

HOVE—PROPERTIES OF GRAPHITE AS AFFECTED BY HIGH TEMPERATURE 501
 SOME PHYSICAL PROPERTIES OF GRAPHITE AS AFFECTED BY
 HIGH TEMPERATURE AND IRRADIATION

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Introduction and review

In the general field of high-temperature solids, graphite has always occupied a very prominent position. Under certain conditions, mainly involving the absence of an oxidizing atmosphere, it is almost unique as a structural material for very-high-temperature use because of such important technological properties as its relatively high strength, ease of fabrication and forming and excellent resistance to thermal shock and thermal stress fracture.

A review of the mechanical strength of a homogeneous grade of graphite is shown on Fig. 1, which compares the short-time breaking stress of this sample (as obtained by Malmstrom, Keen & Green¹) with reported data of other materials which are normally considered suitable for high-temperature use. Above about 1500°, graphite is quite superior and if considered on a strength-to-weight basis is much superior to the other substances. In fairness, it should be pointed out that no reliable measurements have been published on the hard-metal class (carbides, nitrides, etc.), which may well be quite attractive at these temperatures. Although differing grades of graphite show considerably different values and orientation dependence of the strength, they all appear to have values which increase with temperature. This phenomenon occurs in some of the ceramic oxides as well as in graphite and can broadly be interpreted as due to the relief of internal stresses which are caused by anisotropic thermal expansion effects. Unlike other materials, however, graphite shows an appreciable increase in the elastic moduli with temperature and this cannot be explained so readily. There is some evidence to indicate that this increase depends primarily on the expansion of the single crystallites in the c-direction. Neutron irradiation causes both the Young's modulus and the c-spacing to increase and the modulus change per unit c-spacing change for the two entirely different influences (radiation damage and temperature increase) can be calculated and are found to be nearly the same. This calculation is somewhat uncertain because modulus and c-spacing studies have not been made on the same sample and possible alterations of irradiation conditions could cause an appreciable error, mainly because the modulus increases very rapidly with flux. It is possible, in a qualitative fashion, to formulate a simple model whereby thermal expansion closes up some of the void space and locks the crystallites together more tightly to cause the elastic modulus to increase. Additional effects must be present, such as the existence of locked-in stresses coupled with deformation processes which give rise to a modulus defect. Even at room temperature, the static stress-strain behaviour of graphite becomes quite non-linear at low stresses (see the Geneva paper by Currie, Hamister & MacPherson²) which implies appreciable plastic deformation. Studies at present under way of the modulus and internal friction as a function of frequency, temperature and strain amplitude indicate that all these variables are important.

An investigation of the tensile creep properties of graphite was also made by Malmstrom, Keen & Green¹ in the temperature range 1900 – 2900° and a variety of stresses. Applying standard rate theory to the results, they obtained an activation energy for the creep process of

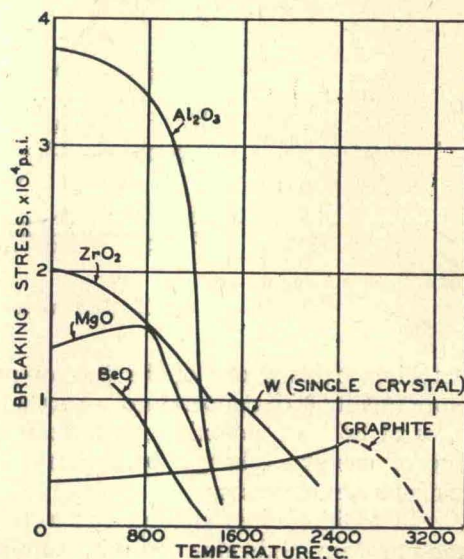


FIG. 1. Short-time breaking strength of various high-temperature materials

220 kcal. per mole. The self-diffusion activation energy for graphite has recently been found to be 170 kcal. per mole by the rather careful experiments of Kanter³ and this value can be shown to fit the creep data even better than 220 kcal. providing the highest temperature point (2900°) is not too much emphasized. The latter action is probably justified since the strain rate was determined for only two stresses at this temperature and it therefore seems fair to say that the creep process in graphite is limited by the self-diffusion mechanism.

An important aspect of the good resistance of graphite to thermal shock is its relatively high thermal conductivity at high temperatures. Fig. 2 compares the temperature dependence up to about 2000° (as obtained by Powell & Schofield⁴) with other materials (Kingery⁵).

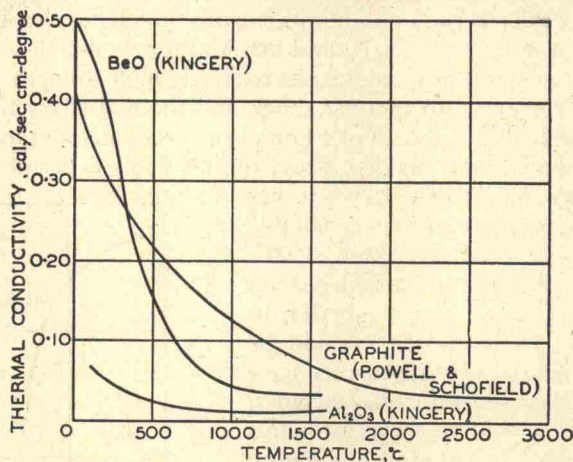


FIG. 2. Thermal conductivity of various high-temperature materials

Beryllium oxide is one of the few comparable substances, although its conductivity decreases more rapidly with temperature. This is probably to be expected since the three Debye temperatures of BeO are doubtless much closer together than in graphite which would imply a greater rate of energy exchange between the modes and hence a shorter relaxation time as the temperature increases.

In addition to its usefulness as a structural material, graphite has the advantage of being a good neutron moderator and this combination has led to a great deal of study of its properties under the conditions to be met in a nuclear reactor. Perhaps the condition of greatest concern, certainly the one which differs most from other applications, is the presence of high-energy particles (neutrons and perhaps fission products) which are capable of physically damaging the solid. For this reason, there have probably been more radiation-damage studies made of graphite than of any other single substance; these studies have had the important secondary effect of increasing our knowledge of the basic physics of graphite, although such an understanding is not helped by the great complexity of the various events which can occur in graphite as the result of nuclear bombardment. Good reviews of the present state of radiation damage studies are to be found in several of the papers presented at the Geneva Conference (1955).

Because of the high temperature (about 3600°) to which graphite remains solid, there have been very few studies made of the physical properties of the material over the entire range of potential usefulness. One of the purposes of the present paper is to review the results of a recently declassified study of the specific heat, thermal expansion, thermal conductivity and electrical conductivity up to the destruction temperature of graphite. This programme was under the direction of Dr. N. S. Rasor of Atomics International, who will publish the details of the work elsewhere. The data obtained in this investigation are of sufficient interest to call for further work to be done, especially along the line of improving the accuracy. Such work is planned but has had to be postponed due to the pressure of other studies. Another topic to be discussed concerns the phenomenon usually termed radiation annealing. This subject is of considerable importance to reactor engineers, even though the behaviour is understood only in a semi-empirical manner, and much of the high-temperature data have now been declassified.

High-temperature properties of graphite

The thermal expansion, specific heat, thermal conductivity and electrical resistivity have been measured in the temperature range from 1000° to about 3600° for several types of graphite. Actually, the electrical resistivity is obtained as a by-product from the specific heat measurements; its accuracy is not as high as could be obtained if it were measured directly, but the behaviour is correct to within an order of magnitude. It is not within the province of this paper to present experimental details, but the difficulties encountered in making measurements at these temperatures are severe enough to warrant a general discussion of the methods used and the problems encountered. In fact, here the validity of the final results depends perhaps more critically than usual on the validity of the experimental techniques.

Theory

Before entering into a discussion of the specific work, it may be of value to give a brief, qualitative review of the theory of these properties in graphite, in so far as it is known. The bulk thermal expansion is not normally a very striking property except perhaps in the low-temperature range where a number of materials (such as U, Pu, Si) actually show a negative volume expansion. Near the melting point, most solids seem to expand to about 2–3% over their room-temperature length. At moderate temperatures at least, graphite shows an anomaly even in this property. The difficulty is the quantitative one in that the bulk expansion is often much less than would be expected from the c-spacing expansion. This does not appear to result from any serious restraint of the c-axis expansion itself in a polycrystalline sample, and the correct explanation is still in some doubt. At very low temperatures (less than 4° K), the specific heat of various types of graphites forms a subject which is of great interest and which is presently under intensive study. Different types of graphite show different dependences on temperature and the interpretation is not yet firm. At somewhat higher temperatures, the specific heat depends on the square of the temperature (which can be explained as due to the two-dimensional nature of the lattice), and at still higher temperatures, follows a Debye behaviour, wherein an average Debye temperature of about 1300° K can be used. The thermal conduction in graphite is caused by lattice wave transport and, at low temperatures, is limited by boundary scattering. This causes the thermal conductivity to increase with temperature in a way similar (but not necessarily equal) to the specific heat. There are some interesting problems in this low-temperature range, but they have been considered elsewhere and will not be mentioned here (see published papers by A. W. Smith, N. Rasor and the author⁶ as well as the paper at this conference by J. C. Bowman). At high temperatures, the thermal transport is limited by exchange of energy between different modes of vibration, which are no longer purely harmonic. This causes the conductivity to decrease with temperature in a way varying from an exponential to a linear dependence on the inverse temperature. Thus, there is a maximum in the thermal conductivity which depends on the crystallite size and which occurs around room temperature for most commercial graphites. At this maximum, the value of the conductivity is high, rivalling most metals.

Experimental procedure

Two types of furnaces were used in making the high-temperature property determinations, a tube furnace for the thermal expansion, specific heat and electrical resistance and a radial-flow helix heater for the thermal conductivity. The tube furnace is illustrated in Fig. 3. Basically, it consists of a graphite tube heated by passing a current through it, which is supplied by a d.c. generator capable of 36 kw. To hold a steady temperature of 3300°, for example, required about 15 kw (1300 amp. at 12 v), although for rapid heating, powers up to 30 kw have been used. The heater consisted of a 30-in. long piece of 2-in. outside diam. and $\frac{1}{4}$ in. thick tube made from type AUF graphite. Three holes pierced the heater on each side for the insertion of sight tubes; external observations were made through Pyrex glass windows, which were mounted off-centre from the sight tubes to allow the windows to be rotated if fogging should occur, thus bringing clear glass back into the line of sight. A slow flow of inert gas was provided as shown, to prevent carbon (or volatile impurities) from migrating back through the sight tubes. The rather interesting phenomenon of a carbon 'snowstorm' was observed before the need

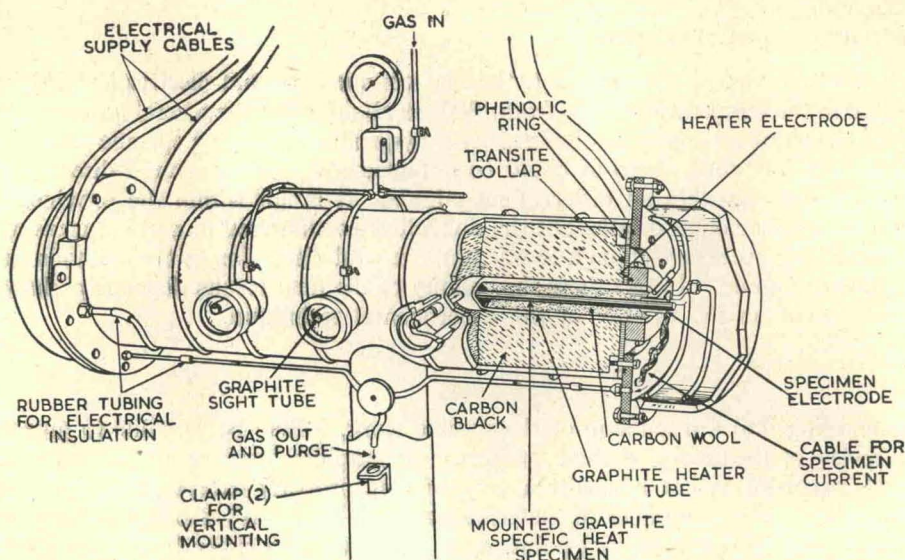


FIG. 3. Graphite tube furnace for specific heat and thermal expansion measurements

for such a gas flow system was realized. In this case, of course, any optical temperature determination would record only the precipitation (or 'dew point') temperature regardless of the true inside temperature. The inside of the furnace was maintained at an inert gas pressure of about 10 atm. to reduce the heater vaporization. In measuring the thermal expansion, samples $\frac{3}{8}$ in. in diam. by 11 in. long rested unconstrained on a carbon black layer spread on a graphite support. The latter rested in the heater tube, but made contact with it over a narrow region only. In practice, the motion of fiducial marks was simultaneously observed through two end sight-holes on one side of the furnace while end and centre temperatures were measured on the other side with optical pyrometers.

The specific heat specimens were $\frac{3}{8}$ in. diam. by 34 in. long, so that independent electrodes could be fastened to their ends, outside the heater tube. An independent power supply (in this case, storage batteries) with a fast relay switch was used to provide power for the sample. The process was then to bring the sample to temperature with the tube heater and pass a constant current pulse through it, measuring the initial rate of temperature rise at the bottom of a slot milled into the specimen and the voltage drop across that part of it containing the slot. By use of a photoelectric pyrometer, the temperature rise could be measured in a time (0.1 sec.) which is less than the minimum time needed for surface radiation effects to perturb the interior (0.2 sec. for this particular geometry and diffusivity). Since the rise time of the current is less than 0.01 sec., the specific heat of an essentially infinite body is thus obtained. Since both the current and voltage must be found to get the power input into the sample, the electrical resistivity is also found. An interesting, but troublesome, problem arose when it appeared that the specimen was shorting to the heater tube. The cause was found to lie in an excessively high thermionic current which was considerably greater than would have been expected with the high ambient pressure of argon gas. It happens that the electron scattering cross-section of argon has a Ramsauer-Townsend minimum at an electron energy which is just about the mean thermal energy at 3350°. Since helium does not show this Ramsauer effect at low energies, it was substituted for argon and the thermionic emission ceased to be a problem. There are not many experimental procedures for studies at high temperature which require a knowledge of quantum mechanics in order to make them work!

The furnace used for the thermal conductivity employed the radial flow method indicated on Fig. 4. Inward flow of heat was necessary since too much power would be required of a centre heater due to the large radiative area ratio. A graphite helical heater was used, which was heated with current from the d.c. generator. In addition, top and bottom guard rings were provided which used an additional a.c. power supply to minimize longitudinal temperature differences. Radial temperature drops were observed through the sight holes shown, which penetrated into the centre of the sample. The specimen had 2 in. diameter and length 3 in.;

below about 1000°, the thermal conductivity was so large and radiant heat transfer so small that thermocouple measurements were necessary to achieve reasonable accuracy. The heat flow was determined by measuring the flow rate and temperature rise of the heat sink cooling water. Great difficulties were found in attempting to use this apparatus above about 2800° because the heater power requirement rose sharply to intolerable values. This is the same behaviour and temperature observed in the specific heat tests mentioned previously and which was ascribed to thermionic emission. It is probably the same effect here (i.e. the helix heater is effectively being by-passed by the specimen), but the emitting surfaces are so large and have such a small separation that even though the use of helium rather than argon reduced the effect, it was not brought down to a tolerable limit. For this reason, a different method was used to measure the conductivity above 2800°, which employed a specific-heat-type sample in the tube furnace. After bringing the specimen to temperature, a steady current (using, in this case, a welding generator) is passed through it, creating a radial temperature drop which is measured by optically observing the temperature at the bottom of a radial hole drilled to the axis of the specimen and at the surface. This method is workable only at these high temperatures where radiant heat transfer is large and the thermal conductivity is small, since the optical surface temperature is not the true temperature due to the presence of the cooler water tube wall. This correction can only be made with acceptable accuracy when the surface and heater wall temperatures are nearly the same and furthermore this correction must be small compared to the radial temperature drop. For the conditions and sample used, it can be shown that these specifications are met satisfactorily above 3000° and thus the method is accurate at this temperature and higher.

The four types of graphite used are characterized in Table I. To avoid variations in density as much as possible, specimens were cut from the centre regions of the bulk forms. The thermal expansion was measured in two sample directions, but this was not practical for the thermal conductivity since cylindrical symmetry was essential; thermal conductivity results are therefore in the poor direction (perpendicular to the extrusion axis) for the extruded graphites and in the good direction (perpendicular to moulding axis) for the moulded graphite. As seen from Table I the anisotropy of these samples is not very great, so that this is not a serious consideration.

Table I
Graphite types

Manufacturer	Designation	Forming	Graphite types Anisotropy*	Density	Structure
Speer Carbon	3474D	extruded	1.08	1.67	Fine-grained
	7087	extruded	1.19	1.63	Coarse-grained with voids and fissures
National Carbon	GBH	moulded	0.78	1.77	Fine-grained
	GBE	extruded	1.18	1.57	Extremely coarse-grained. Voids up to $\frac{1}{8}$ in. diameter.

* As measured by electrical resistivity.

Thermal expansion

Fig. 5 shows the elongation with temperature for type 3474D in two directions. As expected, the expansion is greater in the case where more of the basal planes are oriented normal to the specimen axis. The cooling curve is different from the heating curve (this was observed in all the samples) presumably because the temperature has been raised above the original heat-treatment temperature and additional graphitization and impurity loss occurs. It is interesting to note that in all cases, this resulted in a permanent elongation and that further heating and cooling caused no further change. The type GBE results were similar although, surprisingly in view of the figures of Table I, they did not indicate as much anisotropy. Fig. 6 shows the somewhat different behaviour shown by type 7078, which is similar to that shown by GBH. The hump in the perpendicular heating curve (ending at about 3300°) is presumed to be due to the presence of impurities, which are driven out by the time 3300° is reached. The cooling curve is normal, as is the expansion in the parallel direction. This particular type of graphite evolved a

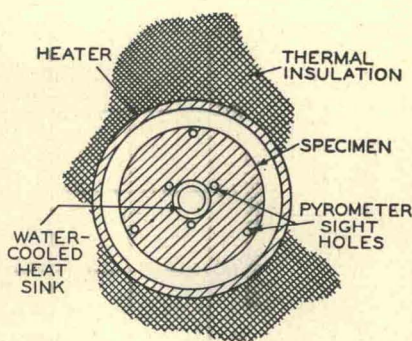


FIG. 4. Radial flow method used in thermal conductivity furnace

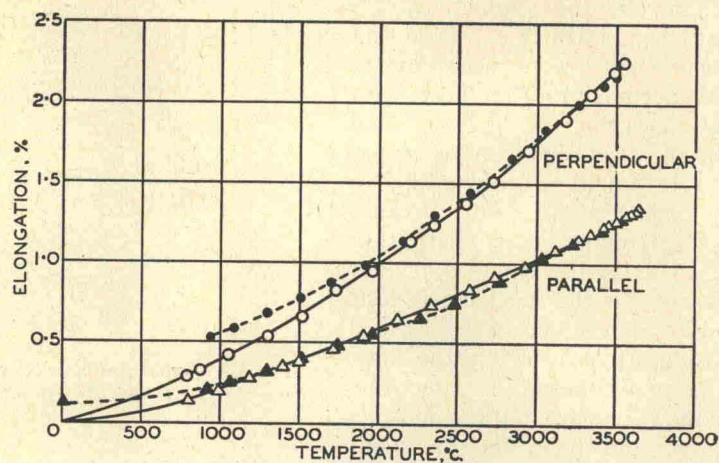


FIG. 5. Thermal expansion for type 3474D graphite

— heating
- - - cooling

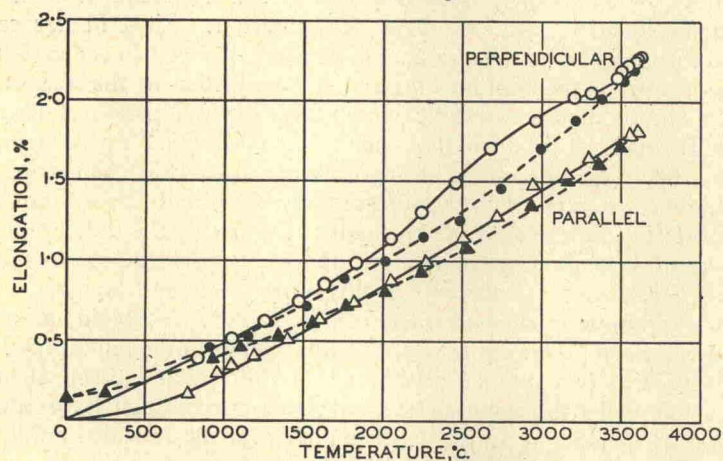


FIG. 6. Thermal expansion for type 7087 graphite

— heating
- - - cooling (further heating and cooling follows this curve)

considerable amount of impurity vapour which condensed as a white deposit. Furthermore it showed a reduction of 0.23% in the c-spacing after being heated; this is of the same order of magnitude as the difference between the heating and cooling curves of Fig. 6.

Specific heat

Fig. 7 includes all the specific heat data obtained. Up to about 3300°, the Dulong-Petit value is observed with very slight deviation. The behaviour above this temperature is a quite

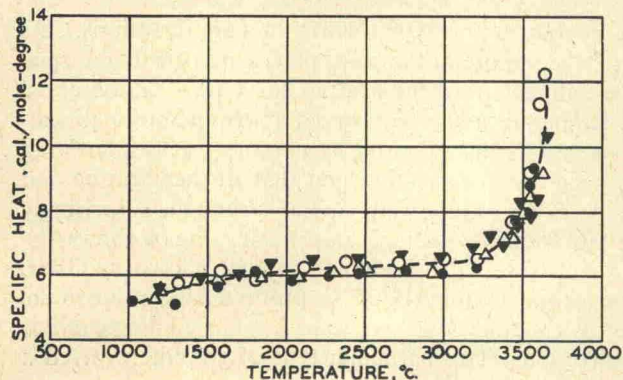


FIG. 7. Specific heat of all graphite specimens

△ 3474D
▼ 7087
× GBH
○ GBE

well-defined exponential and is reproducible. The activation energy associated with this is 7 ev, probably ± 0.3 ev. In principle this could be either vapourization or vacancy formation, since both have activation energies of this order. An analysis of the transient carbon diffusion from the surface indicates, however, that enough heat cannot be removed in this way to account for the large change. Furthermore, by assuming vacancy formation takes 7 ev, the data on Fig. 7 give a concentration of 0.4 at.-% vacancies at 3660°. This is about equal to values obtained in the cases of Na, Pb and Al, all of which show similar specific heat behaviour, and is about that to be expected to make the solid phase unstable. The only serious difficulty in taking this to be vacancy formation is the result of Kanter which gives the self-diffusion energy in graphite as 7ev also. Thus the vacancy migration energy would be very small, a rather startling result. There does not appear to be an answer to this anomaly at the present time.

Thermal conductivity

A composite graph of several types of measurements of the thermal conductivity on type 3474D graphite is shown on Fig. 8. It may be noted that in the region of overlap, the external heating and direct heating methods agree quite well, indicating a satisfactory check on the validity of the methods. Also included are values obtained by Euler⁷ in the high-temperature range, normalized to the present results at 3350°. He obtained these data by a very ingenious method involving stereoscopic examination of pits in a carbon arc electrode base to determine their depth and measuring optical temperatures for a number of depths. It is remarkable how closely his data parallel the present results. The interesting feature of Fig. 8 is the strong decrease at about the same temperature as observed for the sharp increase in specific heat and again this is an exponential decrease with an activation energy of 7ev. As before, it is conceivable that this could be a vapourization effect, but here the effect would be such as to make the apparent radial temperature drop too small and hence make the thermal conductivity appear to increase, not decrease. Thus this is most reasonably interpreted as a vacancy formation which causes additional lattice wave scattering.

It is of interest to note that the sample 7087, which gave a non-reversible change in the thermal expansion, behaves similarly as regards the thermal conductivity, i.e. on initial heating the value was low and almost independent of temperature (even showing a slight increase), but on later heating reproduced the behaviour of Fig. 8 quite satisfactorily.

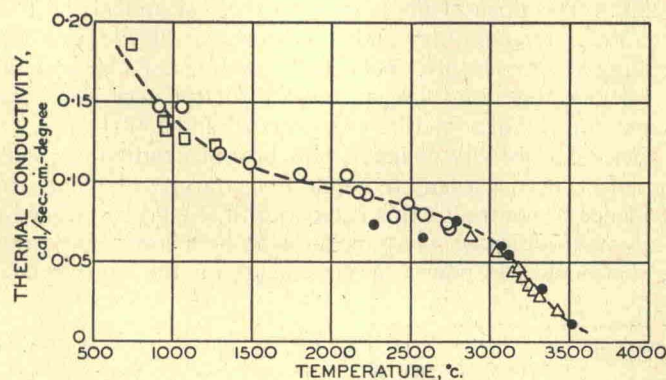


FIG. 8. Thermal conductivity of type 3474D graphite

- thermocouple
- pyrometer
- direct heating
- △ Euler (normalized by a factor of 1.46)

Electrical resistivity

The behaviour of the electrical resistivity as estimated from the data obtained for the specific heat measurements is shown on Fig. 9. Note that not only does it not show the exponential increase of the thermal resistivity, but it actually decreases slightly. This is to be considered a preliminary measurement, but is believed correct as far as the order of magnitude is concerned. The implication is that the electrons at this temperature are not scattered appreciably by the vacancies and that some other effect predominates to increase their conductivity. This is a difficult theoretical consideration, since it is likely that the usual simplified

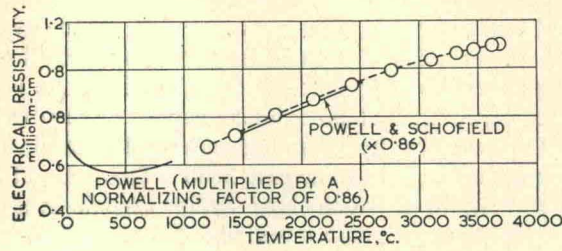


FIG. 9. Electrical resistivity of graphite

band model (i.e., that only energies very near the corner of the Brillouin zone are important) is not valid. Thus, there is no interpretation at present of this behaviour ; since it is intended to remeasure the data, especially to check on the resistivity saturation, it is not especially worth while to speculate on the explanation at this time.

Radiation annealing effects in graphite

It has long been recognized by reactor engineers and physicists that the graphite radiation-damage problem at high temperatures is much less severe than would be indicated by irradiation at a lower temperature followed by thermal annealing at the high temperature. This phenomenon has been called radiation annealing and its effects can be strikingly large. As an example Fig. 10 shows the thermal conductivity, measured at room temperature, as a function of irradiation at various temperatures. As a comparison, the values obtained by thermally annealing samples irradiated at room temperature are also given. The unit of megawatt-day (MWD) is approximately equivalent to an integrated flux of 2×10^{17} fast neutrons per cm^2 . As can be seen, a fairly high irradiation at about 400° produces a change in the thermal conductivity which is less by a factor of ten or more than would be expected from the thermal annealing effect alone.

It seems well established that radiation annealing does not take place at room temperature at low amounts of radiation damage. Thus, Fig. 11 shows the electrical resistivity changes produced in a sample of graphite by cyclotron (proton) irradiations made at temperatures from liquid nitrogen to 250° . The open symbols refer to thermal anneals at room temperature of samples irradiated at lower temperatures and it is seen that at room temperature, the values measured after annealing at temperatures from 103°C and up coincide with the direct irradiation values. At 423°K (150°C), this is no longer true, except for very low fluxes and a radiation annealing effect become important somewhere between 0° and 150°C . Fig. 12 shows the similar dependence of the thermal resistivity changes with proton irradiations and it appears that a radiation annealing effect occurs even at room temperature for high irradiations. While there is no actual evidence to contradict this behaviour, it should be pointed out that the single high-exposure measurement indicated what appeared to be a somewhat erratic annealing spectrum; furthermore, thermoelectric power measurements on the same specimen showed signs

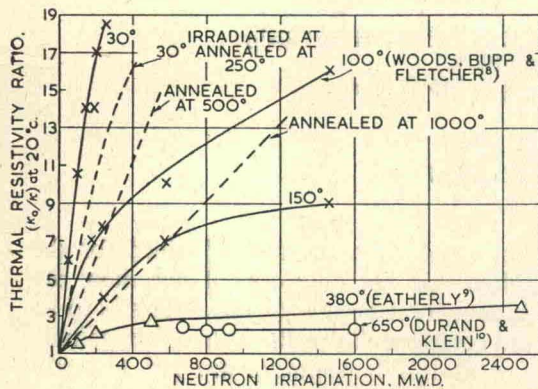


FIG. 10. Thermal conductivity as a function of neutron irradiations at various low temperatures

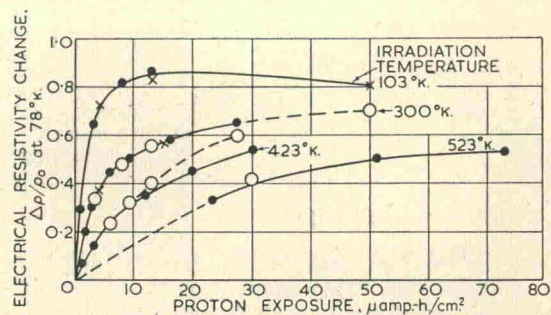


FIG. 11. Electrical resistivity as a function of proton irradiations at various low temperatures
Open symbols represent data from thermally annealed specimens
x neutron

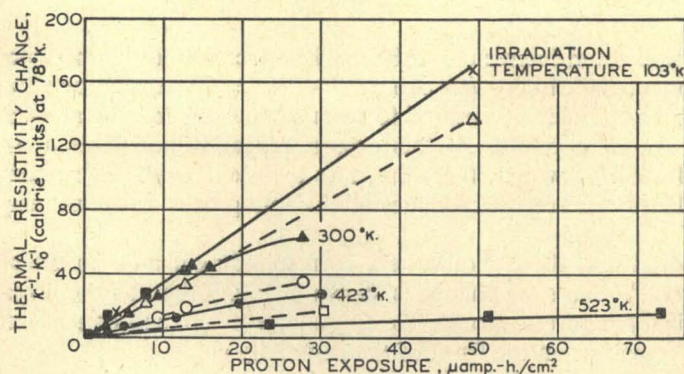


FIG. 12. Thermal resistivity as a function of proton irradiations at various low temperatures
 Open symbols represent data from thermally annealed specimens
 × neutron
 (Note: $\kappa_0^{-1} = 20$ sec.-cm.-deg./calorie at 78°K)

of excess experimental errors of some type. Therefore, while this evidence of radiation annealing at room temperature may be valid, it must be regarded with a certain amount of suspicion. On the other hand, it should also be pointed out that the bombardments on Figs. 11 and 12 are equivalent to neutron damages of less than 100 MWD and it is entirely possible that these fluxes are not high enough to permit radiation annealing to become apparent.

There are a number of qualitative explanations of the effect mentioned above, all of which probably have some degree of validity. In general, radiation damage consists of various numbers and types of defects which exist in the lattice as a result of carbon atoms being displaced from their lattice positions by the neutron flux or its by-products. These defects migrate, combine together and generally show a kinetic reaction behaviour which depends on their temperature, species and location. It is clear that all of these factors are different in the presence of the radiation field than when isolated from this field. Thus a knock-on atom possesses a much higher kinetic energy than do the lattice atoms. Thus excess energy results in creating local regions of high kinetic temperature, either by exciting lattice vibrations or by causing secondary displacements with high kinetic energy. This excitation will affect the various reaction rates. In addition there will be direct radiation-induced back reactions; an example might be the dissociation of an interstitial complex into single atoms or ions which, if the temperature is high enough, will migrate together in a different way or perhaps reintegrate with lattice vacancies completely to annihilate some of the defects. This back-reaction is enhanced by the fact that the neutron energy required to dissociate such a complex is probably considerably less than the energy required to displace a lattice atom. Thus very much more of the neutron flux present is available for such dissociation than for lattice displacement. Such an effect would become stronger as the number of defects increases, which is in qualitative agreement with the observations. While the above discussion cannot be called a theory of radiation annealing, it does serve to point out the complexity of this useful effect and the need for more specific experiments before a quantitative interpretation can be formulated.

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Discussion

Dr. R. W. Powell: I am interested in the marked decrease in thermal conductivity reported at temperatures above 3000°. In my own paper (p. 46), when discussing the abrupt fall by an order of magnitude reported by Euler, I ventured to surmise that this may be associated with a density reduction due to loss of graphite. If this occurs, repeat values made at lower temperatures should also be affected. I would, therefore, like to ask if any such repeat values have been obtained. Even density or resistivity values after cooling back to room temperature would be of interest.

I also anticipated that there should be a corresponding change in the electrical resistivity on heating above 3000°. No such change is shown in Fig. 9, but, as these results were referred to as of a preliminary nature, I would like to enquire if any subsequent determinations have provided more information on the form of the resistivity curve.

Dr. J. E. Hove: I do not believe that the thermal conductivity change at high temperatures can be attributed to density changes. Note that the thermal expansion curves are perfectly smooth and reproducible throughout this temperature region; this would presumably indicate that there are no gross changes in internal microstructure or pore size. In addition, we have just measured the bulk density of a sample, which had been heated to 3600°, and found that it had a value of 1.65 compared with a before-test value of 1.67 on a similar specimen. This variation is well within the probable difference between the two graphite samples. There is, of course, little or no change in the visual appearance of the graphite before and after heating.