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Radiation Effects on Materials
(TID-4500, 13th Ed.)

RADIATION DAMAGE TO GRAPHITE FROM 30 C TO 185 C

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RADIATION DAMAGE TO GRAPHITE FROM 30 C TO 185 C

INTRODUCTION

The effect of reactor irradiations on polycrystalline graphite at room temperature or slightly higher has been thoroughly studied.[1, 2] Changes in properties with neutron exposure are now reasonably predictable. Although the fundamental processes are far from being well understood, considerable progress has been made in this area, and a description of radiation damage in graphite has been developed.[1] The effect of irradiating graphite at different temperatures is much less well known. With the current trend toward higher reactor temperatures it is important to know how graphite will behave under these conditions. Furthermore, a knowledge of the effects of irradiating graphite at both high and low temperatures will aid in developing a more detailed description of radiation damage. This study was undertaken to learn more about the behavior of graphite above 30 C with the hope that the results would contribute to the understanding of radiation damage and yield data of engineering value.

SUMMARY AND CONCLUSIONS

Property changes in polycrystalline graphite resulting from reactor irradiations at temperatures up to 185 C and over a range of exposures up to 1135 MD/CT have been determined. Changes in stored energy, thermal conductivity, sample length, and C_o interlayer crystallite spacing are markedly decreased as exposure temperature is increased. Electrical resistivity changes are also less at higher exposure temperatures, but this property change does not depend as strongly on temperature as the others.

Isothermal annealing studies of C_o changes were conducted on a number of irradiated samples. The data were analyzed assuming a large number of processes distributed in activation energy. The results are summarized in activation energy spectra in which the distribution of C_o

damage is given as a function of the activation energy required for annealing. It is found that not only does a higher exposure temperature decrease the total amount of property change, but also the distribution of damage accumulated is considerably different.

Results are discussed in terms of the radiation damage model suggested by Hennig and Hove.[1] This model is compatible with the experimental results presented.

EXPERIMENTAL

The samples studied in these experiments were standard grade (CSF) graphite in the form of right circular cylinders 0.426 inches in diameter and nominal lengths of three inches. The cylinders were "transverse cut" from graphite bar stock in such a manner that the preferred orientation of the C_o axis coincided with the axis of the cylinder; i.e., the platelets in the graphite crystallites tended to be "stacked" parallel to the ends of the cylinder.

Prior to irradiation the lengths of the samples were measured with a vernier micrometer. Thermal and electrical conductivities were determined by the Kohlrausch method[3] and the lattice C_o spacings were determined by X-ray diffractometry.

Two graphite samples were sealed in an aluminum can one-half inch in diameter. Several such cans were irradiated in assemblies like that shown in Figure 1. Each sample can was inserted in a tubular wire-wound heater. Four of these heater and sample assemblies were mounted in a jointed aluminum housing. The completed assembly was then irradiated in an "annulus tube" facility (Figure 1) consisting basically of two concentric tubes extending through the reactor. The experimental assembly was inserted in a dry central tube, and cooling water flowed in the annulus between the two tubes.

Sample temperatures during irradiation were controlled by varying the electrical power supplied to the heaters through the control system

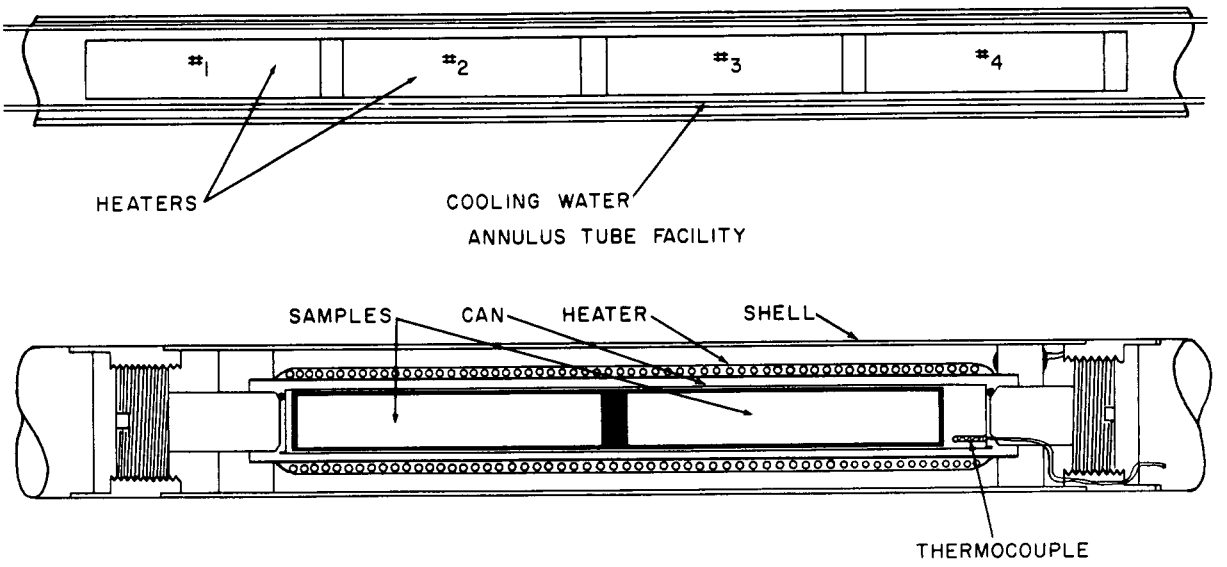

FIGURE 1

Sample and Heater Details of the Experimental Facility

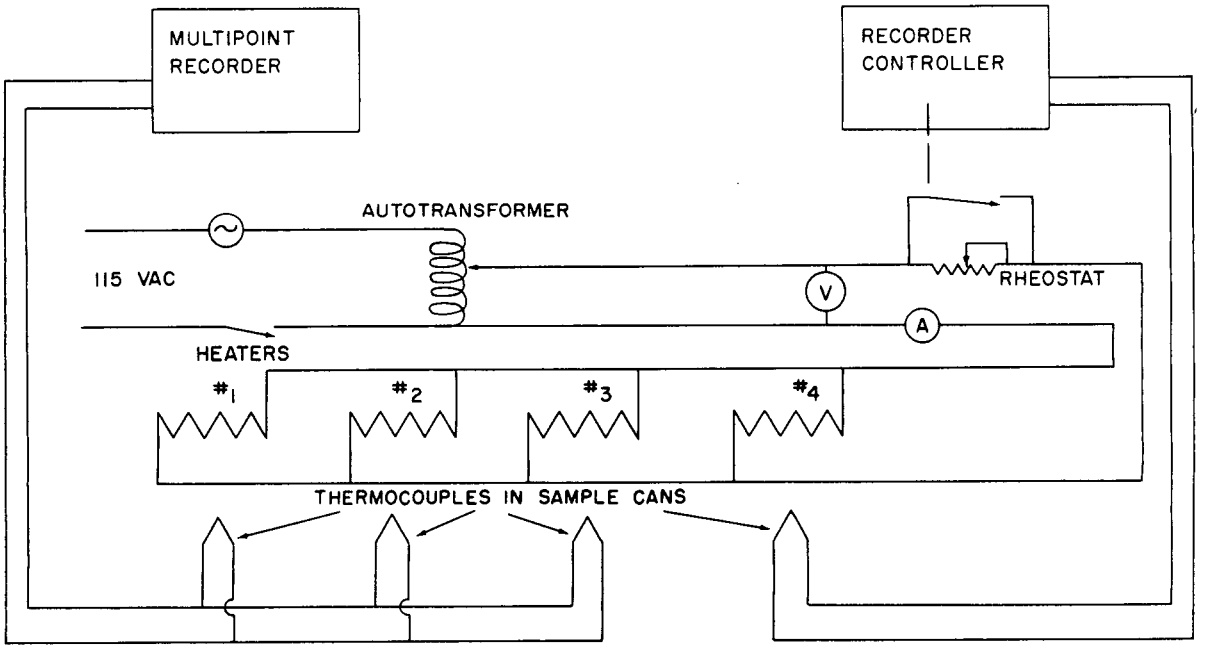

FIGURE 2

Temperature Control System

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shown schematically in Figure 2. Each of the four heaters installed in a given assembly was wound with a different, predetermined electrical resistance. The four heaters were connected in parallel and were supplied with electrical power through a Variac autotransformer. A rheostat connected in series with the heaters attenuated the voltage applied to the heaters. A shunt around the rheostat was opened and closed by the controller, giving a "high-low" modulation of heater power. The "high" and "low" heater powers could be adjusted by varying settings of the Variac and rheostat. Three separate irradiations of the above type were performed to produce the data discussed herein.

IRRADIATION HISTORY

Irradiation histories of the graphite samples are summarized in Table I.[4] The sample exposures are given in terms of "megawatt days per central ton" ($\frac{MWD}{CT}$). This unit is defined as the amount of neutron radiation received by the sample during the time required for a ton of uranium in the central region of a reactor to generate one megawatt-day of fission energy. The exposure figures shown have been normalized to give the equivalent exposures which the samples would have received, during the same periods, in a "standard" test hole position. One $\frac{MWD}{CT}$, as given, is equivalent to an integrated neutron flux of approximately 6.5×10^{17} neutrons per square centimeter. Absolute values of the sample exposures are accurate within ± 20 per cent.[5]

The exposure temperatures shown are mean values over the course of the irradiation. In the case of the "control" samples, whose temperatures were used to control heater powers, the uncertainty in temperature is less than ± 5 C. Temperatures of the other samples, not being directly controlled, were sensitive to small differences in heat leakage rates and to external influences. The temperatures of these samples varied considerably more during irradiation. However, the mean temperature values as shown for all samples are accurate within ± 15 C.

TABLE I[4]

SAMPLE IRRADIATION HISTORY

Sample No.	Exposure $\frac{MWD}{CT}$ (± 20 %)	Temp. °C (± 15 C)
93-95) 93-16)	193	111
93-23) 93-14)	193	139
93-17) 93-18)	193	164
93-99) 93-139)	464	127
93-42) 93-39)	464	158
93-62) 93-61)	464	185
96-22) 96-97)	1135	135
96-84) 96 112)	1135	137
96-29) 96-60)	1135	123
96-14) 96 92)	1135	118

PROPERTY CHANGES

A large number of property changes have been measured on graphite irradiated at room temperature. These include[1, 2] crystal size and unit cell dimensions, stored energy, electrical resistivity, thermal conductivity, sample length, elastic modulus, Hall coefficient, susceptibility, magneto-resistance, thermoelectric power, paramagnetic resonance, neutron transmission, and chemical properties. Since any radiation damage model must be compatible with the observed property changes, it is important to determine radiation effects on as many properties as possible. Certain properties may be more useful in this respect due to a better fundamental understanding of a property change or the availability of a sensitive experimental tool. In addition, a few property changes are of considerable practical interest in the construction and operation of reactors containing graphite. We have measured four properties in this study: stored energy (SE), length (L), thermal conductivity (K) and interlayer spacing (C_o). Electrical resistivity measurements were also made but it was found that this property does not depend strongly on irradiation temperature. The slight decrease in damage at higher temperatures was not much greater than the experimental error of measurement and so this property has not been included here.

As a result of reactor irradiation the energy content of graphite increases. The difference between the energy content of a perfectly crystalline sample and a sample containing crystalline defects is termed stored energy. It may be measured through conventional heat of combustion measurements from which total stored energy is obtained, or as an apparent decrease in the specific heat from which the stored energy release rate may be obtained as a function of annealing temperature. The total stored energy as a function of irradiation temperature for 193 MD/CT and 464 MD/CT samples is given in Figure 3. The stored energy decreases very markedly as exposure temperature is increased from 30 C to 185 C.

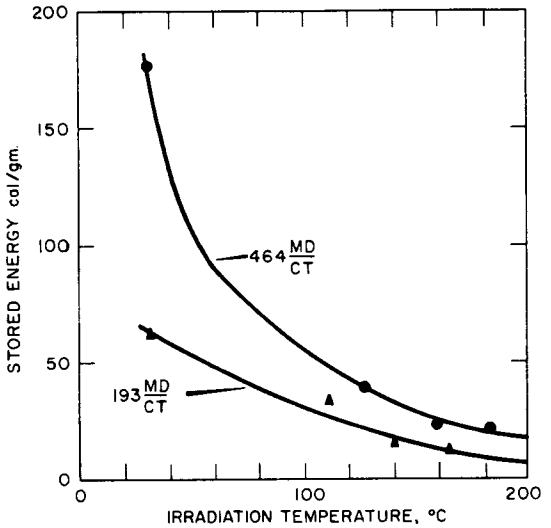

FIGURE 3

Changes in Stored Energy
With Irradiation Temperature

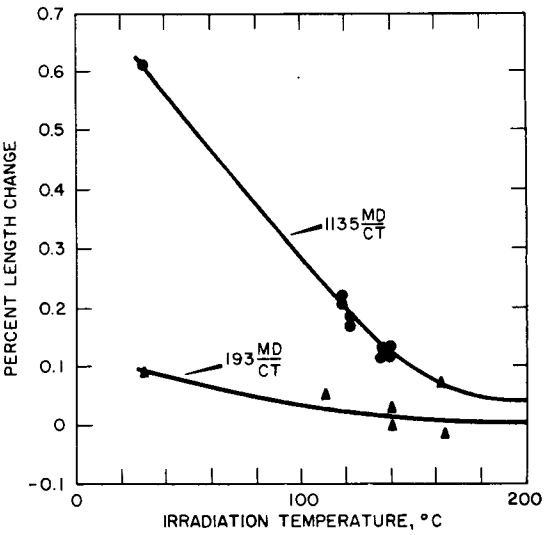

FIGURE 4

Changes in Length
With Irradiation Temperature

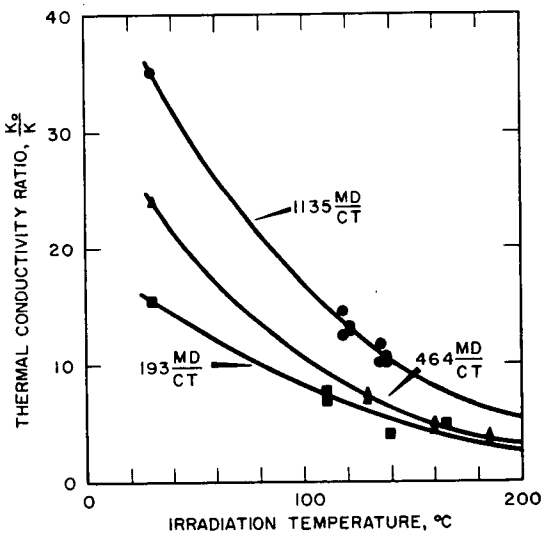

FIGURE 5

Changes in Thermal Conductivity
Ratio with Irradiation Temperature

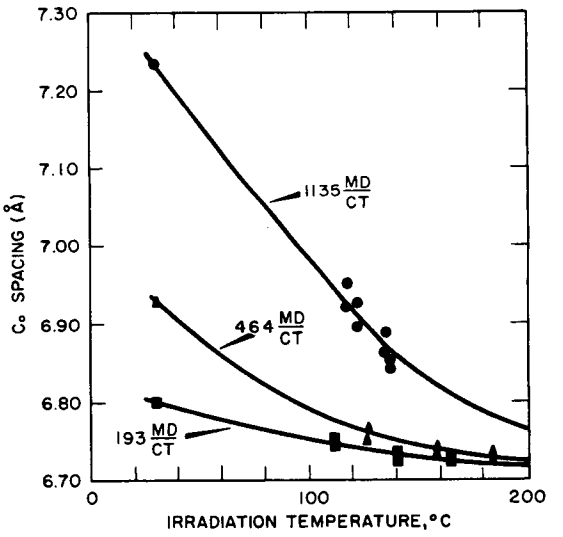

FIGURE 6

Changes in C_o Spacing with
Irradiation Temperature

The dimensional stability of graphite exposed at 30 C has been determined as a function of neutron exposure.[2] In general, artificial graphite cut transverse to the extrusion axis expands in a reactor at room temperature while parallel cut graphite contracts slightly. The more highly crystalline the sample, the more pronounced is the expansion of transverse samples. Figure 4 shows the effect of exposure temperature on length changes for transverse samples exposed to 193 MD/CT and 1135 MD/CT. An error was made in measuring the original lengths of samples in the 464 MD/CT series and these results were discarded. Length changes are considerably reduced at the higher exposure temperatures.

The thermal conductivity of graphite is decreased by reactor irradiation. The ratio of initial to final conductivity, $\frac{K_o}{K}$, as determined by the Kohlrausch method,[3] has been plotted in Figure 5 for samples with 193, 464, and 1135 MD/CT exposure. K_o values varied from 2.10 to 2.65 with an average of 2.37 cal/cm sec ^oC.

Irradiation damage to graphite causes an increase in the C_o spacing, the distance between alternate carbon layer planes within the crystal. These changes, calculated from the shift of the 002 X-ray reflection peak, are shown in Figure 6. The pre-irradiation C_o spacing for these samples was 6.70 Å. This increased to 7.23 Å after an exposure of 1135 MD/CT at 30 C. At 185 C the increase is only to 6.78 Å.

LATTICE SPACING ANNEALING

Radiation damage in graphite may be reduced by thermal annealing. It has been found that for samples exposed at about 30 C, damage is removed beginning at about 75 C and continuing to very high temperatures, even approaching graphitization temperatures of 2500 to 3000 C. Attempts have been made to extract fundamental information from the annealing kinetics which would aid in developing a model for radiation damage in graphite. A number of fundamental quantities govern the rate of annealing of a particular property but in most cases the number of experimental quantities determined

is not sufficient to obtain accurate values for any of these. One approach which seems to be most useful is that used by Vand[6] in which the concept or a large number of processes distributed in activation energies was introduced. Neubert[7] later applied this idea to annealing of radiation damaged graphite. Primak[8] has recently generalized the Vand treatment and a brief review of this work is necessary to discuss the graphite annealing experiments.

By measuring the rate of change $\frac{dP(t)}{dt}$ of some physical property, P(t), during an anneal, a function, $p_o(E)$, may be calculated which shows the way damage is distributed among the activation energies. A plot of $p_o(E)$ vs E is termed the activation energy spectrum. The equation governing the isothermal annealing process is

$$P(t) = \int_o^\infty p_o(E)\ \theta_n(E,t)\, dE$$

where $p_o(E) = f q_o(E)$

$$\theta_n(E,t) = \left[1 - (1-n)Bt \exp(-E/RT)\right]^{\frac{1}{1-n}}$$

in which $B = A(f/p_o)^{1-n}$

q_o = initial concentration of possible kinetic processes
f = change in the property accompanying one kinetic process
n = kinetic order
A = collisional constant of the Arrhenius equation
E = the activation energy
R = gas constant
T = annealing temperature.

If the activation energy spectrum extends over a range many times nRT, $\theta(E,t)$, the characteristic annealing function, may be approximated by a step function. Also, if the annealing behavior is dominated by the strong dependence of $\theta(E,t)$ on E, the dependence on B and f may be treated as a second order effect. For isothermal annealing under these conditions,

$$P(t) \simeq \int_{E_o}^{\infty} p_o(E_o)\, dE$$

where $E_o = RT \ln Bt$

and $p_o(E_o) = \frac{t}{RT} \frac{dP(t)}{dt}$

The effect of making the mathematical approximation to $\theta_n(E,t)$ is to decrease the "resolution" of the activation energy spectrum so that any sharp peaks will be broadened out to a width the order of nRT. This is still quite satisfactory when dealing with a distribution over a range many times this. In order to use the above equations, B must be known or estimated. If B is incorrectly chosen the parts of the activation energy spectrum will not fit together and this may serve as a test for an estimated value of B.

Figure 7 gives an activation energy spectrum obtained for a series of isothermal anneals of C_o spacing damage on a sample exposed to 556 MD/CT at about 30 C.[9] Dotted lines are drawn through the experimental points for a single isothermal anneal. The envelope of these dotted lines shows the way C_o damage was distributed among activation energies before any annealing was done. Kinetic processes involving the total range of activation energies studied, from 26 to 80 kcal/g-atom, contributed to the observed annealing rate. It is clear that there is a sharp peak at about 33 kcal/g-atom, but the height is not well defined. This peak is also found in spectra from other property changes.[7]

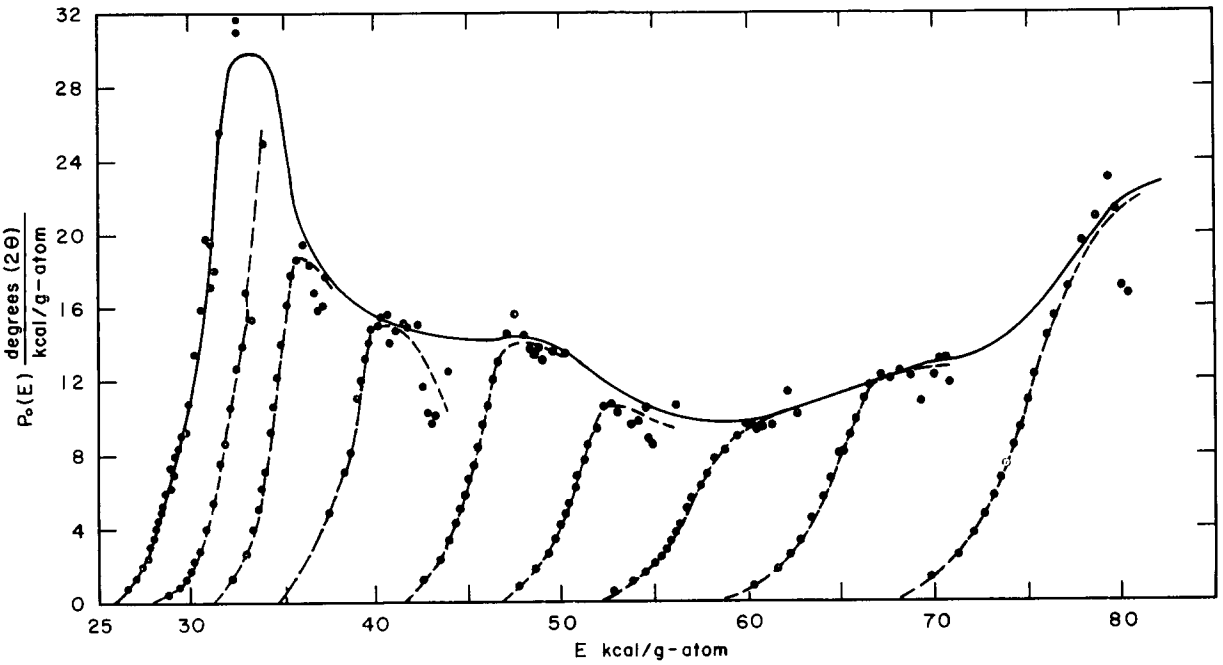

FIGURE 7

Activation Energy Spectrum - 556 $\frac{MD}{CT}$ at 30 C

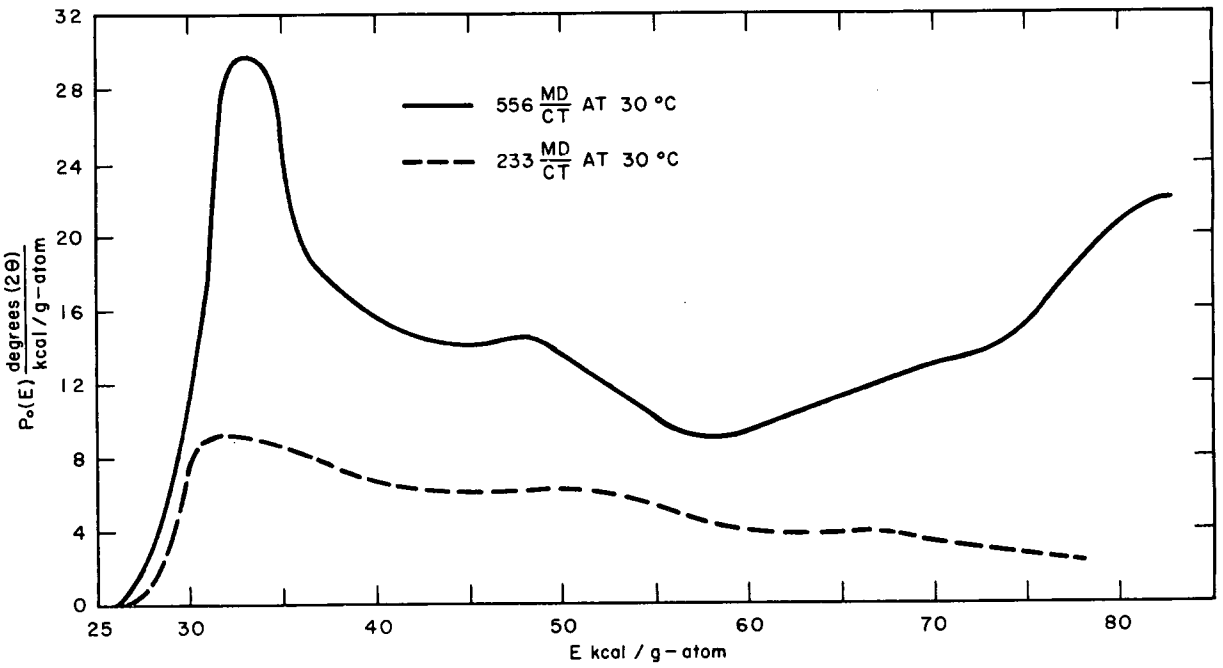

FIGURE 8

Activation Energy Spectrum-556 $\frac{MD}{CT}$ and 233 $\frac{MD}{CT}$ at 30 C

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A constant value of B = 7.5×10^{13} sec^{-1} was used and seems to give satisfactory fit. However, this method of determining B is not particularly sensitive and requires precise experimental data to be useful. For this reason a somewhat pessimistic approach was taken when drawing the envelope of the annealing curves and the "fine structure" was averaged out. The extent to which this was done was also governed by the reproducibility of spectra on duplicate samples and an estimate of the resolution of this method. It is believed that the general features of the spectra as shown are real, although the true shape of some peaks may be considerably sharper than shown because of the limited resolution.

The amount of property annealed isothermally after any time t may be calculated from

$$P(t) = \int_o^{E_o} \frac{dP(t)}{dt} \, dE_o = \frac{RT}{t} \int_o^{E_o} p_o(E_o) \, dE_o$$

which may be obtained from the area under the activation energy spectrum out to E_o. Annealing of damage may be considered to occur by the advance of the characteristic annealing function as it moves across the activation energy spectrum from left to right, sweeping out the damage behind it.

If the rate data and the assumptions made in calculating the activation energy spectrum are sufficiently accurate, it should be possible to obtain other parameters (such as the order, n,) which more accurately describe the annealing process. No attempt has been made to calculate these other parameters since it was felt that the method of determining B, when combined with uncertainties in the experimental data, is not sufficiently sensitive to warrant this.

Aside from the value of activation energy spectra and any fundamental parameters in giving information concerning the annealing mechanism, activation energy spectra give a very convenient method of describing the

damaged state. One method which has been commonly used in the past has been to plot damage removed vs annealing temperature. However, different experiments are comparable only if the annealing times are the same and so are of limited value. Activation energy plots should be directly comparable, within the limitations of the Vand method, since the time factor is included. For example, the same amount of damage should be removed by annealing at T_1 °C for t_1 seconds as is annealed at T_2 °C for t_2 seconds when the temperature and time are chosen so as to give the same E_o. This particular result of the Vand theory has not been sufficiently tested, but in the few cases for which we have data the theory predicts this characteristic of the annealing behavior very satisfactorily. If future studies confirm this point, this will offer a convenient method of predicting the amount of radiation damage annealed under a wide range of temperature-time conditions.

DISCUSSION

The description of radiation damage in graphite by Hennig and Hove[1] is the most detailed one yet suggested. Their model was developed from property changes occurring between -190 C and 30 C. They discussed several possible processes, some of which are represented by the following set:

$$\text{Graphite} \xrightarrow[\text{flux}]{\text{neutron}} C^- \qquad (1)$$

$$C^- + \text{vacancy} \longrightarrow \text{Graphite} \qquad (2)$$

$$2C^- \longrightarrow C_2 + 2e^- \qquad (3)$$

$$C_2 + \text{vacancy} \longrightarrow \text{Graphite} \qquad (4)$$

$$C + C_n \longrightarrow C_{n+1} \qquad (5)$$

The paramagnetic and other properties of irradiated graphite suggest that after a carbon atom slows down to near the average kinetic energy of the lattice it picks up an electron from the conduction band to become a carbon ion, C^-, as represented by (1). In the temperature range 100 to 200 C these reintegrate with the lattice at a rate dependent upon the temperature (2). Carbon ions may also combine to form C_2 molecules (3); the activation energy for which is apparently such that it occurs concurrently with (2). Reintegration of C_2 molecules (4) may also occur with a somewhat higher activation energy. At high neutron exposures more complex damage centers are formed, as represented by (5), which are progressively more difficult to anneal. As the damage builds up the path along which interstitials may travel easily becomes blocked by larger, less mobile defects, and it becomes increasingly more difficult for processes such as (2), (3), and (4) to occur.

Diffusion of vacancies has been considered, but it seems likely that such processes require much higher activation energies than steps (1) through (5) and can be neglected at low temperatures. Thus, if processes such as (2), (3), and (4) can be made to occur at a much higher rate than polymerization reactions (5), radiation damage will be considerably reduced. The damage accumulated at 185 C is only 10 to 20 per cent that accumulated at 30 C. If reactions such as (2), (3), and (4) have activation energies of some 30 to 40 kcal/g-atom, they would be considerably accelerated at 185 C. Reactions such as (5) which form higher complexes must occur near room temperature. It is probable that they are activated to a large extent by hot carbon atoms which still carry some kinetic energy in excess of the lattice energy.

$$C^* + C_n \longrightarrow C_{n+1} \qquad (5A)$$

It may not be necessary for the hot carbon atoms to actually strike C_n, but if a large amount of energy is transferred to the lattice immediately surrounding a $C - C_n$ pair, they may have several opportunities to react before temperature equilibrium with the lattice is established. Another

type of hot carbon reaction, in which a C_n complex reintegrates with a vacancy to form graphite and the lower complex, seems necessary to explain the phenomenon of irradiation annealing. These types of reactions would be more dependent on flux and less on temperature than (2), (3), and (4).

The mobility of defects depends upon their environment with the result that the annealing kinetics is complex. The C_o activation energy spectra (Figures 7-10) show that many overlapping processes contribute to the annealing rate. The peak at about 33 kcal in Figure 7 is usually attributed to reintegration reactions such as (2) and (4). Because the C_o lattice constant changes almost linearly with exposure out to about 1000 MD/CT at 30 C, it has been suggested[1] that (3) has little effect on C_o. Stored energy release[10] also shows a maximum at 30-35 kcal. The processes associated with higher activation energies are not well defined but are due to annealing of more and more complex types of defects.

In Figures 8-10 the band envelope for the isothermal anneals of several different samples are compared. The effect of exposure at 30 C is shown in Figure 8. The build up of a 33 kcal peak has begun in the 233 MD/CT, but very little damage has accumulated at high activation energies. It seems necessary that lower activation energy damage build up first. If reactions such as (5) are largely flux dependent and require a $C - C_n$ collision or a $C - C_n$ pair close to a hot part of the lattice, then the rate of formation of higher complexes will be proportional to the concentration of lower complexes. Also, as low activation energy damage builds up, mobility of C^- and C_2 becomes less and the chance for polymerization reactions increases.

The effect of three exposure temperatures at the same neutron exposure is shown in Figure 9. At the lowest temperature of 127 C a small maximum is found at 39 kcal. Had the sample been exposed at 30 C, then annealed for several months at 127 C, the characteristic annealing function would have moved to about 39 kcal. A small amount of damage remained below 39 kcal. While graphite was irradiated at 127 C, damage was also

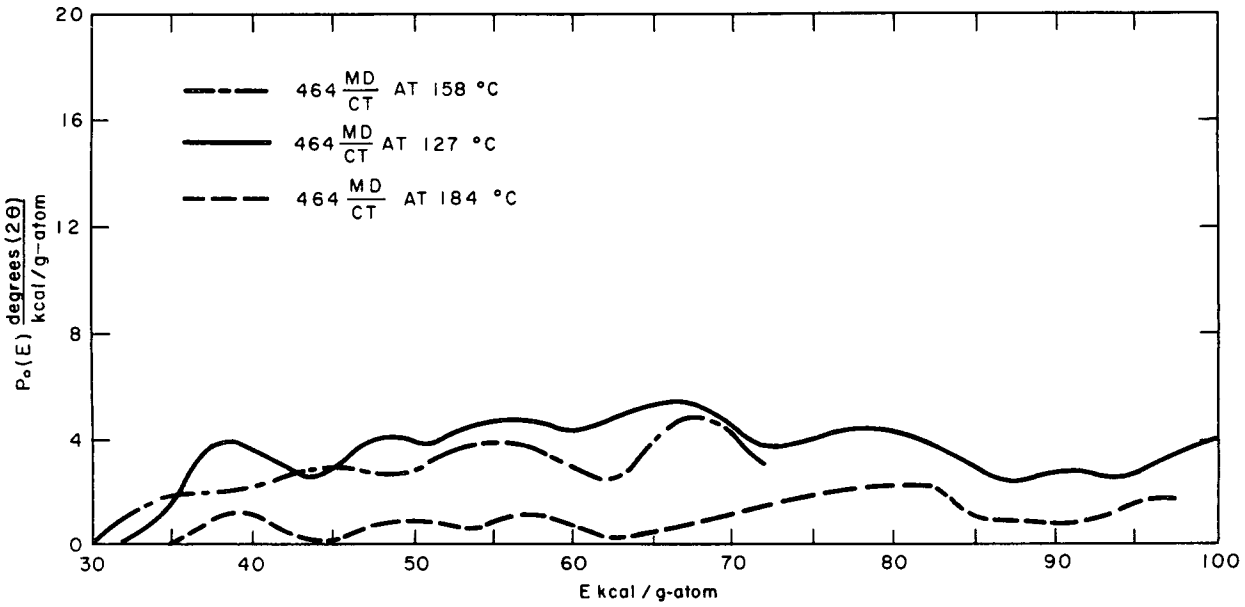

FIGURE 9

Activation Energy Spectrum - 464 $\frac{MD}{CT}$ at 127 C, 158 C and 184 C

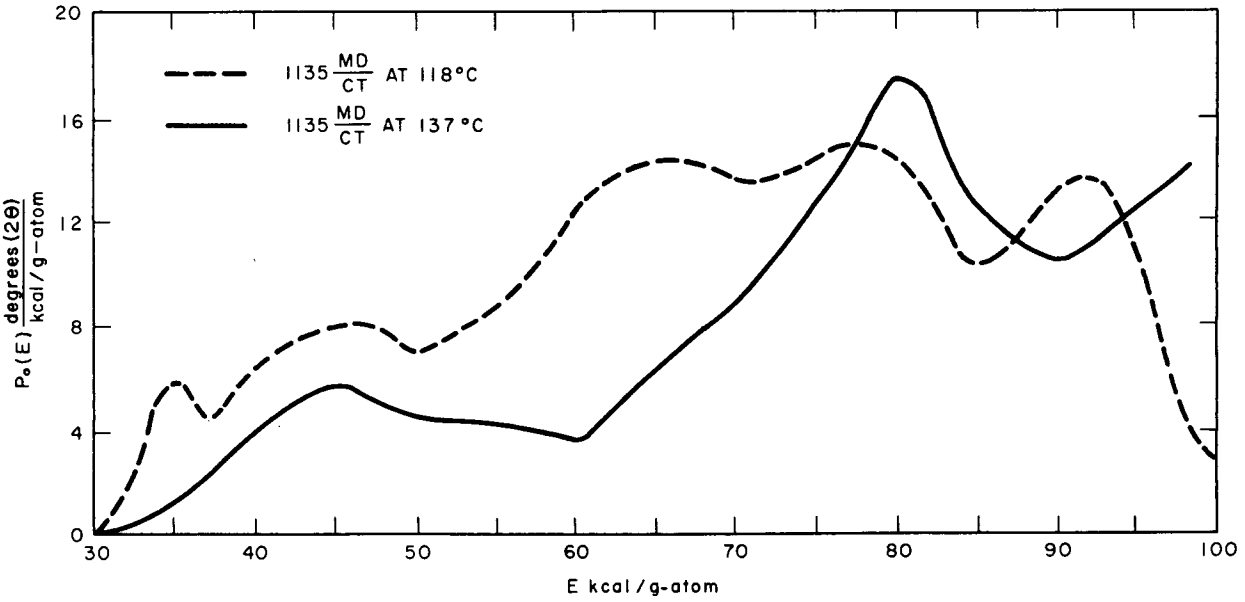

FIGURE 10

Activation Energy Spectrum 1135 $\frac{MD}{CT}$ at 118 C and 137 C

being produced at lower activation energies so that a small tail may exist below 39 kcal, the character of which depends on both flux and temperature. At higher exposure temperatures of 158 C and 185 C, damage is again distributed almost uniformly although the total amount is less.

Figure 10 shows that after 1135 MD/CT at 118 C and 137 C graphite is much more seriously damaged. While the total damage (given by the area under the curve) of the 556 MD/CT sample exposed at 30 C is not much greater than the 1135 MD/CT sample exposed at 118 C, the distribution of damage is considerably different. The greatly over-simplified series of reactions (1)-(5) indicate that if C^- ions could reintegrate immediately at high temperatures, no high activation energy damage would be formed. However, some close interstitials will combine, a few close $C^- - C_2$ pairs will react, and so on. Also, there are certainly other important ways of accumulating damage of high activation energy such as multiple vacancy formation which have not been discussed. The result is that at higher exposure temperatures considerably less high activation energy damage and much less low activation energy damage accumulates.

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