point bend tests conducted at General Atomic Company on H-451 near-isotropic graphite (Ref. 18). The corrected flexural strengths averaged

Fig. 8. Correction factors for bend-test data to account for nonlinear stress-strain curve (H-451 graphite)

about 50% higher than the tensile strengths of companion samples, whereas uncorrected moduli of rupture averaged 80% higher. It is noteworthy that high bend strength-to-tensile strength ratios are also found in baked carbon bodies and neutron-irradiated graphites, even though these materials exhibit linear stress-strain behavior.

The difference between bend and tensile strengths probably occurs because the strength of graphite is controlled by flaws. Models such as the Weibull theory of fracture (discussed more fully in Section 2.10) assume that the material contains a distribution of weakening flaws, and failure is linked to the probability of encountering a major flaw in a highly stressed zone. Weibull calculations have been found to be in fairly good agreement with the bend-to-tensile strength ratio of unirradiated needle-coke H-327 graphite (Ref. 20) and near-isotropic H-451 graphite (Ref. 18).

2.7. EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES

As the test temperature is increased, the strength of graphite rises progressively up to about 2500°C. A typical example of the variation of ultimate tensile strength with temperature (Ref. 21) is shown in Fig. 9. At low strain rates (less than 0.5/min), the strength typically increases over its room-temperature value by 25% at 1000°C, by 50% at 2000°C, and by 100% at 2500°C. Above 2500°C, plastic deformation becomes extensive and the strength again declines.

Time-dependent creep is insignificant at temperatures below 2000°C (except under the influence of irradiation). Above 2000°C, creep rates increase sharply with both temperature and stress, and tensile elongations to fracture rise steeply from a fraction of a percent at 2000°C to several percent at 2500°C. In this temperature range, both the elongation and strength decrease with increasing strain rate.

Elastic modulus measurements at high temperatures have usually employed dynamic techniques. Up to about 2000°C, Young's modulus increases with

Fig. 9. Transverse tensile strength of H4LM graphite as a function of temperature when tested in static helium at 0.005/min (from Ref. 21)

temperature, increasing by 10% to 15% at 1000°C and 30% to 40% at 2000°C. An example is shown in Fig. 10 (Ref. 22). The dynamic Young's modulus passes through a maximum at 2000° to 2200°C (Ref. 23). When measured by static techniques, the initial increase with temperature is less marked and the modulus starts to decrease at a lower temperature, possibly because of the influence of plastic strain (Ref. 24).

2.8. MECHANICAL BEHAVIOR UNDER MULTIAXIAL STRESSES

Several studies have been made in which graphite tubes have been tested to failure under a combination of internal pressure and axial tension or compression. The failure points have been compared with the predictions of various theories for failure under multiaxial stress, but few definitive conclusions have been reached. The different failure theories are summarized in Section 2.11, while the experimental data are outlined in this section.

Some results for Graph-1-Tite grades A and G obtained by Ely (Ref. 25) are typical and are reproduced in Fig. 11. In the tension-tension quadrant, failure generally occurs under somewhat lower tensile stresses than in uniaxial tension. In the tension-compression quadrant, the tensile strength is not reduced until the applied orthogonal compressive stresses exceed the applied tensile stress, but it decreases toward zero as the compressive stress approaches the uniaxial ultimate compressive strength. Ely concluded that failure corresponding to a maximum strain energy, or a modification of the maximum strain energy failure criterion due to Stassi-D'Alia, was in fair agreement with the data in the tension-compression quadrant, but the tension-tension data agreed better with the maximum normal stress failure condition. In a later series of tests (Ref. 26), Ely concluded that an alternative modification of the maximum strain energy criterion, in which the three principal stresses are normalized according to the corresponding ultimate strength for the same direction, fitted the data in both the tension-tension and tension-compression quadrants (see Section 2.11).

Fig. 10. Dynamic Young's modulus versus temperature for representative commercial graphites (from Ref. 22)

Fig. 11. Strength of Graph-1-Tite tubes under combined stresses: (a) grade A, batch 1; (b) grade A, batch 2; and (c) grade G (from Ref. 25)

Broutman et al. (Ref. 27) tested Poco AXM-5Q graphite and found that biaxial tensile strengths in the tension-tension quadrant were reduced considerably below the uniaxial tensile strength. They found that the tension-compression failure envelope was in fair agreement with the modified maximum strain energy theory or the Coulomb-Mohr theory ($\sigma_1/S_T - \sigma_2/S_C = 1$, where σ_1 and σ_2 are the maximum and minimum principal stresses and S_T and S_C are the ultimate tensile strength and ultimate compressive strength). In the tension-tension quadrant, the maximum strain energy theory was considered a better fit than the maximum normal stress theory but still overestimated the strength.

ATJ-S cylinders equipped with strain gauges were tested by Babcock et al. (Ref. 28) in an apparatus which enabled the compression-compression quadrant to be explored in addition to tension-tension and tension-compression. The compressive fracture stress data could be approximated by either the Coulomb-Mohr theory or Norris' modification of the Von Mises yield criterion. A plot of the strains at failure was very scattered and showed no systematic failure envelope.

Similar tests on grade ATJ graphite by Tu-Lung Weng (Ref. 29) showed that Ely's modification of the maximum strain energy failure criterion made the best overall fit to the data in all four quadrants.

Strain-gauged tubes of Poco AXF-5Q graphite were tested under biaxial conditions by Jortner (Ref. 30) at 70°F, and additional tests were made at 3000° and 4000°F. The failure stress and failure strain envelopes at 70°F are shown in Fig. 12 (a and b). The failure stresses in the tension-tension envelope were found to fit predictions based on the Weibull theory for the strength of brittle solids, but the Weibull theory does not predict the decrease in tensile strength at high orthogonal compressive loads. The maximum shear stress (Mohr-Coulomb) failure criterion was a better fit in this region. The tensile strains at failure (Fig. 12b) were reduced below the uniaxial fracture strain in the tension-tension quadrant and increased above it in the tension-compression quadrant. For the higher testing

Fig. 12. Biaxial fracture data for AXF-5Q graphite tubes tested at room temperature: (a) fracture stresses; (b) fracture strains (from Ref. 30)

temperatures, the fracture stress envelopes were similar in shape to the room-temperature envelopes but were displaced toward higher stresses.

Biaxial tests by Yahr et al. (Ref. 31) on an unspecified "extruded specialty graphite" showed results in reasonable agreement with either the maximum normal stress criterion or Ely's modification of the maximum strain energy criterion. The maximum shear stress criterion disagreed with the tension-compression data.

An assessment of the experimental results summarized above and the alternative failure criteria is given in Section 2.11.

2.9. APPLICATION OF FRACTURE MECHANICS TO GRAPHITE

The analytical methods of fracture mechanics are used to predict the nominal stress levels at which flaws in a material can propagate and cause brittle fracture. Although originally developed for high-strength steel structures, the techniques should be applicable to a brittle material containing flaws, such as graphite. For conditions of plane strain (those most likely to cause a propagating crack), the basic equation relating the nominal fracture stress, σ , to the flaw size, a , is

$$\sigma = \frac{K_{IC}}{Q \sqrt{\pi a}} \quad , \quad (1)$$

where Q is a function of the loading geometry and flaw size and K_{IC} is a material property called the plane strain fracture toughness or critical stress intensity factor. K_{IC} is usually measured by machining notches or forming controlled cracks in tensile or beam specimens and measuring the fracture stress. Q is either calculated analytically or measured from the specimen compliance under load as a function of the notch depth. The machined notch must be sharp enough to ensure plane strain conditions at the tip and must be deep compared with the inherent flaws in the material. If the notch is not deep compared with the inherent flaw size, the material will not be notch-sensitive and Eq. 1 will not be valid.

Slind (Ref. 32) measured the load required to propagate cracks in double cantilevered beam specimens of SGBF nuclear graphite. The tests yielded consistent values for K_{IC} which were independent of the specimen width and crack length as expected from fracture mechanics concepts. A mean value of $1.39 \text{ ksi } \sqrt{\text{in.}}$ for with-grain fractures was obtained for K_{IC} .

Notched-beam specimens of EGCR-type AGOT graphite were tested by Corum (Ref. 33). Results were calculated using both the analytical fracture mechanics formula and the alternative method in which the compliance of the beam is measured as a function of crack depth. The two methods gave similar results. Consistent results were obtained provided that the ratio of crack depth to specimen width was greater than 0.1 to 0.2. However, Corum noted that if the effective crack depth was assumed equal to the artificial crack depth plus one or two grain diameters, consistent results would be obtained for specimens with all crack depths. Mean values of K_{IC} were $0.84 \text{ ksi } \sqrt{\text{in.}}$ for specimens stressed parallel to extrusion and $0.54 \text{ ksi } \sqrt{\text{in.}}$ for specimens stressed perpendicular to extrusion. In the latter case, there was no difference between the values obtained when cracks propagated parallel and perpendicular to extrusion.

The most extensive series of fracture mechanics tests reported to date was performed by Yahr et al. (Refs. 34-36). Notched beams of Poco AXM graphite and Union Carbide ATJ graphite were tested with several different sizes and notch shapes and depths, together with notched tensile specimens and double cantilevered beams (Ref. 34). K_{IC} values showed a tendency to decrease as the crack sharpness increased, and they were independent of specimen configuration provided that the crack depth-to-specimen width ratio was greater than 0.1 for ATJ graphite or 0.05 for the finer-grained AXM. The following values for K_{IC} ($\pm$ one standard deviation) were obtained:

AXM, all orientations:	$1.17 \pm 0.08 \text{ ksi } \sqrt{\text{in.}}$
------------------------	--

ATJ, with-grain loading, with-grain cracking:	$0.82 \pm 0.05 \text{ ksi } \sqrt{\text{in.}}$
--	--

ATJ, with-grain loading,
across-grain cracking:

$$0.81 \pm 0.06 \text{ ksi } \sqrt{\text{in.}}$$

ATJ, across-grain loading,
with-grain cracking:

$$0.69 \pm 0.04 \text{ ksi } \sqrt{\text{in.}}$$

It was noted that if these values are combined with the observed tensile strength of the materials, critical flaw sizes of 0.01 in. for ATJ graphite and 0.003 in. for AXM graphite are calculated. These sizes are in reasonable agreement with the largest intergranular pores observed micrographically.

Two later series of tests (Refs. 35,36) examined the application of fracture mechanics to the same two graphites under complex stress conditions. In the first series, circular or elliptical disks of ATJ and AXM graphite with slits cut at the center were loaded in compression along the diameter or major axis until failure occurred by cracks propagating from the slits (Ref. 35). The stress at the center is biaxial; the principal stresses are tension perpendicular to loading and compression parallel to loading. It was found that the load at failure could reasonably be predicted from fracture mechanics considerations, using the values of K_{IC} listed above, provided that the testing geometry was not invalidated by crushing at the points of contact with the loading platens. The second series of tests (Ref. 36) used thick-walled, closed-end cylinders with circumferential notches machined diagonally at the wall-to-end junction. The cylinders were pressurized until they failed by the end breaking off. The failure pressures were in good agreement with predictions based on fracture mechanics.

Mean K_{IC} values of 1.54 and 1.36 ksi $\sqrt{\text{in.}}$ were obtained for two United Kingdom nuclear isotropic graphites by Marshall and Priddle, who used the "compact tension" notched slab specimen (Ref. 37). In a subsequent paper (Ref. 38), the same authors suggested that fracture mechanics concepts might be used to explain the difference between the flexural strength and tensile strength of graphite specimens. If a flexural or tensile specimen

is regarded as a uniform beam with an edge notch equal to the maximum pore size, fracture mechanics formulae may be used to predict the ratio of bend strength to tensile strength. The specimens tested were 6 mm wide and contained pores between 0.6 and 2 mm wide. For these flaw sizes, the predicted bend-to-tensile strength ratio is 1.2 to 1.5, in rough agreement with the experimental results. However, this approach cannot be correct in general because it would require that if large specimens or fine-pored graphites are used, the flexural strength will equal the tensile strength, and this is not observed.

Andersson and Salkovitz (Ref. 39) measured the fracture toughness of Union Carbide grades ZTA and ATJ and Poco graphite grades AXZ and AXF-5Q graphites. They used center-notched plate specimens under uniaxial tension and used the results to calculate both K_{IC} and the critical strain energy release rate, G_{IC} . [For plane strain conditions, $G_{IC} = K_{IC}^2 (1-\nu^2)/E$, where ν is Poisson's ratio and E is Young's modulus.] The results were as follows:

<u>Material</u>	<u>Orientation (to molding axis) (degrees)</u>	<u>K_{IC} (psi-in.^{1/2})</u>	<u>G_{IC} (psi-in.)</u>
ZTA	0	660	0.660
ZTA	90	1440	0.760
ATJ	0	750	0.580
ATJ	90	905	0.580
AXF-5Q	--	1120	0.590
AXZ	--	720	0.360

The data indicate that, for a given material, G_{IC} is less orientation-dependent than K_{IC} . Further, G_{IC} for the four graphite grades is approximately a linear function of the fractional porosity, extrapolating to 0.97 psi-in. for fully dense material.

To summarize, analyses based on fracture mechanics may be applied to notched graphite test specimens provided that the notch depth is large

compared with the inherent pore size and that the conditions at the crack tip are approximately plane-strain. Experimental values for K_{IC} range between 0.5 and 1.5 ksi- $\sqrt{\text{in.}}$ with no clear systematic dependence on graphite type. G_{IC} values appear to increase with decreasing porosity.

2.10. APPLICATION OF WEIBULL STATISTICAL THEORY TO GRAPHITE

Several characteristics of the strength of many brittle solids (for example, the variability in strength, the dependence of mean strength on specimen volume, and the difference between ultimate tensile strength and flexural strength) are qualitatively consistent with Weibull's statistical theory of strength (Refs. 40,41). Weibull's model assumes that the material contains a distribution of flaws of varying size. When the material is subjected to a tensile stress, the strength is controlled by the combination of the highest local stress and the most severe flaw. The probability of survival, $S(\sigma)$, of a small volume element under a tensile stress σ is assumed to have the form

$$\ln [S(\sigma)] = -\left(\frac{\sigma - \sigma_u}{\sigma_o}\right)^m .$$

This yields the following expression for the failure probability of the bulk specimen, F :

$$F = 1 - \exp \left[- \int_V \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m dV \right] , \quad (2)$$

where σ is the tensile stress in the element, σ_u is the "minimum failure stress" below which the failure probability is zero, σ_o is a normalizing parameter, and m is a parameter called the Weibull modulus. σ_u , σ_o , and m are determined from experimental strength measurements by best-fit methods. For simple non-uniform stress systems, Eq. 2 may be integrated analytically to give the following type of expression for the ratio of the

median strength under non-uniform stress, σ_{nu} , to the median tensile strength, σ_t :

$$\frac{\sigma_{nu}}{\sigma_t} = \left(\frac{V_t}{V_{nu}} \right)^{1/m} f(m, \text{stress gradient}) , \quad (3)$$

where V_t and V_{nu} are the volume of the tensile specimens and non-uniform stress specimens, respectively. The last term in Eq. 3 depends on the stress gradient and is different for three-point bending, four-point bending, tubes with internal pressurization, biaxial loading, etc.; its value depends on the Weibull modulus, m . Some numerical values are given in Ref. 20.

Several tests of the applicability of the Weibull model to graphite have been made. Greenstreet et al. (Ref. 16) tested batches of 24 to 32 replicate specimens of AGOT, using two sizes of tensile specimen and two sizes of bend specimen. Each set of data was analyzed to obtain m , σ_u , and σ_o . Reasonably consistent values for m (in the range 3 to 4) were obtained, except for the data on large bend specimens, but no consistent values for σ_u and σ_o . The dependence of mean strength on specimen size was much less marked than that predicted by Eq. 3. It was concluded that the data did not fit the Weibull theory.

By contrast, Lungagrami and Krefeld (Ref. 17) tested tensile specimens of a Gilsocarbon-based nuclear graphite with volumes between 0.7 and 6.2 cm^3 and obtained a single set of values for m , σ_u , and σ_o which fitted the strength distribution of all specimen sets with the possible exception of the smallest size.

Price and Cobb (Ref. 20) fitted Weibull parameters to sets of tensile data on H-327 needle-coke graphite from different parts of the log. Bend tests and thick-walled tube-burst tests showed higher mean strengths than the tensile tests; however, the data could be made to fit the Weibull model only if the volume dependence term (Eq. 3) was ignored.

Brocklehurst and Darby (Ref. 19) drew somewhat similar conclusions from tensile, bend, tube-burst, and ring diametral compression tests on isotropic nuclear graphite. They observed the expected increase in mean strength with increasing stress gradient, but found that the m-value deduced from the distribution of tensile strengths greatly overestimated the volume dependence of strength, while slightly underestimating the dependence on stress gradient. It was concluded that while it is possible to evaluate Weibull parameters for a single test, no consistent values can be derived which fit all the data.

Comparison of bend and tensile tests on a variety of Pechiney graphites (Ref. 42) also led to the conclusion that the Weibull model overestimates the specimen volume effect.

Experiments at General Atomic Company on near-isotropic H-451 graphite (Ref. 18) showed that the distribution of tensile strengths was fairly well represented by a Weibull distribution function with an m-value averaging about 9. The mean tensile strength of 0.5-in.-diameter by 3-in.-long specimens was about 5% lower than that of 0.25-in.-diameter by 0.9-in.-long specimens, whereas the Weibull model with $m = 9$ would predict a difference in mean strengths of 25%. The flexural strength of 0.25-in.-diameter specimens averaged 50% higher than the tensile strengths of companion specimens, in fairly good agreement with a value of 59% predicted by the Weibull theory.

The last four studies all assigned a value of zero to σ_u . This is a conservative assumption, and it has been justified by measurements on Poco graphite which showed that there was no significant difference in the fit of the experimental data obtained by taking a best-fit value for σ_u and taking $\sigma_u = 0$ (Ref. 43).

It may be concluded that while the Weibull model appears to fit the tensile strength distribution and ratios of tensile strength to tube-burst strength and flexural strength of many graphites, it greatly overestimates

the volume effect. Thus, it does not constitute a self-consistent model for the behavior of most graphites.

2.11. FAILURE CRITERIA FOR GRAPHITE

Since plastic flow in graphite is negligible, failure occurs by brittle fracture with the propagation of a crack across a load-bearing section. When uniaxial tensile or compressive tests are conducted, the condition for failure may arbitrarily be taken as a critical stress, a critical strain, or some other parameter calculated from the stress or strain. However, when graphite is loaded by a complex or non-uniform system of stresses, the question of choosing a failure criterion becomes important.

A number of different failure criteria have been suggested, but none has been generally adopted as applicable to graphite under all loading conditions. Several of these failure criteria may be distinguished by their different predicted failure envelopes for multiaxial stress systems. The failure envelopes for an isotropic graphite (with Poisson's ratio = 0.2 and the ultimate compressive strength three times the ultimate tensile strength) under a biaxial stress are shown in Fig. 13 for six of the most commonly tested criteria. In the definitions of failure criteria which follow, isotropic elastic behavior is assumed. σ_1 , σ_2 , and σ_3 are the three principal stresses; $\sigma_{\max}$ and $\sigma_{\min}$ are the maximum and minimum principal stresses; S_T and S_C are the ultimate tensile and compressive strengths, respectively; ν is Poisson's ratio; and E is Young's modulus.

2.11.1. Maximum Normal Stress

$$\sigma_{\max} = S_T \quad \text{or} \quad \sigma_{\min} = S_C \quad .$$

This is the simplest and most commonly used failure criterion. It is conservative when used to predict failure in systems with a stress gradient, such as beams in bending, but tends to overestimate the strength of graphite in biaxial tension (see Section 2.8).

Fig. 13. Biaxial failure envelopes predicted by different failure criteria for an isotropic graphite with Poisson's ratio = 0.2 and ultimate compressive strength = 3 x ultimate tensile strength

2.11.2. Maximum Normal Strain

$$\sigma_1 - \nu(\sigma_2 + \sigma_3) = S_T \quad .$$

It has been suggested that failure in graphite should be controlled by a limiting tensile strain, largely on the grounds that processes which increase Young's modulus (such as neutron irradiation) or decrease Young's modulus (such as graphitization) have a similar effect on the strength. However, examination of batches of tensile data from replicate specimens shows larger coefficients of variation for the fracture strains than for the fracture stresses. Furthermore, a constant strain criterion would imply substantially higher strengths in biaxial tension than uniaxial tension (see Fig. 13), which is contrary to observations. There seems to be no experimental justification for such a failure criterion.

2.11.3. Maximum Shear Stress (Coulomb, Tresca, or Mohr Criterion)

$$\frac{\sigma_{\max} - \sigma_{\min}}{2} = \tau_{\max} \quad (\text{shear stress at failure}) \quad .$$

This criterion is often applied to yielding in metals. When applied to the failure of brittle materials, it is necessary to assign a coefficient of friction between the shear planes to account for the higher ultimate strength in compression as compared with tension (Fig. 13). A number of authors (Refs. 27,28,30) have compared biaxial failure results with this criterion and observed fair agreement with experimental data in the tension-compression quadrant. However, two objections may be raised against the application of this criterion to graphite: (1) a criterion based on yielding in shear is not realistic for graphite, which fractures by crack propagation; and (2) the criterion requires that the shear strength be half the uniaxial tensile strength, which is not true for graphite.

2.11.4. Distortion Energy (Octahedral Shear Stress or Von Mises Criterion)

$$\left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 \right]^{1/2} = \sqrt{2} S_T \quad .$$

This yield criterion also was developed from the plastic yield of metals. In the form given above, it predicts symmetrical behavior in tension-tension and compression-compression (see Fig. 12). A modification by Norris (Ref. 44) allows the failure envelope to pass through the uniaxial compressive strength point; in this form the criterion has been applied to tension-compression failure in graphite (Refs. 28,29). However, it predicts that biaxial strength in the tension-tension quadrant should be higher than the uniaxial tensile strength, which is contrary to observations.

2.11.5. Maximum Strain Energy

The strain energy criterion most often used for graphite is that of Ely (Ref. 26). For an isotropic graphite under biaxial loading, the appropriate equations for tension-tension and tension-compression, respectively, are

$$\left(\frac{\sigma_1}{S_T} \right)^2 - 2\nu \left(\frac{\sigma_1}{S_T} \right) \left(\frac{\sigma_2}{S_T} \right) + \left(\frac{\sigma_2}{S_T} \right)^2 = 1$$

and

$$\left(\frac{\sigma_1}{S_T} \right)^2 - 2\nu \left(\frac{\sigma_1}{S_T} \right) \left(\frac{\sigma_2}{S_C} \right) + \left(\frac{\sigma_2}{S_C} \right)^2 = 1 \quad .$$

This failure criterion has been found to be in reasonable agreement with both tension-tension and tension-compression experiments (Refs. 25-27, 29,31) and correctly predicts the lowered strengths in biaxial tension-tension.

2.11.6. Weibull Theory

As discussed in Section 2.10, the Weibull statistical theory goes a long way toward explaining many aspects of failure in graphite, but fails to provide a completely self-consistent model because it overestimates the effect of specimen volume. The theory may be extended to multiaxial stress states because, if the internal flaws are considered planar, the survival probability is influenced by the presence of multiaxial stresses (Refs. 20, 40). The failure envelope for a graphite with a Weibull modulus, m , of 5 is shown in Fig. 13. The predicted envelope is in fairly good agreement with experimental data in the tension-tension quadrant (Ref. 30), but fails to predict a decrease in strength as the ultimate compressive strength is approached.

The application of the Weibull theory to multiaxial stress states for the purpose of designing with brittle materials is covered in detail in Ref. 45.

2.11.7. Fracture Mechanics

In principle, the techniques of fracture mechanics (Section 2.9) can be extended to any complex stress state. Yahr et al. (Refs. 35,36) successfully applied fracture mechanics calculations to the biaxial stress state present in circular and elliptical graphite disks loaded along the major axis and to pressurized closed-end cylinders with circumferential notches at the wall-end junction. However, analytical expressions have been worked out only for a few stressing modes and crack geometries, and it is not yet possible to quote generalized failure criteria based on fracture mechanics.

Fracture mechanics techniques may be used during the engineering design of brittle materials to impose a conservative stress limit (Ref. 46). If the material is likely to contain weakening flaws, the size of the largest flaw which could escape detection by nondestructive examination is substituted into the appropriate formula (Eq. 1), and if the corresponding

stress is lower than the safe working stress obtained from strength measurements, the more conservative value must be used.

2.11.8. Mean Local Strain Energy Density in Plane of Crack

Brocklehurst and Darby (Ref. 19) advanced the hypothesis that, in the case of four-point bend tests and pressurized cylinders, fracture occurs when the mean strain energy density averaged over the tensile part of the failure cross section equals the strain energy density for tensile fracture, $S_T^2/2E$. This approach predicts that the four-point flexural strength should be 1.73 times the ultimate tensile strength, and that the failure hoop stress, S_H , in an internally pressurized cylinder of diameter ratio R (internal radius divided by external radius) should be given by

$$\frac{S_H}{S_T} = \left(\frac{1+R^2}{2R^2} \right)^{1/2} .$$

Although neither relationship exactly fits the data, the general trends are accounted for. However, this criterion cannot be applied to cases where the stress varies in two dimensions (such as a three-point bend test) because the result depends on the volume of material over which averaging is carried out.

2.11.9. Conclusions on Failure Criteria

It may be concluded that the maximum normal stress failure criterion is adequate for graphite under a predominantly uniform, uniaxial stress (provided that the statistical variation in strength is taken into account). It is conservative when applied to systems with a stress gradient, but non-conservative when applied under multiaxial stresses. Under multiaxial stresses, Ely's maximum strain energy criterion provides the best overall agreement with the data. Where a stress gradient is present, the factors predicted by Weibull's theory fit the observations best, but this model

overestimates the effect of specimen volume. Clearly, the subject of failure criteria in graphite would benefit from more theoretical and experimental investigation.

2.12. EFFECT OF OXIDATION ON STRENGTH OF GRAPHITE

Oxidizing gases attack the binder phase of polycrystalline graphite in preference to the filler. This results in a very rapid decrease in strength and elastic modulus. Most of the published data refer to nuclear graphites oxidized in carbon dioxide or air at temperatures between 500° and 1000°C (Refs. 47-50). The results show a parallel decline in tensile, compressive, flexural, and shear strength, with the strengths falling 50% for weight losses between 5% and 10%. Typical results (Ref. 47) are shown in Fig. 14. Young's modulus decreases at about the same rate as the strength, leaving the strain to failure almost unchanged.

The only anticipated oxidizing impurity in the HTGR coolant is a small amount of water vapor from occasional heat-exchanger leaks. Measurements of the tensile strength of H-327 graphite oxidized in a steam-helium mixture (Ref. 51) are very similar to the results on graphite oxidized in air or carbon dioxide, with the strength dropping by half after about 5% burnoff.

Krefeld et al. (Ref. 52) have reported experiments in which four different types of graphite were alternately loaded and unloaded in tension while exposed to an argon - 1500 vpm water atmosphere at 1000°C. The rate of oxidation (as measured by the change in gas composition) increased by a factor of 3 to 6 when tensile stresses between 280 and 770 psi were applied.

Fig. 14. Compressive strength of Pile Grade A graphite as a function of the extent of oxidation (from Ref. 47)

3. MECHANICAL PROPERTIES OF IRRADIATED GRAPHITE

3.1. EFFECT OF IRRADIATION ON STRESS-STRAIN CURVE

The most important effects of neutron irradiation on the mechanical properties of graphite result from the pinning of basal dislocations by irradiation-induced defect clusters. This reduces the amount of basal slip in a crystallite during mechanical loading and increases the effective shear modulus. One result is to reduce greatly the curvature of the stress-strain curve. Taylor et al. (Ref. 10) found that irradiation at 150°C progressively straightened the stress-strain curves of United Kingdom isotropic graphite both in tension and compression. An example is shown in Fig. 15.

Everett and Ridealgh (Ref. 53) found that curvature in the tensile stress-strain curve of a pressed Gilsocarbon graphite (Dragon code No. 95) was greatly reduced, but not eliminated, after irradiation at 1200°C to 10^{21} n/cm² (NDE). The "permanent set" observed during mechanical loading of unirradiated graphite was reduced to less than 0.01% strain (after loading to 70% of the tensile strength and unloading) following irradiation.

Stress-strain measurements at General Atomic Company on H-451 near-isotropic graphite and H-327 needle-coke graphite irradiated at temperatures between 600° and 1350°C in capsule OG-1 (Ref. 54) showed that the elimination of curvature is more nearly complete at low irradiation temperatures than at high irradiation temperatures. Examples of stress-strain curves are shown in Fig. 16. In the tests from which these curves were obtained, the stress was first increased to 1000 psi, then reduced to 100 psi, and then increased until the specimen failed.

Fig. 15. Variation in stress-strain curves in tension with increasing neutron fluence at 150°C; isotropic nuclear graphite (from Ref. 10)

Fig. 16. Typical tensile stress-strain curves for unirradiated and irradiated H-451 graphite (from Ref. 54)

3.2. EFFECT OF IRRADIATION ON YOUNG'S MODULUS

Irradiation-induced pinning of basal dislocations increases the effective C_{44} shear modulus of the crystallites, which in turn increases the elastic moduli of the bulk specimen. Most reported measurements of irradiation-induced increases in Young's modulus have been obtained from sonic measurements of the vibrational frequency of cylindrical samples, because such measurements are nondestructive and the specimen may be reirradiated. Other reported data are derived from static stress-strain curves. It is important to note that the two techniques may not give identical results, especially with unirradiated or lightly irradiated material. In particular, the fractional increase in sonic modulus may be lower than the fractional increase in static modulus, because the sonic modulus of unirradiated material is usually higher than the static modulus, whereas the two measurements are closer in irradiated material. All measurements reported so far have been made at room temperature.

The largest body of data on the effects of neutron irradiation on Young's modulus of graphites comes from Dragon project sonic modulus measurements on cylindrical specimens irradiated to progressively higher fluences (Refs. 55-59). Curves showing the percent increase in sonic modulus as a function of fast neutron fluence are shown in Figs. 17 through 21 for (1) extruded anisotropic petroleum coke graphite (Dragon code No. 59/2); (2) extruded near-isotropic petroleum-coke graphite (Dragon code No. 120); (3) extruded pitch-coke graphite (Dragon code No. 100); (4) molded pitch-coke graphite (Dragon code No. 113); and (5) molded Gilsocarbon graphite (Dragon code No. 95).

The Dragon data show that the irradiation-induced changes in Young's modulus follow a similar pattern in all types of graphite. A rapid initial increase is followed by a plateau whose level decreases with increasing irradiation temperature. The plateau is followed by a second rise in modulus which may extend to 2.5 times the unirradiated level. The modulus then passes through a maximum and then decreases, finally falling below

Fig. 17. Fractional changes in dynamic Young's modulus of PGA graphite with double pitch impregnation (Dragon code No. 59/2) (from Ref. 56)

Fig. 18. Fractional changes in dynamic Young's modulus of extruded petroleum-coke graphite (Dragon code No. 120) (from Ref. 59)

Fig. 19. Fractional changes in dynamic Young's modulus of extruded pitch-coke graphite (Dragon code No. 100) (from Ref. 59)

Fig. 20. Fractional changes in dynamic Young's modulus of molded pitch-coke graphite (Dragon code No. 113) (from Ref. 59)

Fig. 21. Fractional changes in dynamic Young's modulus of molded Gilsocarbon graphite (Dragon code No. 95) (from Ref. 58)

the unirradiated level at very high fluences. The second increase in modulus starts sooner at higher irradiation temperatures; however, the height of the subsequent peak is less for the highest irradiation temperatures studied (1300° to 1400°C) than it is in the vicinity of 1000°C. The sequence of changes may be explained in terms of structural changes in the graphite. The rise to the first plateau may be attributed to the pinning of basal dislocations by small point defect clusters. The second rise may be explained by the progressive tightening of the structure as microcracks close and intercrystalline restraint increases. The final decline is probably caused by the opening of new intercrystalline pores as the graphite enters its volume expansion phase.

With the exception of highly oriented graphites, the fractional change in Young's modulus is similar for the axial and the radial directions. There are some differences in behavior between different types of graphite. The increases are generally higher for Gilsocarbon-based graphites (Fig. 21) than for petroleum-coke- or pitch-coke-based graphites. There is also a tendency for the maximum in the modulus-fluence curve of petroleum-coke or pitch-coke graphites to be higher for pressed graphites than for graphites extruded using the same filler (compare Figs. 19 and 20).

Sonic modulus measurements on isotropic graphites irradiated at lower temperatures by United Kingdom Atomic Energy Authority workers (Refs. 60-62) show trends similar to the Dragon data. At an irradiation temperature of 350° to 440°C, modulus increases up to 300% were observed after irradiation in the Dounreay Fast Reactor to 2×10^{22} n/cm² (NDE) (Ref. 62). An example is shown in Fig. 22. French data on a Gilsocarbon graphite irradiated at 500° to 700°C (Ref. 63) and German data on Gilsocarbon and petroleum-coke graphites irradiated at 600° to 1200°C (Ref. 64) also show trends similar to the Dragon results.

Sonic modulus data have been measured on many varieties of graphite irradiated at Pacific Northwest Laboratory (Refs. 65-68). Unfortunately, much of the data is so scattered that it is impossible to compare with other results. Some smoothed curves obtained from a needle-coke graphite

Fig. 22. Fractional changes in dynamic Young's modulus, E , and strength, S , of isotropic graphites at temperatures of 350° to 440°C in Dounreay Fast Reactor and 700°C in BR-2 (from Ref. 62)

(grade CSF) are shown in Fig. 23 (Ref. 66). Although the curves show an initial temperature-dependent rise roughly similar to the Dragon curves, they do not show the second rise and peak in modulus characteristic of the Dragon results. It may be that the scatter in the data masks the details of the curves.

Young's modulus was measured from the stress-strain curves of tensile specimens irradiated at 600° to 1400°C in capsules OG-1 and OG-2 by General Atomic Company (Refs. 69,70). The specimens were 0.25-in.-diameter by 0.9-in.-long cylinders of H-327 (needle coke), H-451, and TS-1240 (near-isotropic petroleum coke) graphites irradiated in batches of 8 to 12 replicates and subsequently tension-tested to failure. The modulus was measured from the reloading part of the curve after loading to 1000 psi, unloading to 100 psi, and reloading to failure (see Fig. 16). The increase in modulus is shown in Figs. 24 through 26 and the absolute values are listed in Table 1. The error bars on the curves indicate $\pm$ one standard deviation. The fractional increases in modulus are considerably greater than would be expected from the Dragon curves. The most probable cause of the discrepancy is that the sonic modulus of the unirradiated graphites tested by the Dragon project is significantly higher than the static modulus measured from the stress-strain curve; but after irradiation the sonic and static moduli agree. The General Atomic Company static modulus increases and the Dragon sonic modulus increases can be brought into agreement if the static modulus of unirradiated graphite is assumed to be 80% of the sonic modulus (see Section 4.3).

3.3. EFFECT OF IRRADIATION ON OTHER ELASTIC CONSTANTS

The only measurements of elastic constants other than Young's modulus for irradiated polycrystalline graphite were reported by Goggin and Reynolds (Ref. 9). Complete sets of elastic constants were obtained on a needle-coke graphite (PGA) and a near-isotropic graphite irradiated at 25°C until the initial increase in modulus was complete (10^{19} n/cm² NDE). The results are shown in Table 2. It may be seen that all the compliance constants decrease in about the same ratio.

Fig. 23. Fractional changes in Young's modulus versus fluence for an anisotropic reactor graphite. A = axial, R = radial (data from Refs. 66 and 67)

Fig. 24a. Tensile strength and static Young's modulus of H-327 needle-coke graphite as a function of fast neutron fluence. Error bands indicate $\pm$ one standard deviation. Irradiation temperature 640° to 900°C. (From Ref. 70)

Fig. 24b. Tensile strength and static Young's modulus of H-327 needle-coke graphite as a function of fast neutron fluence. Error bands indicate $\pm$ one standard deviation. Irradiation temperature 940° to 1200°C. (From Ref. 70)

Fig. 25. Tensile strength and static Young's modulus of H-451 near-isotropic petroleum-coke graphite as a function of fast neutron fluence. Error bands indicate $\pm$ one standard deviation. (From Ref. 70)

Fig. 26. Tensile strength and static Young's modulus of TS-1240 near-isotropic petroleum-coke graphite as a function of fast neutron fluence. Error bands indicate $\pm$ one standard deviation. (From Ref. 70)

TABLE 1
SUMMARY OF MECHANICAL PROPERTY DATA ON GRAPHITES IRRADIATED IN CAPSULES OG-1 and OG-2

Material	Orientation	Location in Log	Mean Irradiation Temperature (°C)	Fluence x 10 ⁻²¹ (n/cm ²) (E > 0.18 MeV)	Ultimate Tensile Strength (psi)		Young's Modulus x 10 ⁻⁶ (psi)	
					Observed (± std. dev.)	Predicted ^(a)	Observed (± std. dev.)	Predicted ^(a)
H-451	Axial	Midlength - center	--	0	1564 ± 135	--	1.14 ± 0.09	--
H-451	Axial	Midlength - center	580-630	1.6	2424 ± 265	2140	2.63 ± 0.20	2.2
H-451	Axial	Midlength - center	580-630	2.2	2485 ± 177	2160	2.22 ± 0.11	2.2
H-451	Axial	Midlength - center	580-630	2.9	2481 ± 322	2180	2.54 ± 0.20	2.2
H-451	Axial	Midlength - center	910-950	3.4	2371 ± 271	2040	2.06 ± 0.24	2.0
H-451	Axial	Midlength - center	890-970	5.9	2777 ± 261	2200	2.55 ± 0.15	2.4
H-451	Axial	Midlength - center	1340-1370	3.6	2233 ± 115	2000	1.79 ± 0.08	1.9
H-451	Radial	Midlength - center	--	0	1671 ± 242	--	1.00 ± 0.05	--
H-451	Radial	Midlength - center	900-950	3.1	2648 ± 270	2190	1.88 ± 0.13	1.8
TS-1240	Axial	Midlength - center	--	0	1571 ± 252	--	0.90 ± 0.10	--
TS-1240	Axial	Midlength - center	620	1.3	1844 ± 452	2140	1.72 ± 0.32	1.7
TS-1240	Axial	Midlength - center	765	1.9	2070 ± 385	2100	1.72 ± 0.17	1.6
TS-1240	Axial	Midlength - center	920	2.8	2123 ± 259	2070	1.66 ± 0.13	1.6
TS-1240	Axial	Midlength - center	1105	3.3	1899 ± 540	2050	1.69 ± 0.21	1.6
TS-1240	Radial	Midlength - center	--	0	1383 ± 298	--	0.90 ± 0.11	--
TS-1240	Radial	Midlength - center	805	1.9	1951 ± 327	1850	1.50 ± 0.26	1.6
H-327	Axial	Midlength - center	--	0	1321 ± 158	--	1.30 ± 0.12	--
H-327	Axial	Midlength - center	740	2.5	1944 ± 377	1790	2.25 ± 0.40	2.4
H-327	Axial	Midlength - center	750-810	4.0	2423 ± 341	1800	2.77 ± 0.49	2.4
H-327	Axial	Midlength - center	835-850	5.0	2435 ± 361	1810	2.55 ± 0.32	2.5
H-327	Axial	Midlength - center	860-900	2.6	2127 ± 281	1740	3.02 ± 0.27	2.3
H-327	Axial	Midlength - center	960-1035	3.0	1940 ± 331	1730	2.48 ± 0.40	2.3
H-327	Axial	Midlength - center	940-1035	6.4	2222 ± 468	1890	2.32 ± 0.31	2.6
H-327	Axial	Midlength - center	1040-1200	7.0	2003 ± 585	1850	2.32 ± 0.30	2.5
H-327	Axial	Midlength - edge	--	0	2186 ± 293	--	1.93 ± 0.25	--
H-327	Axial	Midlength - edge	640-700	2.9	3152 ± 278	3000	3.85 ± 0.33	3.7
H-327	Axial	Midlength - edge	770-810	2.1	2787 ± 249	2930	3.10 ± 0.26	3.5
H-327	Axial	Midlength - edge	1050-1100	3.7	2822 ± 254	2860	3.23 ± 0.19	3.4
H-327	Radial	Midlength - center	--	0	820 ± 286	--	0.64 ± 0.08	--
H-327	Radial	Midlength - center	960-1020	3.4	962 ± 198	1070	1.23 ± 0.20	1.1

^(a) Predicted from Figs. 39 and 40.

TABLE 2
MEASURED ELASTIC COMPLIANCE MODULI OF GRAPHITE BLOCKS
IN UNITS OF 10^{-14} CM²/DYNE (FROM REF. 9)

Material	Modulus	Value of Modulus x 10^{14} (cm ² /dyne)	
		Unirradiated	Irradiated (10^{19} n/cm ² at 25°C)
PGA	S_{11}	2150	520
PGA	S_{33}	1087	279
PGA	S_{13} (from S_{13}/S_{33})	-162	-42
PGA	S_{13} (from S_{13}/S_{11})	-123	-19
PGA	S_{12} (from S_{12}/S_{11})	-127	-41
PGA	S_{12} (from $1/2 S_{44} + S_{11} - S_{12}$)	-530	-123
PGA	S_{44}	3333	925
Isotropic	S_{11}	1370	355
Isotropic	S_{12} (from S_{12}/S_{11})	-148	-32
Isotropic	S_{12} (from $S_{11} - 1/2 S_{44}$)	-145	-99
Isotropic	S_{44}	3030	909

3.4. EFFECT OF IRRADIATION ON POISSON'S RATIO

In the work described above, Goggin and Reynolds (Ref. 9) measured Poisson's ratio for an isotropic graphite irradiated at room temperature. Compressive loads of 5 to 40 psi were applied, and the strains were measured by Cornu's method. The observed values were plotted against the reciprocal of the load and extrapolated to infinite load. The values obtained decreased slightly from 0.108 (unirradiated) to 0.090 (at 10^{19} n/cm²). Changes in the three Poisson's ratios of the highly oriented PGA graphite were as follows:

$$-\frac{S_{12}}{S_{11}}: \text{ unirradiated } 0.059, \text{ irradiated } 0.079$$

$$-\frac{S_{13}}{S_{11}}: \text{ unirradiated } 0.057; \text{ irradiated } 0.037$$

$$-\frac{S_{13}}{S_{33}}: \text{ unirradiated } 0.149; \text{ irradiated } 0.150$$

Brocklehurst and Lyman (Ref. 71) found that the Poisson's ratios of PGA graphite (measured under compressive loads between 400 and 4000 psi) increased on irradiation at 150°C, with $-(S_{13}/S_{33})$ changing from 0.13 before irradiation to about 0.3 at an exposure of 6.5×10^{20} n/cm². The data are shown in Fig. 27. In contrast, Taylor et al. (Ref. 10), using a similar technique, found no systematic trends in the Poisson's ratio (initially 0.17) of two types of isotropic graphite irradiated at 150°C to a peak fluence of 1.1×10^{21} n/cm² (NDE). The data are given in Table 3.

It may be concluded that experiments so far have found no systematic effect of irradiation on Poisson's ratio.

3.5. EFFECT OF IRRADIATION ON STRENGTH

Losty and Orchard (Ref. 1) measured the flexural strength of an unspecified type of reactor graphite after irradiation at 60°C to fluences

Fig. 27. Variation of Poisson's ratio of PGA graphite with fast neutron fluence (from Ref. 71)

TABLE 3A
PHYSICAL AND MECHANICAL PROPERTIES OF UNIRRADIATED GRAPHITES (FROM REF. 10)

Type	Density (g/cm ³)	Direction of Cut	Mean C.T.E. (20°-120°C) (x 10 ⁻⁶ deg/K)	Mean K (cal/cm/ sec/°K)	Tension			Compression			Dynamic Modulus (x 10 ¹⁰ dynes/ cm ²)
					Average Strength (kg/mm ²)	Standard Deviation (kg/mm ²)	Average % Strain to Fracture	Average Strength (kg/mm ²)	Standard Deviation (kg/mm ²)	Average % Strain to Fracture	
1	1.78 ±0.05	Parallel Perpendicular	3.4	0.335	1.06 }	0.13	0.244	4.95 }	0.35	2.15	9.7
			3.8	0.315	1.07 }		0.260	4.78 }		2.42	8.7
2	1.85 ±0.04	Parallel Perpendicular	3.5	0.320	1.51 }	0.21	0.215	7.65 }	0.43	2.75	11.4
			4.1	0.290	1.40 }		0.215	7.23 }		3.02	10.4
3	1.86 ±0.04	Parallel Perpendicular	3.0	0.390	1.13 }	0.22	0.19	6.75 }	0.57	2.56	12.0
			3.4	0.370	1.34 }		0.20	6.85 }		2.50	11.0

TABLE 3B
CHANGE IN MECHANICAL PROPERTIES AFTER NEUTRON IRRADIATION (FROM REF. 10)

Property	Direction of Cut	Type 1 Material							Type 2 Material							Type 3 Material				
		Neutron Dose (n/cm ² x 10 ²⁰)																		
		0 ^(a)	0.75	1.55	2.91	3.60	7.5	11.5	0 ^(a)	0.75	1.55	2.70	3.60	7.5	11.5	0 ^(a)	0.85	2.90	4.70	7.5
Change in tensile strength, $\sigma T/\sigma T_0$		1	1.85	2.09	1.71	2.07	2.08	1.75	1	2.11	2.40	2.00	1.92	1.79	1.98	1	1.49	1.58	1.05	1.65
	⊥	1	1.88	2.02	1.90	1.97	1.94	2.05	1	1.97	1.95	1.90	1.81	1.81	1.71	1	1.35	1.90	1.63	1.45
Change in tensile strain to fracture		1	0.47	0.66	0.53	0.97	0.49	0.59	1	0.74	1.30	0.69	0.97	0.48	0.63	1	0.57	0.77	0.53	0.52
	⊥	1	0.40	0.63	0.68	0.77	0.62	0.69	1	0.71	0.85	0.98	0.93	0.95	0.38	1	0.55	0.94	0.72	0.49
Change in compressive strength, $\sigma C/\sigma C_0$		1	2.13	2.51	2.90	2.99	3.11	3.21	1	1.95	2.32	2.65	2.76	3.02	2.94	1	2.08	2.58	3.04	3.30
	⊥	1	2.01	2.55	2.90	3.04	3.23	3.47	1	1.97	2.30	2.54	2.90	3.00	3.14	1	1.88	2.44	2.91	3.25
Change in compressive strain to fracture		1	0.64	0.70	0.71	0.84	0.62	0.58	1	0.63	0.69	0.75	0.88	0.58	0.56	1	0.61	0.83	0.51	0.53
	⊥	1	0.64	0.64	0.64	0.86	0.59	0.51	1	0.58	0.59	0.58	1.00	0.46	0.47	1	0.65	1.12	0.52	0.56
Poisson's ratio	--	0.17	0.13	0.17	0.19	0.22	0.13	0.14	0.17	0.17	0.17	0.18	0.21	0.10	0.09	--	--	--	--	--
Hardness (kg/mm ²)	--	16.5	36	55	70	79	147	167	19.8	42	64	90	100	135	158	18.5	34	75	100	122
Strain energy (dynes x 10 ⁴)		5.9	11.2	13.3	12.1	13.3	11.9	9.3	9.85	18.2	28.7	18.0	23.0	14.9	15.5	5.25	4.5	7.8	2.3	10.8
	⊥	6.6	10.3	12.3	13.0	13.6	12.0	11.7	9.4	16.0	19.2	17.8	16.5	16.9	12.8	8.2	7.2	15.4	10.2	9.2
Compressive strength		4.7	4.8	5.1	6.7	6.8	6.9	8.1	5.0	5.1	5.2	7.4	7.2	8.8	8.3	6.0	9.7	8.6	20.3	10.2
Tensile strength	⊥	4.5	4.9	6.1	6.9	6.7	7.5	7.9	5.2	5.4	6.1	7.3	8.7	8.3	9.6	5.1	7.2	6.6	9.8	11.1

(a) See Table 3A for absolute values of fracture strength and strain.

up to 10^{19} n/cm². They found that the strength increased at a slower rate than Young's modulus, with the strain energy per unit volume to fracture (assuming linear elastic behavior) remaining approximately constant. The strength, S, and Young's modulus, E, for several fluences and both orientations were related by the expression

$$\frac{S^2}{E} = K \quad ,$$

where K is constant.

Tensile and compressive strengths of three prototype grades of isotropic petroleum-coke graphites irradiated at 150°C to 1.1×10^{21} n/cm² (NDE) were reported by Taylor et al. (Ref. 10). The data are shown in Table 3. The tensile strength of all three materials rose rapidly to about twice the unirradiated value, while the compressive strength increased more slowly to about three times the unirradiated level. In contrast to the results of Losty and Orchard, the strain energy to failure increased significantly on irradiation, while the fracture strain decreased. The shear strength of several samples was measured and was found to increase in the same proportion as the tensile strength.

Reference 62 includes data on the fractional increase in the tensile strength and brittle-ring strength (similar to flexural strength) of molded and extruded isotropic graphites irradiated in the Dounreay Fast Reactor at 350° to 440°C. The type of filler coke was not specified but was said to have a microstructure similar to Gilsocarbon. The results are included in Fig. 22. The strength increased at a lower rate than Young's modulus. The fractional strength increases were proportional to the square root of the fractional Young's modulus increases, in agreement with Losty and Orchard's suggestion of a constant strain energy to fracture. Data on samples taken to higher exposures at 350° to 500°C are reported in Ref. 72 and are reproduced in Fig. 28. Beyond about 2×10^{22} n/cm² the strength fell below the level predicted by the constant strain energy

Fig. 28. Mechanical property changes in extruded isotropic graphite irradiated at 350° to 500°C (from Ref. 72): (a) strength; (b) Young's modulus

relation, but remained above the unirradiated level. The fall-off was attributed to pore generation.

Strength measurements on specimens irradiated at higher temperatures by the Dragon project are reviewed in Ref. 53. A series of cylindrical specimens about 0.4 in. in diameter by 4 in. long made from pressed Gilsocarbon graphite (Dragon code No. 95) were irradiated at 1200°C to 1×10^{21} n/cm² (NDE) during the course of an irradiation creep experiment in the Dragon reactor. After the creep experiment was completed, some of the stressed specimens and some unstressed companions were tested in tension. The strengths were found to be closely proportional to the sonic modulus, with data for unirradiated and irradiated (both stressed and unstressed) specimens falling on the same line (see Fig. 29). A curve corresponding to the constant strain energy condition ($S^2 = KE$) did not fit the data.

Four-point bend-test specimens measuring 0.2 in. in diameter by 2.6 in. in length and made from extruded Gilsocarbon-based graphite fuel sleeves were tested after irradiation in four Dragon fuel elements (Ref. 53). Temperatures ranged from 850° to 1250°C and fluences ranged up to 3×10^{21} n/cm² (NDE). The results are shown in Fig. 30. As in the case of the creep specimens, the strength increases were in better agreement with an $S = KE$ relationship than an $S^2 = KE$ relationship.

A few bend strength results on a fine-grained petroleum-coke graphite irradiated in the Dragon reactor and the Petten High Flux Reactor (HFR) to 5 to 6×10^{21} n/cm² (NDE) at 600°, 900°, and 1100°C were reported by Delle (Ref. 64). At 600°, 900°, and 1100°C the strength increases were 40%, 25%, and 20%, respectively. Other data showed the bend strength increasing after irradiation approximately with the square root of Young's modulus, but the irradiation conditions were not stated.

The first experiments intended to show the effects of irradiation on the scatter of the strength measurements were reported by Lungagnani and Krefeld (Ref. 17). Groups of 12 to 35 bend specimens of a Gilsocarbon

Fig. 29. Strength - Young's modulus relationship for irradiated graphite (Dragon code No. 95). Fluence = 1×10^{21} n/cm² (NDE temperature = 1200°C (from Ref. 53)

Fig. 30. Flexural strength as a function of static Young's modulus for irradiated graphite (from Ref. 53)

graphite were irradiated in the HFR (Petten) at 600° and 1150°C to fluences up to 7×10^{20} n/cm² (NDE). For irradiation at 600°C, both the strength and the experimental scatter increased with irradiation. At 1150°C, the strength first increased, as expected, but then rapidly decreased to below the preirradiation level. These results were in striking contrast to all previous measurements and could not be confirmed in subsequent tests. It was later concluded that the specimens had been slightly oxidized during irradiation (Ref. 73), and the results of these tests should be disregarded.

A considerable amount of mechanical property data was obtained on H-327 (needle coke) graphite irradiated by General Atomic Company in capsules G-13, F-26, F-28, and F-29 (Ref. 74). Results of tensile tests on 0.2-in.-diameter tensile specimens irradiated at 1100° to 1150°C to fluences up to 5×10^{21} n/cm² ($E > 0.18$ MeV) in capsule G-13 are shown in Fig. 31. The tensile strength and Young's modulus increase with fluence. A considerable number of bend tests were performed on H-327 specimens irradiated in capsules F-26, F-28, and F-29 at temperatures in the range 575° to 775°C to fluences up to 8×10^{21} n/cm² ($E > 0.18$ MeV). Axial specimens from the mid-length region of the parent log and radial specimens from the end region of the log were tested in a helium atmosphere at room temperature or at a higher temperature, either close to the irradiation temperature or at 1000° to 1200°C. The flexural strength measurements are shown as a function of fluence in Fig. 32. Although the data are quite scattered, the flexural strengths generally increase with increasing fluence. The effect of testing temperature is not very well defined, but in general the strengths of samples tested at elevated temperatures lie toward the top of the scatter bands, while the room-temperature strengths tend to be lower.

Groups of 8 to 12 replicate specimens of H-327 (needle coke), H-451, and TS-1240 (near-isotropic petroleum coke) graphites were tested in tension at room temperature at General Atomic Company after irradiation in the Oak Ridge Reactor (ORR) in capsules OG-1 and OG-2 (Refs. 69,70). The results are summarized in Table 1, and the increases in strength, together

Fig. 31. Tensile strength and Young's modulus of needle-coke H-327 graphite irradiated in capsule G-13 at 1100° to 1150°C (Ref. 74)

Fig. 32a. Flexural strength of axial specimens of needle-coke H-327 graphite irradiated in capsules F-26, F-28, and F-29 at 575° to 775°C. Closed points were obtained at room temperature and open points at elevated temperature. The first number by each point indicates the irradiation temperature and the second number indicates the testing temperature. (Ref. 74)

Fig. 32b. Flexural strength of radial specimens of needle-coke H-327 graphite irradiated in capsules F-26, F-28, and F-29 at 575° to 775°C. Closed points were obtained at room temperature and open points at elevated temperature. The first number by each point indicates the irradiation temperature and the second number indicates the testing temperature. (Ref. 74)

with standard deviations, are plotted in Figs. 24 through 26. The coefficient of variation of the strength values showed no systematic and statistically significant change with irradiation, with the exception of the groups of TS-1240 and H-327 specimens irradiated to the highest fluences and temperatures, where the coefficient of variation underwent a significant increase at the 95% confidence level.

The tensile strengths of the specimens irradiated in capsules OG-1 and OG-2 are plotted against Young's modulus (measured from the reloading portion of the curve; see Fig. 16) in Figs. 33 through 35. Lines corresponding to $S/E = K$ and $S^2/E = K$ are drawn through the data points obtained for unirradiated specimens. For H-327 and TS-1240 graphites, the data points obtained for irradiated specimens are in fairly good agreement with Losty and Orchard's constant strain energy condition ($S^2/E = K$). For the H-451 graphite, the data points for irradiated specimens generally fall above the $S^2/E = K$ line. If the data points from the lowest temperature and fluence conditions (closed squares in Fig. 34) are excluded, the remaining measurements on axial specimens of H-451 graphite are in fair agreement with the $S/E = K$ failure condition proposed in Ref. 53.

Additional results on the effect of irradiation on the spread in strengths have been published by Matthews (Ref. 75). Rectangular specimens (2 in. by 0.24 in. by 0.12 in.) of Poco AXF-5Q graphite were irradiated at 400°C to fluences up to 3×10^{21} n/cm² ($E > 1$ MeV). Young's modulus was measured from the frequency of flexural vibrations, and the modulus of rupture of groups of 11 to 29 specimens was measured in four-point bending. The mean strengths increased proportionally to the square root of the increase in Young's modulus (Fig. 36), in accordance with the constant strain energy failure condition. The distribution of strengths within each group was fitted to the Weibull distribution function (Eq. 2), assuming $\sigma_u = 0$. The results for σ_0 and m are given in Table 4, together with the corresponding stress for a failure probability of 10^{-6} . It was found that the spread in strengths increased somewhat after irradiation (shown by the decrease in m), with the result that stress calculated for

Fig. 33. Tensile strength-Young's modulus relationships for needle-coke H-327 graphite irradiated in capsules OG-1 and OG-2

Fig. 34. Tensile strength-Young's modulus relationships for near-isotropic petroleum-coke H-451 graphite irradiated in capsules OG-1 and OG-2

Fig. 35. Tensile strength-Young's modulus relationships for near-isotropic petroleum-coke TS-1240 graphite irradiated in capsule OG-2

Fig. 36. Young's modulus and flexural strength of AXF-5Q graphite as a function of fast neutron fluence ($E > 1 \text{ MeV}$) at 400°C . (from Ref. 75)

TABLE 4
WEIBULL ANALYSIS OF IRRADIATED POCO AXF-5Q GRAPHITE
(from Ref. 75)

Dose (n/cm ² x 10 ⁻²⁰)	$\bar{\sigma}$ (MN/m ²)	No. of Samples	m	σ_0 (MN/m ²)	$\sigma \cdot 10^{-6}$ (MN/m ²)
As-received	93.4	24	16	61.5	40.5
1.1	102.5	28	15	66.0	41.2
3.3	103.2	29	10	45.0	29.0
6.0	108.2	11	11	49.4	32.6
10	109.5	26	8	37.2	21.0
30	111.1	29	11	49.3	32.1

a failure probability of 10^{-6} decreased. Matthews associated the increased spread with the volume expansion experienced by Poco graphite on irradiation, which may indicate that a new system of internal cracks was generated.

At very high neutron fluences, where graphite swells rapidly and new pores are generated, it is probable that the strength falls. However, no definitive data have been published.

The published data on the strength of irradiated graphite may be summarized as follows. The tensile and flexural strength increase with neutron fluence in a manner related to the increase in Young's modulus. For irradiation temperatures below about 500°C, the fractional increase in strength is proportional to the square root of the fractional increase in Young's modulus. This is equivalent to the strain energy to fracture remaining constant, provided that Young's modulus is measured in a static test to a high strain and not in a dynamic test. At higher irradiation temperatures, the strength increase for some isotropic graphites is somewhat higher than expected from the constant strain energy failure condition.

Neither the effect of irradiation on the scatter in the strength measurements nor the effect of testing temperature on the strength of irradiated graphite is well defined. The shear strength of graphite

increases on irradiation in about the same ratio as the tensile strength, while the compressive strength increases are higher. The strain to failure is reduced by irradiation.

3.6. STRENGTH OF GRAPHITE OXIDIZED UNDER IRRADIATION

A considerable amount of work has been reported on the effect of oxidation by carbon dioxide in a radiation field on the strength of reactor graphites (Refs. 76,77). The radiolytic dissociation of carbon dioxide by gamma radiation increases the overall corrosion rate, but causes the attack to be less localized than during oxidation in the absence of radiation. The rate of decrease in strength with burn-off is therefore lower than for out-of-reactor experiments. Reference 77 includes data on the strength of tensile specimens, diametrically compressed rings, and internally pressurized rings of isotropic graphite oxidized in flowing carbon dioxide in the BR-2 and DIDO reactors at 300° to 500°C. The direct effect of radiation damage on the mechanical properties was separated out by irradiating some companion specimens sealed in silica capsules and by annealing out the irradiation damage in some oxidized specimens before testing. The decrease in Young's modulus and strength with weight loss is shown in Figs. 37 and 38. The data show about a 20% reduction in Young's modulus and strength for 5% burn-off, compared with up to 50% reduction for 5% burn-off in out-of-reactor tests (see Section 2.12). There was no indication of different behavior for different graphite types, irradiation facilities, or test methods. It was concluded that the effects of irradiation damage and oxidation are separable, and oxidation effects may be allowed for by multiplying the expected strength of irradiated, unoxidized graphite by a factor obtained from plots such as Fig. 38. Later results (Ref. 72) suggest that strengths calculated in this way may underestimate the true strength.

No equivalent body of data relating to in-reactor oxidation by wet helium have been published in the open literature, but measurements on graphite irradiated in the Dragon reactor suggest that both the oxidation

Fig. 37. Effect of radiolytic oxidation by CO_2 on Young's modulus of Pile Grade A and isotropic graphites (from Ref. 77)

Fig. 38. Effect of radiolytic oxidation by CO_2 on the strength of isotropic graphites (from Ref. 77)

rate and the strength versus burn-off relation (Section 2.12) are unaffected by irradiation. It is noteworthy that the oxidation-induced fractional decrease in Young's modulus is sufficiently close to the decrease in strength that the elastic strain to failure remains almost unchanged.

4. RECOMMENDED DESIGN PRACTICE

4.1. BASIS FOR RECOMMENDATIONS

The recommendations presented in this section are based on a critical assessment of the literature combined with in-house data on the mechanical properties of near-isotropic graphites. Because many gaps still remain in the experimental results, it has been sometimes necessary to make "best-guess" judgments. Wherever significant uncertainty remains, a conservative approach has been taken. For example, Young's modulus of unirradiated graphite is known to increase with measurement temperature, but no experimental measurements at elevated temperatures have been made on irradiated material. Young's modulus of irradiated graphite has therefore been assumed to increase with temperature similarly to that of unirradiated graphite. On the other hand, the strength of unirradiated graphite increases with temperature somewhat faster than Young's modulus, but the data on irradiated material are inadequate to prove the existence of a similar increase with temperature. Therefore, no corresponding increase in strength with temperature has been assumed for irradiated graphite.

The largest remaining area of uncertainty surrounds the selection of failure criteria. There is no single model for failure which has been shown to apply to graphite under all conditions of loading. Even the Weibull statistical theory, which fits many aspects of graphite strength behavior, has not been demonstrated to provide a self-consistent model. The application of design criteria based on fracture mechanics considerations is not yet verified by experimental data from large graphite structures. In the absence of a universal and verified failure criterion, different failure criteria are recommended for different loading conditions, based on the results of tests under a similar stress state.

It should be noted that the term "failure criterion" refers here to the stress conditions which can be assumed to cause failure in a single element of material, such as a crack across the web between a fuel and coolant hole in an HTGR moderator block.

4.2. TYPE OF ELASTIC BEHAVIOR

For graphite irradiated under any conditions, Hookean behavior can be assumed.

For unirradiated graphite, the departure from linearity may be great enough to introduce important errors if linear elasticity is assumed. The following relationships between uniaxial stress, σ , and strain, ϵ , based on the work of Jenkins (Ref. 12), are recommended for unirradiated graphite:

$$\text{Initial loading curve: } \epsilon = A\sigma + B\sigma^2 ,$$

Unloading from peak stress, σ_m , and peak strain, ϵ_m :

$$\epsilon_m - \epsilon = A(\sigma_m - \sigma) + \frac{1}{2} B(\sigma_m - \sigma)^2 ,$$

Reloading from σ_o :

$$\epsilon - \epsilon_o = A(\sigma - \sigma_o) + \frac{1}{2} B(\sigma - \sigma_o)^2 .$$

It should be noted that ignoring the nonlinearity and assuming $A = 1/\text{Young's modulus}$ and $B = 0$ will generally result in overestimation of stresses and therefore errs on the side of conservatism.

4.3. ELASTIC CONSTANTS

Young's modulus under service conditions should be obtained by multiplying the appropriate room-temperature, unirradiated value, E_o , by a

factor accounting for the increase in modulus with temperature and a second factor giving the increase with irradiation. Thus,

$$E = E_0 (1 + 1.5 \times 10^{-4} T) F_1(\phi t, T_{irr}) ,$$

where T is the temperature in °C. In the absence of experimental data on irradiated graphite, the temperature dependence term was taken from the measurements of the dynamic modulus of unirradiated graphite by Mason and Knibbs (Ref. 78). These data were used in preference to the less-temperature-dependent results on the static modulus of unirradiated graphite (Ref. 24), because the static elastic modulus of irradiated graphite agrees with the dynamic modulus. Values for $F_1(\phi t, T_{irr})$ are shown in Fig. 39.

The values for $F_1(\phi t, T_{irr})$ were calculated from Dragon project dynamic modulus data (Ref. 59) on a pitch-coke graphite (code No. 100), whose properties and irradiation behavior resemble those of near-isotropic petroleum-coke graphites, by making the following assumptions:

1. Fluence in units of n/cm^2 (NDE) = 0.6 x fluence in units of n/cm^2 ($E > 0.18$ MeV).
2. Static Young's modulus of unirradiated graphite = 0.8 x dynamic Young's modulus.
3. Static Young's modulus of irradiated graphite = dynamic Young's modulus.

The curves calculated using these assumptions (Fig. 39) are in good agreement with measurements of the static Young's modulus of graphite irradiated in capsules OG-1 and OG-2 (Refs. 69,70). Values predicted from Fig. 39 are included in Table 1 for comparison with the measurements.

Fig. 39. Fractional increase in static Young's modulus as a function of fast neutron fluence (recommended for design purposes)

For conditions where oxidation by low-level impurities is significant, the value of Young's modulus should be reduced by multiplying by a factor $(1-WL)^{10}$, where WL is the fractional weight loss due to oxidation.

4.4. POISSON'S RATIO

In the absence of definitive data establishing a trend for the effect of irradiation on Poisson's ratio, it may be assumed that Poisson's ratio does not change on irradiation.

4.5. FAILURE CRITERIA

1. Predominantly uniform uniaxial tension or compression:

$$\sigma_{\max} = S_T \text{ (tension) } ,$$

$$\sigma_{\min} = S_C \text{ (compression) } ,$$

where $\sigma_{\max}$ and $\sigma_{\min}$ are the maximum and minimum principal stresses, and S_T and S_C are the ultimate tensile and compressive strengths, respectively.

2. Stress gradient progressing from tension to compression:

$$\sigma_{\max} = S_F ,$$

where S_F is the flexural strength.

3. Biaxial stresses: The effect of biaxial stresses is not fully defined at present. For interim calculations the use of Ely's maximum strain energy criterion is suggested:

$$\left(\frac{\sigma_1}{S_T}\right)^2 - 2\nu \left(\frac{\sigma_1}{S_T}\right)\left(\frac{\sigma_2}{S_T}\right) + \left(\frac{\sigma_2}{S_T}\right)^2 = 1 \text{ (tension-tension) } ,$$

$$\left(\frac{\sigma_1}{S_T}\right)^2 - 2\nu \left(\frac{\sigma_1}{S_T}\right) \left(\frac{\sigma_2}{S_C}\right) + \left(\frac{\sigma_2}{S_C}\right)^2 = 1 \text{ (tension-compression) } ,$$

where σ_1 and σ_2 are the principal stresses and ν is Poisson's ratio.

4.6. EFFECT OF IRRADIATION ON STRENGTH

The tensile, flexural, and compressive strengths may be assumed to increase on irradiation in proportion to the square root of the fractional increase in Young's modulus. Thus,

$$S = S_o F_2(\phi t, T_{irr}) ,$$

where $F_2(\phi t, T_{irr}) = \sqrt{E/E_o}$ and S_o is the unirradiated strength measured at room temperature. Values of $F_2(\phi t, T_{irr})$, calculated from the modulus increases shown in Fig. 39, are plotted in Fig. 40. Strength values predicted from Fig. 40 are included in Table 1 for comparison with measurements on specimens irradiated in capsules OG-1 and OG-2. For H-451 graphite and most H-327 specimens, the strength values predicted by Fig. 40 are conservative. However, there are not yet sufficient experimental measurements to justify the use of a strength-modulus relationship less conservative than that shown in Fig. 40.

When oxidation by low-level impurities in the coolant gas is significant, the strength values should be multiplied by $(1-WL)^{10}$, where WL is the fractional weight loss due to oxidation.

4.7. STATISTICAL VARIATION IN STRENGTH

Strength data reported in the literature have been expressed either in terms of the Weibull distribution parameter, m , or in terms of a standard deviation. There are insufficient data to establish the form of the distribution function. For calculational convenience it is recommended that

Fig. 40. Fractional increase in tensile or flexural strength as a function of fast neutron fluence (recommended for design purposes)

the strengths of individual samples be assumed to have a Gaussian distribution. Evidence on the effect of irradiation on the spread of strengths is not well defined. However, strength measurements on near-isotropic (H-451) graphite samples irradiated in capsules OG-1 and OG-2 (Table 1) showed no significant change in coefficient of variation from the unirradiated results. It is recommended that the coefficient of variation (standard deviation divided by the mean) be assumed to remain constant during irradiation.

4.8. NUMERICAL VALUES FOR UNIRRADIATED MECHANICAL PROPERTIES

Representative values for the mechanical properties of the unirradiated near-isotropic petroleum-coke graphites H-451, TS-1240, and SO-818 are shown in Table 5. Measurements are given for the midlength-center (weakest location) and midlength-edge regions of a "typical" log. The data in Table 5 are interim values based on a limited number of tests on prototype lots and may not be typical of production material.

TABLE 5
TYPICAL MECHANICAL PROPERTIES OF UNIRRADIATED NEAR-ISOTROPIC GRAPHITES ($\pm$ STANDARD DEVIATION)

Grade	Orientation	Location	Property (Mean Value $\pm$ 1 Standard Deviation)						
			Young's Modulus $\times 10^{-6}$ (a) (psi)	Poisson's Ratio	Tensile Strength (psi)	Compressive Strength (psi)	Flexural Strength ^(b) (psi)	Stress-Strain Curve A Parameter $\times 10^6$ (c) (psi ⁻¹)	Stress-Strain Curve B Parameter $\times 10^{10}$ (c) (psi ⁻²)
H-451	Axial	Midlength - center	1.17 $\pm$ 0.09	0.13 $\pm$ 0.01	2011 $\pm$ 211	8350	3690 $\pm$ 371	0.91 $\pm$ 0.07	1.8 $\pm$ 0.6
H-451	Axial	Midlength - edge	1.26 $\pm$ 0.09	0.12 $\pm$ 0.01	2686 $\pm$ 306	8300 $\pm$ 178	4332 $\pm$ 425	0.87 $\pm$ 0.07	1.1 $\pm$ 0.2
H-451	Radial	Midlength - center	0.98 $\pm$ 0.08	0.13 $\pm$ 0.01	1557 $\pm$ 293	7867 $\pm$ 153	3257 $\pm$ 414	1.04 $\pm$ 0.06	1.8 $\pm$ 0.6
H-451	Radial	Midlength - edge	1.03 $\pm$ 0.09	--	1964 $\pm$ 342	--	3706 $\pm$ 380	1.04 $\pm$ 0.06	1.4 $\pm$ 0.3
TS-1240	Axial	Midlength - center	1.14 $\pm$ 0.12	0.11 $\pm$ 0.01	2122 $\pm$ 364	7288 $\pm$ 132	--	1.02 $\pm$ 0.05	1.6 $\pm$ 0.1
TS-1240	Axial	Midlength - edge	1.17 $\pm$ 0.10	--	2301 $\pm$ 406	7808 $\pm$ 376	--	1.05 $\pm$ 0.10	1.7 $\pm$ 0.3
TS-1240	Radial	Midlength - center	1.02 $\pm$ 0.07	--	1572 $\pm$ 503	--	--	1.14 $\pm$ 0.04	1.7 $\pm$ 0.1
TS-1240	Radial	Midlength - edge	1.06 $\pm$ 0.06	--	1802 $\pm$ 354	--	--	1.12 $\pm$ 0.06	1.7 $\pm$ 0.3
SO-818	Axial	Midlength - center	1.09 $\pm$ 0.11	--	1980 $\pm$ 216	8303 $\pm$ 175	--	0.93 $\pm$ 0.05	1.3 $\pm$ 0.1
SO-818	Axial	Midlength - edge	1.16 $\pm$ 0.12	--	2267 $\pm$ 290	--	3603 $\pm$ 476	0.86 $\pm$ 0.04	1.6 $\pm$ 0.1
SO-818	Radial	Midlength - center	1.03 $\pm$ 0.07	--	1891 $\pm$ 206	8583 $\pm$ 306	--	0.89 $\pm$ 0.05	1.7 $\pm$ 0.1
SO-818	Radial	Midlength - edge	1.04 $\pm$ 0.11	--	1797 $\pm$ 203	--	2900 $\pm$ 398	0.96 $\pm$ 0.02	1.9 $\pm$ 0.2

(a) Measured for the 100- to 1000-psi portion of the reloading curve after loading to 1000 psi, unloading to 100 psi, and reloading to failure.

(b) Modulus of rupture, not corrected for nonlinearity of the stress-strain curve.

(c) $\epsilon = A\sigma + B\sigma^2$ (σ in psi).

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