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LOW-TEMPERATURE IRRADIATION AND ANNEALING EXPERIMENTS IN GRAPHITE

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LOW-TEMPERATURE IRRADIATION AND ANNEALING EXPERIMENTS IN GRAPHITE

BY

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The author gratefully acknowledges the contributions of J. E. Hove, who proposed the experiment of electron irradiation of graphite at liquid-helium temperature and contributed to its execution, and the contribution of T. G. Berlincourt, who also assisted during the irradiation. Irradiation of samples in the Brookhaven Reactor was by courtesy of D. T. Keating and G. J. Dienes, of Brookhaven National Laboratory.

ABSTRACT

Recent measurements of physical property changes in graphite exposed to nuclear radiation at temperatures near that of liquid nitrogen, and during subsequent thermal annealing, showed considerably more damage occurred and remained at this low temperature than during room-temperature irradiation. To provide more information for formulating and testing radiation damage and annealing models, further experiments were performed. The experiments included:

a) Electron irradiation of graphite near 4° K, followed by measurement of the electrical resistivity changes due to annealing at temperatures up to room-temperature.

b) Neutron irradiation at temperatures below 125° K, followed by measurement of the annealing-out of radiation-induced crystallographic c-axis expansion at temperatures up to room-temperature.

In addition calculations based on previous stored-energy release experiments have been applied to a proposed model of interior-to-boundary diffusion of lattice defects to test plausibility of the model. The results of these experiments and calculations are discussed in relation to a recently proposed model for explaining radiation damage and thermal annealing.

I. INTRODUCTION

Early in the history of nuclear reactor technology, radiation damage to graphite was predicted by Wigner[1] and thereafter verified by cyclotron irradiation at Washington University.[2] The most obvious manifestation of exposure to damaging radiation (energetic nuclear particles such as neutrons, protons, etc.) is a pronounced dimensional expansion, but many other physical properties also exhibit remarkable changes.[3] Since graphite has excellent neutron-moderating properties, and since it serves as the structural body in many reactor designs, it is important to know how the physical properties, such as dimensional expansion, thermal conductivity, and structural strength, are altered by neutron flux and reactor temperature. In order to predict amount of damage and to choose methods of minimizing undesirable property changes, considerable research has been conducted to discover the nature of radiation damage in graphite, both as to its gross physical properties and as to the detailed, atomistic mechanism by which damage and subsequent thermal annealing proceeds. Until recently, all such studies have been conducted at room temperature or above. Some success in explaining the electronic properties of graphite[12] has been achieved. Attempts to identify the processes of annealing, and therefore the end product of damaging radiation, from annealing kinetics have not been so successful, for annealing seems to be complicatedly characterized by a continuum of activation energies which makes it difficult, if not impossible, to specify other rate-governing factors (such as order of reaction). The detailed mechanism of annealing and radiation damage remained, therefore, incompletely specified.

More recently, irradiations have been applied at temperatures near that of the boiling point of liquid nitrogen.[14] The resultant property changes are considerably larger than those resulting from similar irradiations at room temperature. Annealing following irradiation at low temperatures demonstrates that a considerable part of the damage resulting from irradiation at low temperature can be annealed out by annealing to room temperature. Presumably, therefore the lesser damage retained during irradiation at room temperature is due to partial annealing occurring during irradiation. Therefore, that annealing phenomena above room temperature appear complex is not surprising since the phenomena are a combination of radiation damage and the products of partial annealing. Study of

low-temperature irradiation and annealing seems to have more promise of revealing at least some of the mechanisms basic to radiation damage.

Following the preliminary low-temperature experiments, Hennig and Hove[5] proposed a mechanism of radiation damage and annealing for all temperatures. A brief resume of the low-temperature processes, described in detail by Hennig and Hove, is as follows. Irradiation produces, at low temperatures, a number of vacancies and interstitials in the graphite crystal lattice, with most of the interstitial atoms occurring in clusters; also a number of electron traps are created. At the lowest temperatures of active annealing, recombination of close interstitial-vacancy pairs occurs, and break-up of interstitial clusters begins. Drifting apart of clustered interstitials and recombining of close interstitials and vacancies continue as annealing temperature increases to room temperature. The observed property changes are theorized to be the result of redistribution of the interstitials and release of electrons from traps as the traps are annihilated. In the proposed model it was recognized that the experimental data is far from complete, and that some of the assumptions involved are open to question. Therefore, this report deals with more recent experiments on low-temperature irradiation damage and its recovery.

II. ELECTRICAL RESISTIVITY DURING ELECTRON IRRADIATION AT 4° K AND ANNEALING AT TEMPERATURES UP TO 300° K[12]

The model for irradiation damage, described by Hennig and Hove,[5] is extensively based on information obtained from neutron and proton irradiations at nominal liquid-nitrogen temperatures and on subsequent annealing data. It was further postulated that the damage retained at this temperature represents essentially all the damage formed. The experiment described herein was conducted to verify this assumption. This experiment was to irradiate graphite with electrons at the lower temperature of liquid helium. The graphite sample was maintained at, or close to, 4° K during irradiation, and then pulse-annealed at successively higher temperatures up to about room temperature. Electrical resistivity was measured during irradiation and annealing.

Two samples of AWG graphite were prepared for this experiment. The samples were 1.62-in. long, 0.125-in. wide, and 0.040-in. thick (Sample 1), and 0.020-in. thick (Sample 2). For the purpose of making electrical resistivity measurements on each sample, four potential probes were placed about 1/8 in. apart along the center of the sample; electrical contact was made at each end of the sample for passage of a standard direct current. In Sample 1 the probes were 0.010-in. tungsten pins embedded in holes drilled in the sample. For the probes on Sample 2, narrow copper bands ($\sim$0.010 in.) were electroplated across the graphite specimen. Then copper wires, as potential leads, and one thermocouple junction were soldered onto these pins or copper bands.

Irradiation was applied with the sample immersed in liquid helium as shown in Fig. 1. The sample was placed, as shown in Fig. 2, about 1/8 in. from the copper window to permit irradiation of the area between one pair of probes, leaving the area between the other pair to be used for control measurements. Irradiation was with 1.25-Mev electrons degraded in energy only by the window and 1/8 in. of liquid helium. Annealing was done in place by resistance heating with cooling bath removed. The annealing pulses were of one-minute duration, at successively higher temperatures, and with each pulse followed by a resistivity measurement at 4° K. (An additional minute at the annealing temperature produced no further measureable change).

The total irradiations produced a change of resistivity of 7 per cent and 4 per cent respectively in the first and second samples. The rate of damage with irradiation was far from uniform; a piece of scotch tape placed before Sample 2 showed by discoloration that the electron beam did not strike the sample where expected, and it was concluded that the beam wandered around on each sample during irradiation, yielding an apparent irregular damage rate. Also, the results of the first irradiation caused some concern about possible heating in the sample by the electron beam; therefore, the second irradiation was performed with the sample temperature continuously monitored, with the beam current reduced, with a thinner sample, and with the thermocouple more closely coupled to the sample by soldering it directly onto one of the electroplated copper bands. During this irradiation the maximum temperature did not exceed 10° K, and for the most part, remained close to 4° K. Because of the similarity of the two annealing runs, it was believed that the sample temperature also remained low during the first run.

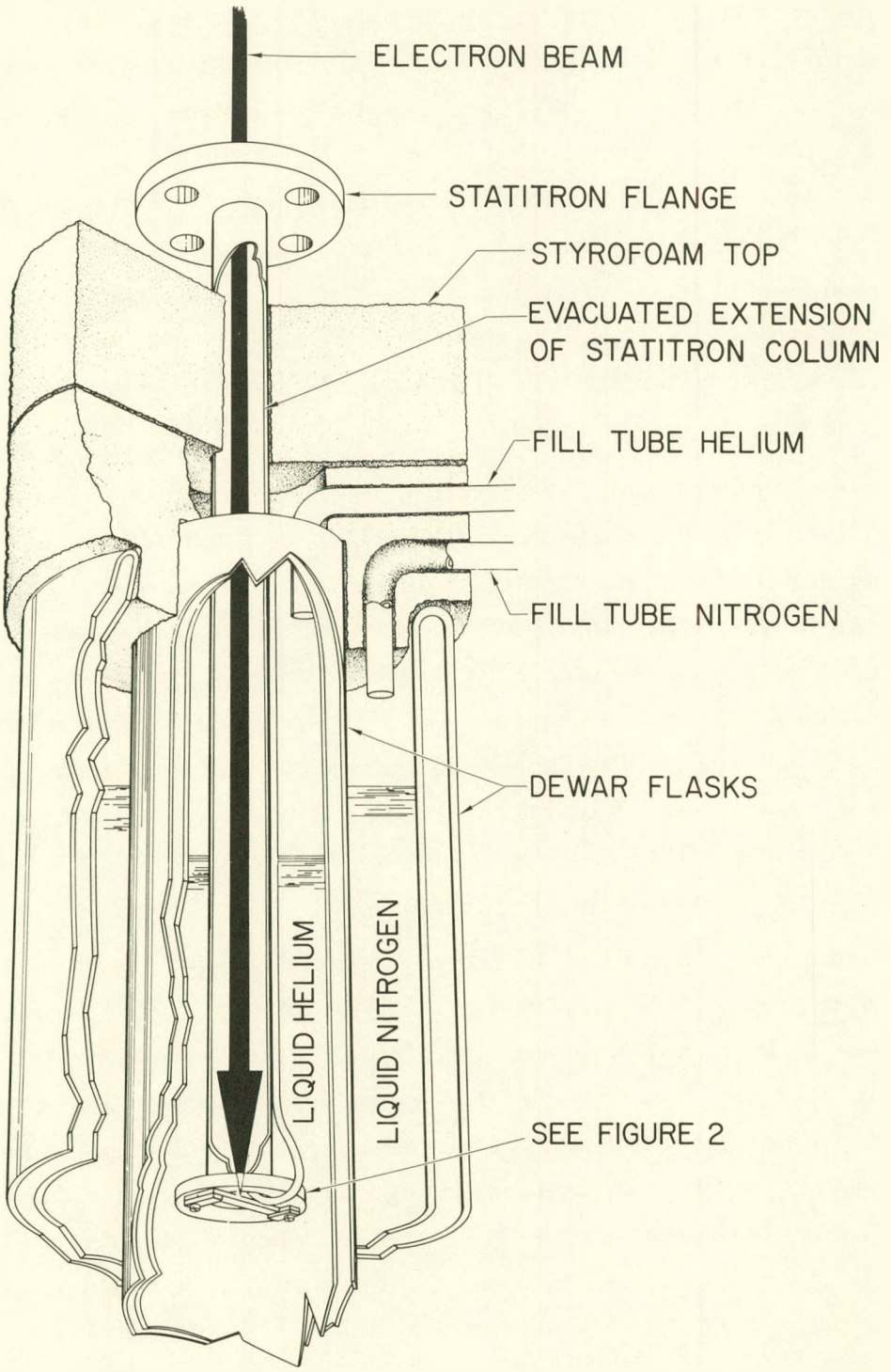

Fig. 1. Schematic Arrangement of Irradiation Apparatus

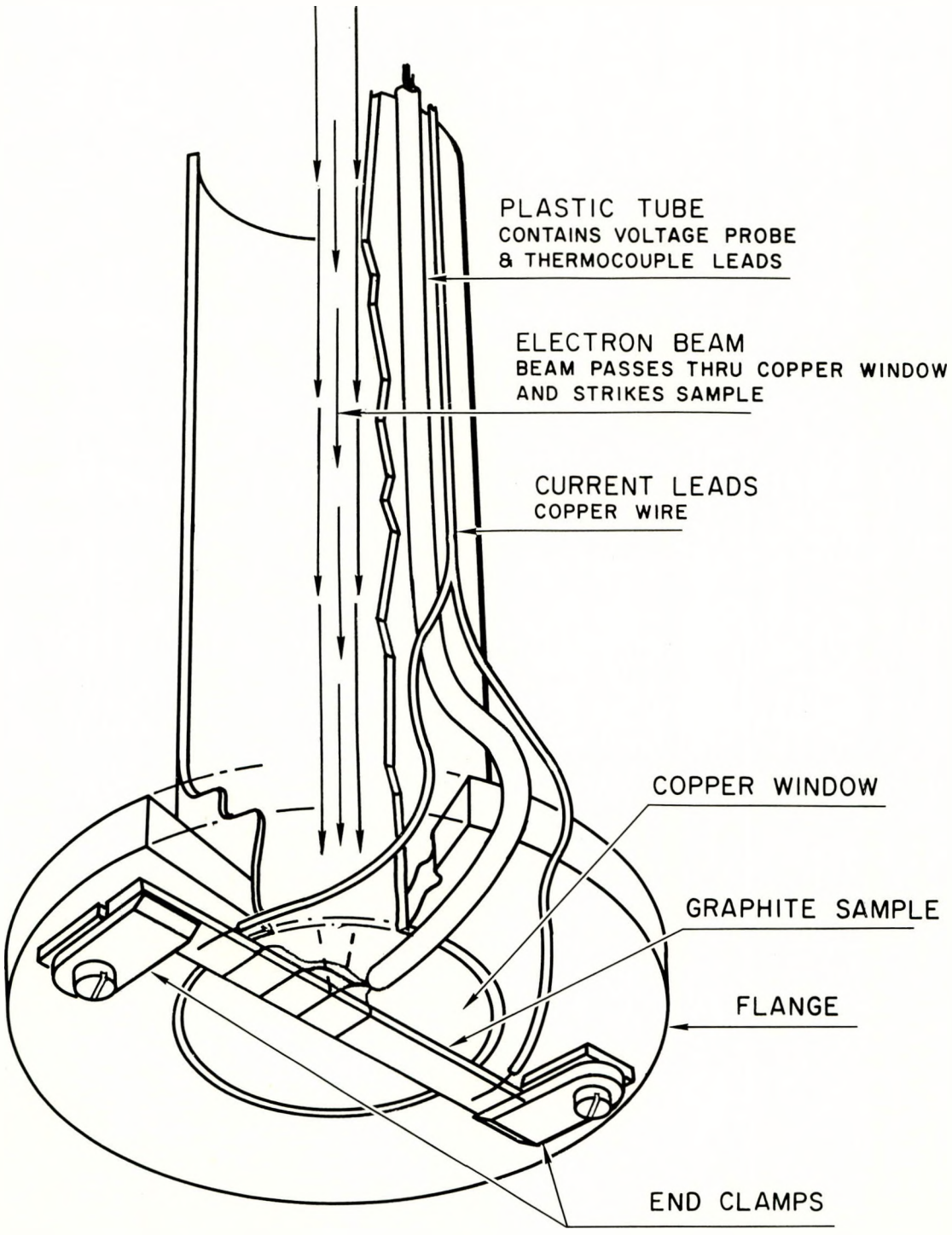

Fig. 2. Sample Details of Apparatus

Annealing data are presented in Fig. 3, showing the fractional change in resistivity. Practically no effect below about 80° K appears. Slight annealing may take place, but it is too small to warrant attention, especially when compared to the striking behavior at higher temperatures. The abruptness of the annealing between 80° and 100° K is notable when compared with the initial assumption that essentially no annealing took place during the earlier irradiations. Consideration of the actual temperatures during the normal "liquid nitrogen" irradiation indicates maximum temperatures in the range of 103° K (protons) to 125° K (neutrons). These temperatures are high enough to suggest most of the annealing in the region 80° to 110° K must have taken place during irradiation. Figure 4 compares the present data with that previously obtained following neutron and proton irradiation.[4] The smaller peaks of the latter data are, presumably, due partially to the annealing at the higher irradiation temperatures, although some dependence of the annealing behavior on the total exposure also exists. On the basis of this comparison, it may be concluded that, although the assumption by Hennig and Hove[5] (that all the damage was retained by the samples irradiated at

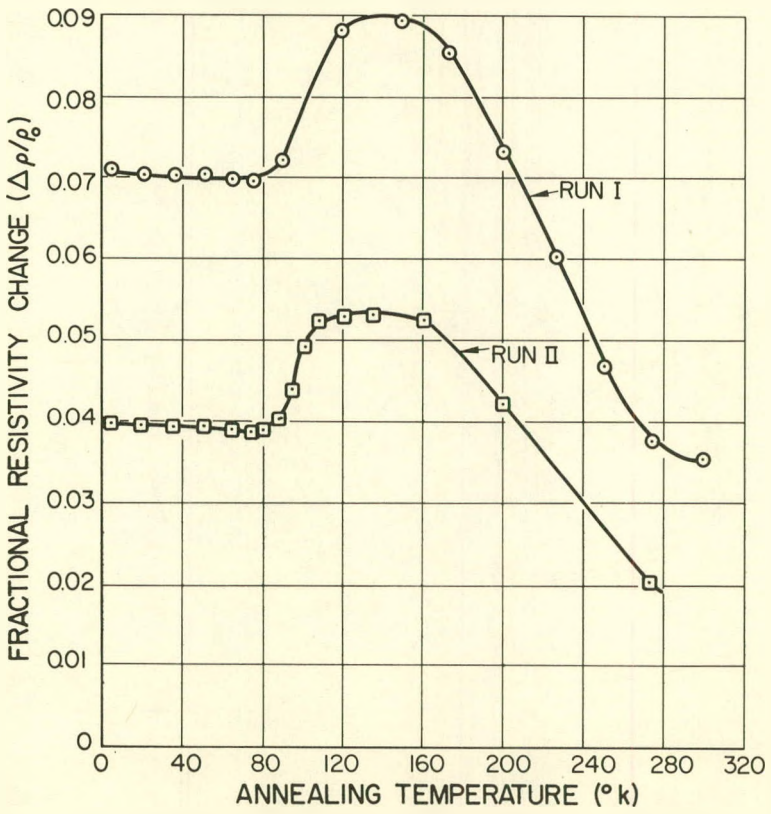

Fig. 3. Resistivity Annealing Curves. $\frac{\Delta\rho}{\rho_o}$ vs Annealing Temperature

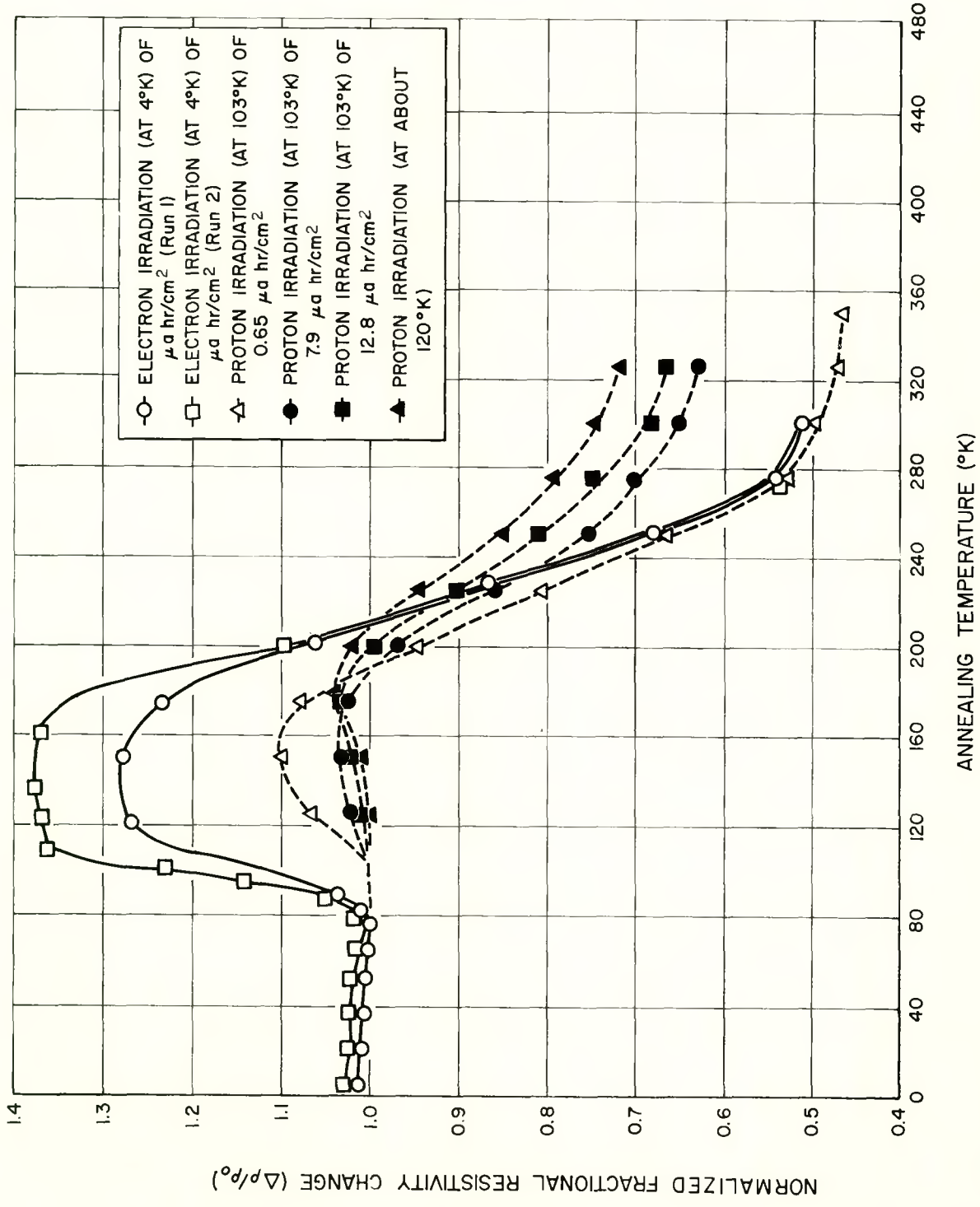

Fig. 4. Normalized-Resistivity Annealing Curves Compared to Previous Data; $\frac{\Delta\rho}{\rho_o}$ vs Annealing Temperature

nominal liquid nitrogen temperature) is not completely justified, probably no new type of damage center is introduced by the lower temperature irradiation. In other words, the previous annealing experiments reveal some of the behavior reported here but not all of it. From the standpoint of radiation damage studies, it is of interest to note that, if the sample temperature is carefully held below 80° K, an irradiation at liquid nitrogen temperature is sufficient to inhibit all thermal annealing of the damage centers.

The decrease in electrical resistivity which occurs between 160° K and room temperature has been accounted for by a proposed reintegration of close interstitial-vacancy pairs. However, no presented explanation clearly applies to the observed sharp increase between 80° K and 110° K. As a generality, the increase in resistivity should be attributable either to an increase in the number of scattering centers or to a release of electrons from electron traps which, in graphite, leads to a decrease in conducting electrons. Experimental data to indicate which of these processes is responsible for the observed sharp increase in resistivity are not presently available.

Experiments which would be useful to interpretation of the annealing characteristics between 80° K and 110° K are those which would be sensitive to only electron release or to the concentration of scattering centers. Such experiments might involve measuring magnetic susceptibility for the former, or measuring specific heat or c-axis spacing for the latter. (The c-spacing measurements of the next section do not extend to low enough temperatures to check this). Since no annealing appears to occur at 77° K or below, future experiments of this kind can be performed with liquid nitrogen as a coolant rather than the more expensive coolants like liquid hydrogen or helium.

III. X-RAY MEASUREMENTS OF C-AXIS SPACING

Keating,[7] of Brookhaven National Laboratory, has recently shown that there is a greater rate of c-axis expansion, with neutron irradiation at a temperature near that of liquid-nitrogen than has been observed with irradiation at about 300° K. This is further evidence of enhanced irradiation damage at low temperature. The experiment to be described was undertaken to observe the change in c-axis dimension during annealing following irradiations at a temperature near that of liquid nitrogen.

Crystallographic measurements were made by X-ray diffraction with nickel-filtered copper radiation. With CuK_α X-rays, the (00.8) diffraction line of graphite appears in the vicinity of the angle (2θ) of 130 to 135 degrees and (00.6) about 87 degrees. The sample was mounted on a Philips diffractometer in a mount designed to maintain temperatures as low as liquid nitrogen in vacuo and which permitted mounting the sample without warm up.

The samples used in this experiment were cut from pitch-bonded, natural graphite which had relatively large graphite crystallites. The samples were irradiated in the liquid nitrogen facility of the Brookhaven Reactor. Samples 1 and 2 were irradiated to 5919 Mwh and Sample 3 to 22,293 Mwh (or 3.4×10^{18} and 1.3×10^{19} nvt, respectively, of neutron flux having energy >1/2 Mev). The maximum temperature during irradiation was limited to 125° K, but stored-energy release indicated[8] that samples having the heavier irradiation may have been annealed to 150° K sometime before experimental use. Samples 1 and 2 were 0.030-in. thick and Sample 3 was 0.010-in. thick; the area exposed to the X-ray beam was about 1/4 x 3/8 in.

Pulse annealing was conducted by heating the sample and sample mount to a maximum temperature, termed the annnealing temperature, then quickly cooling the sample to temperatures suitable for determining the c-axis contraction resulting from the annealing pulse. Data, discussed below, indicate this was probably a sufficient anneal at each temperature.

Samples 1 and 2 were annealed by allowing the temperature to rise slowly to the desired maximum temperature, then immediately cooling the sample to the base temperature of 115° K for redetermining the c-axis dimension. Sample 3 was annealed in a somewhat different manner. The temperature of the sample was quickly brought up to the annealing temperature, then stabilized for an ~15-min. period in order twice to scan the diffraction peak position. The temperature then was reduced to a base temperature for measuring peak position. Measurements were also made at intermediate temperatures.

Figure 5 shows the annealing behavior of the three specimens. The data are given as the amount of remaining c-axis expansion following anneal at the corresponding annealing temperature. The data are compared to the value of c for unirradiated graphite, as measured on the (00.8) line. Due to instrumental effects,

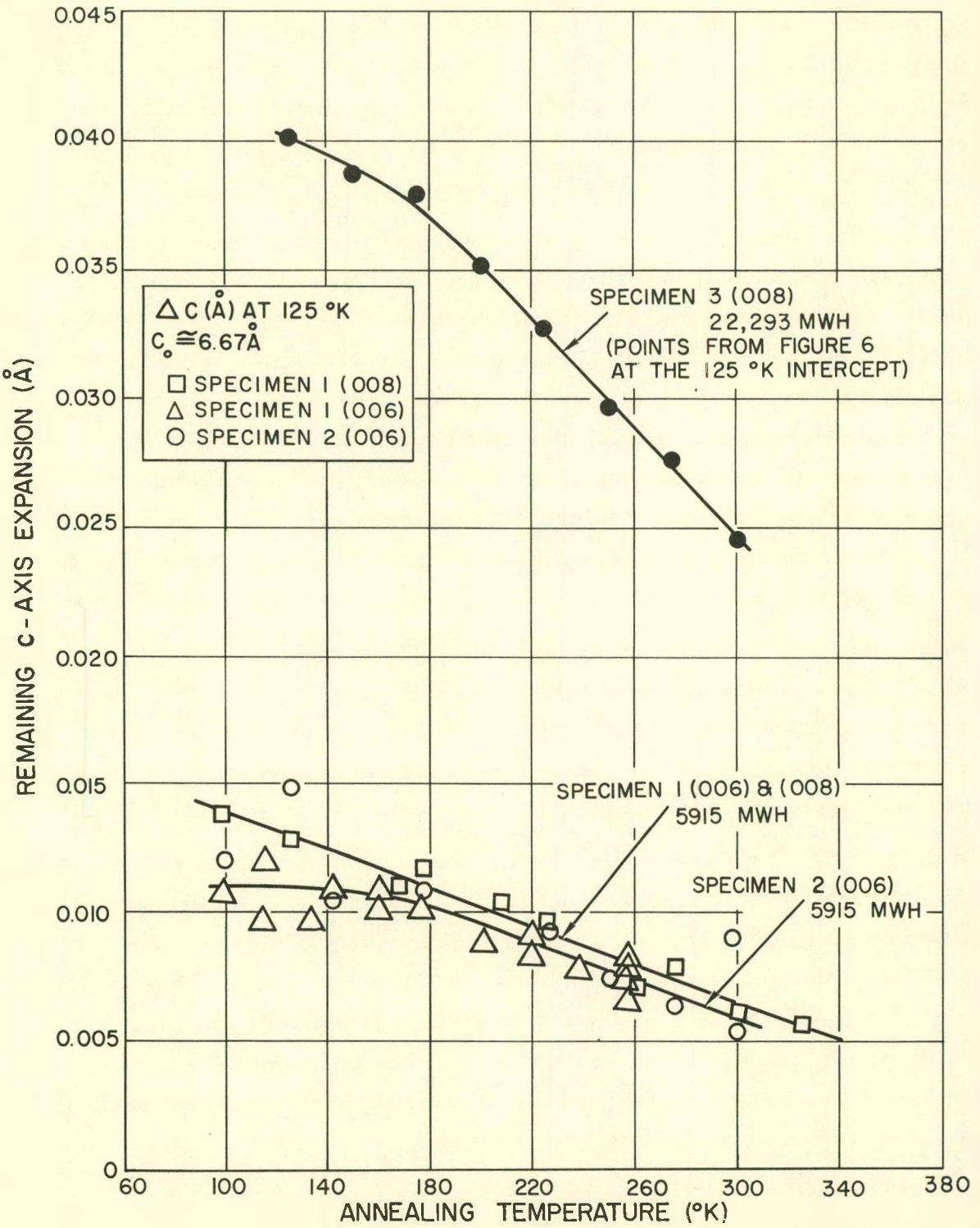

Fig. 5. c-axis Annealing Curves. Δc vs Annealing Temperature

the (00.6) line yielded, for Sample 1, values of c which were 0.0045 Å higher on the average than for the (00.8) line. Therefore, the c-values from the (00.6) lines on Sample 1 and 2 were lowered by this amount to coincide with (00.8) on the Δc-scale. The points on the upper curve (for Sample 3, (00.8) line) were taken from Fig. 6 at the intersection of the solid lines and T=125° K.

Figure 6 shows the effect of annealing temperature as well as the temperature dependence at temperatures below each of the annealing temperatures. No change in the thermal coefficient of expansion outside the limits of experimental scatter is present. There seems to be a tendency for the data to be low at the center of the straight lines for 250 to 300° K anneal; calculations indicate this may be due to irreproducibility of sample position relative to the ideal diffraction-focusing geometry caused by thermal non-equilibrium in the sample-mounting structure during rather rapid temperature excursions. Since sample position is slightly effected by the temperature of the mount, the slope of the c vs T line is also affected. Because of this, the slopes in Fig. 4 do not represent the true thermal expansion of graphite. The important point is the comparison of slopes before irradiation, and before and after annealing. This comparison indicates that, at least up to an interstitial concentration of 0.6 per cent (estimated from neutron scattering experiments[9] and the known irradiation of this sample), the bonding forces between planes is not significantly altered.

Figure 6 indicates the effect of the initial 15-minute anneal and a subsequent two-day anneal at 300° K. Since, during two days additional at 300° K, only a little more than double the amount of annealing, as compared to a 15-minute anneal, occurred, than an essentially rapid and complete (for practical purposes) anneal must be assumed as each higher annealing temperature is reached. This lends justification to the rapid annealing used for Samples 1 and 2, as well as Sample 3.

Figure 5 indicates the annealing rate is continuous and uniform above about 175° K and, although uncertain, less at lower temperatures. If this decrease is interpreted as being due to a reduction of the number of interstitial (interplanar) atoms, the explanation is consistent with other property changes (i.e., electrical and thermal conductivity[4] and stored energy release[8]). The more or less steady change indicates a correspondingly steady annealing of the interstitial carbon atoms

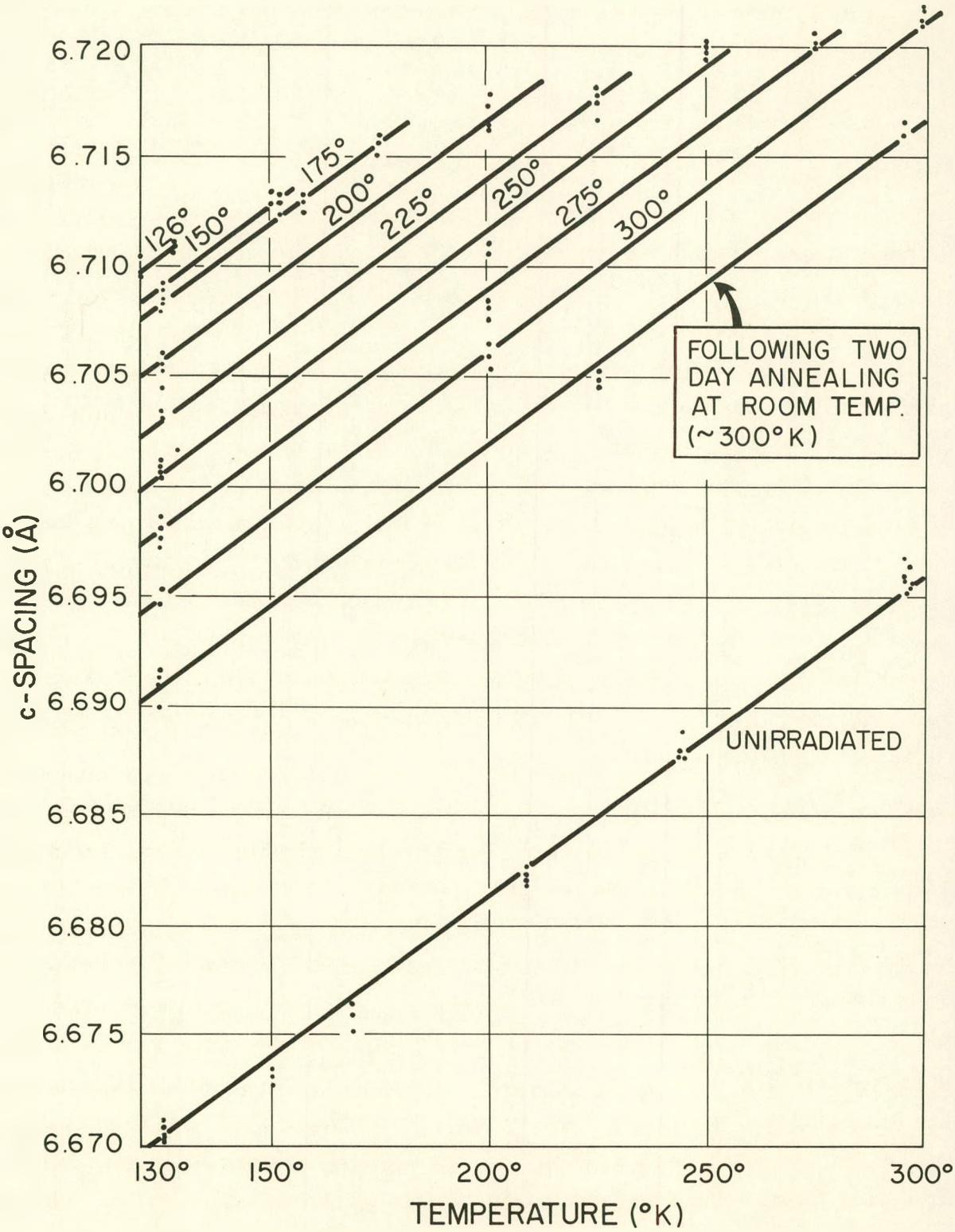

Fig. 6. c-spacing as a Function of Temperature after Annealing to Successively Higher Temperature (22,293 Mwh irradiation)

to some other lattice positions such as crystallite boundaries or lattice vacancies. This again is in qualitative agreement with the other property changes.

The total change in Δc to 300° K is about 40 per cent for Sample 3 and about 55 per cent for Samples 1 and 2. In the same temperature range, the relative amount of stored energy release is about 25 per cent[8]. If Δc is proportional to the concentration of interstitials, comparison of these data would indicate that the interstitials which can anneal at below 300° K, in addition to having a lower activation energy, do not yield as much energy on the average to the lattice per atom as those which anneal but above 300° K. Consequently, it seems reasonable to consider these early-moving interstitials to have lower strain-energy by virtue of being close to either lattice vacancies or crystallite boundaries. Hennig-Hove suggested that close interstitial-vacancy pairs involve roughly one-third of the displaced atoms, and that these contribute mainly to the low-temperature annealing. To such "close pairs" can now be ascribed a lower energy state than the more widely separated interstitials and vacancies. This gives some support to the concept of close pairs.

IV. DIFFUSION ANNEALING IN TEMPERATURE REGION OF 100° TO 250° K

A previous report[8] described the stored-energy release from graphite which had been irradiated below 125° K in the Brookhaven Reactor. It was found that as the temperature was slowly raised, the temperature at which the maximum rate of release occurred depended on the graphite type. Since the prominent difference between graphite types was crystallite size, this difference was tentatively credited for the difference in temperature of peak release, and a mechanism of defect diffusion to the crystallite boundaries was suggested, governed by an activation energy of 0.43 ev. The preliminary calculations which yielded these figures have been extended to examine the plausibility of the diffusion process, and it is apparent that the simplest mechanisms do not suitably predict the observed temperature breadth of the annealing peak. However, such a model is partially successful and might be modified to more satisfactorily predict experimental results. The simple model and calculations based on it will be briefly outlined here.

In the simple model it is hypothesized that some type of lattice defect, created by neutron irradiation and initially uniformly distributed, diffuses two-dimensionally through the graphite crystal lattice with constant activation energy. As each

defect arrives at a crystallite boundary, it is eliminated and releases stored energy which appears as thermal energy. By this mechanism, the rate of stored-energy release is highly dependent on crystallite size. As the sample temperature is slowly raised, the bulk of annealing will occur more quickly and, therefore, at a lower temperature for small crystallites than for large ones.

The basic equations from which derivations were made are stated briefly for reference as follows:

$$\frac{\partial c}{\partial t} = D\nabla^2 c \qquad \ldots(1)$$

$$D = \nu l^2 e^{-E/kT} \qquad \ldots(2)$$

$$-\frac{\partial L}{\partial T} = A \int_0^{r_0} \frac{\partial c}{\partial T} r\, dr \qquad \ldots(3)$$

$$T = at \qquad \ldots(4)$$

where

c = concentration of defects

ν = frequency factor = 10^{13}

l = length of individual jumps from one metastable position to the next = 2×10^{-8} cm

E = activation energy

A = proportionality constant

a = rate of temperature rise, approximately 0.05 degrees/sec for the experiment

r_0 = radius of cylindrical or disc-shaped crystallite

L = stored energy

r = distance from center of crystallite

D = diffusion coefficient

k = Boltzman constant

∇^2 = Laplacian Operator

t = time

T = absolute temperature

With initially uniform defect-concentration throughout the crystallite and with infinite trapping capacity at the circular boundaries, these equations lead to an expression for stored energy release as follows:

$$-\frac{dL}{dT} = BD\sum_{s=1}^{\infty} exp\left(-\frac{a_s^2}{\alpha r_0^2}\int_0^T DdT\right) \qquad ...(5)$$

where

a_s = roots of Bessel function $J_o(a_s) = 0$

and B = proportionality constant

Experimentally, it was found that AWG graphite has a peak rate of stored energy release at about 215° K, and SA-25 at about 185° K. Theoretical treatment of the thermal conductivity of these two graphite types has indicated crystallite sizes on the order of 3000 Å for AWG and 300 Å for SA-25.[10] When these sizes are used the experimentally observed temperatures correspond to an activation energy of 0.43 ev and a value of 10^{13} for the frequency factor.[8]

In the calculation to this point it was assumed that each graphite type consisted of uniformly sized crystallites. With this assumption, the peak shape for 3000 Å size was calculated from equation (5). This computed shape (curves A) is compared with experimental data for AWG graphite and for PBNG graphite (curves C) in Fig. 7.

The breadth of the computed curve is too narrow. However, a wide distribution of crystallite sizes in a given graphite type probably exists; the size-distribution curve shown in Fig. 8 was arbitrarily chosen as intuitively reasonable. This distribution curve was used as a weighting function modigying equation (5), so that the stored energy release from crystallites of all sizes is as follows:

$$-\frac{dL}{dT} = CD\int_0^{\infty}\frac{N(r_o)}{r_0^2}\left[\sum_{s=1}^{\infty} exp\left(-\frac{a_s^2}{\alpha r_0^2}\int_0^T DdT\right)\right]dr_o \qquad ...(6)$$

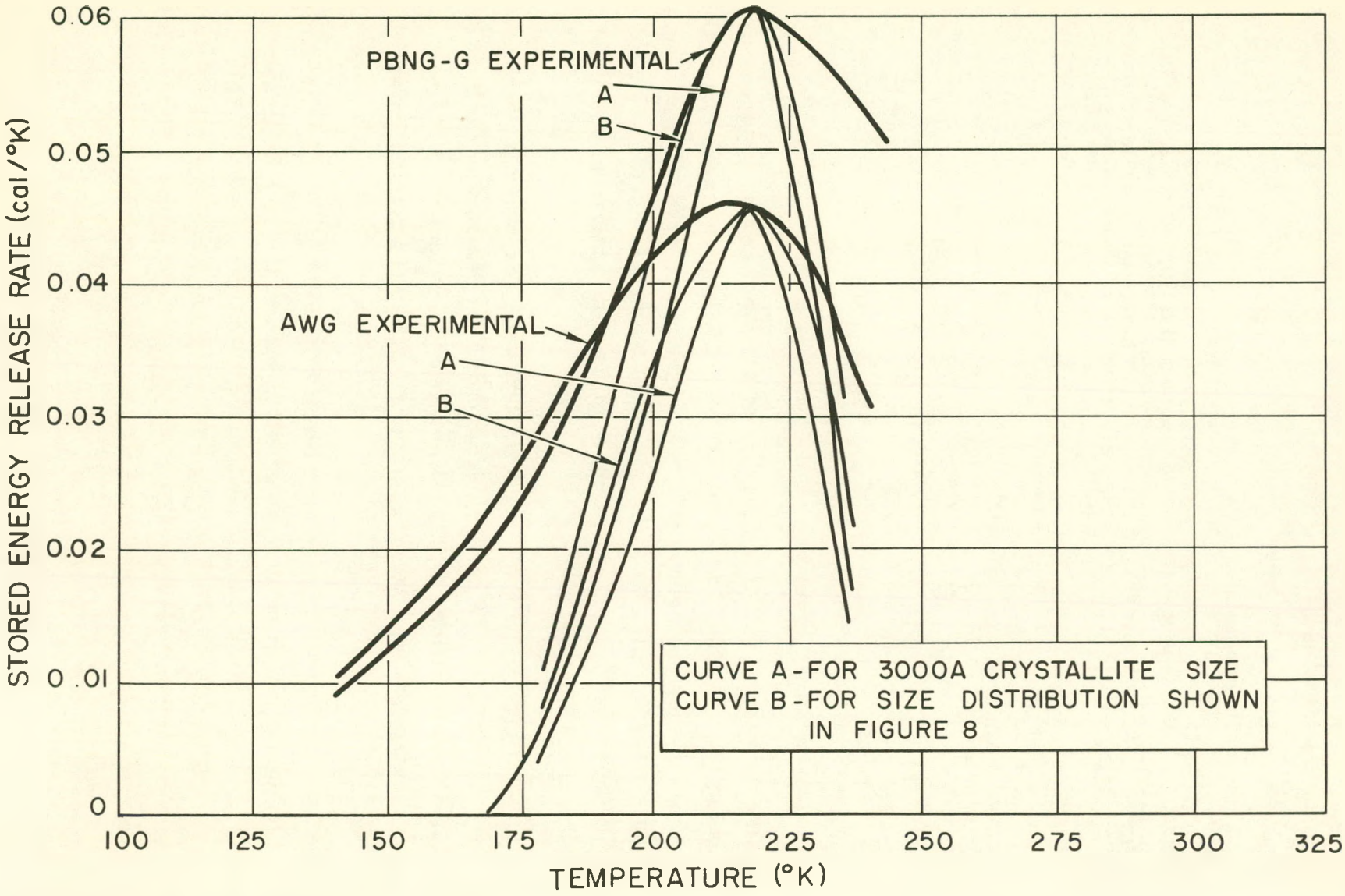

Fig. 7. Experimental and Calculated Stored-Energy Release Rates for PBNG-G and AWG Graphites

where

$N(r_o)$ = relative mass of crystallites of radius

C = proportionality constant

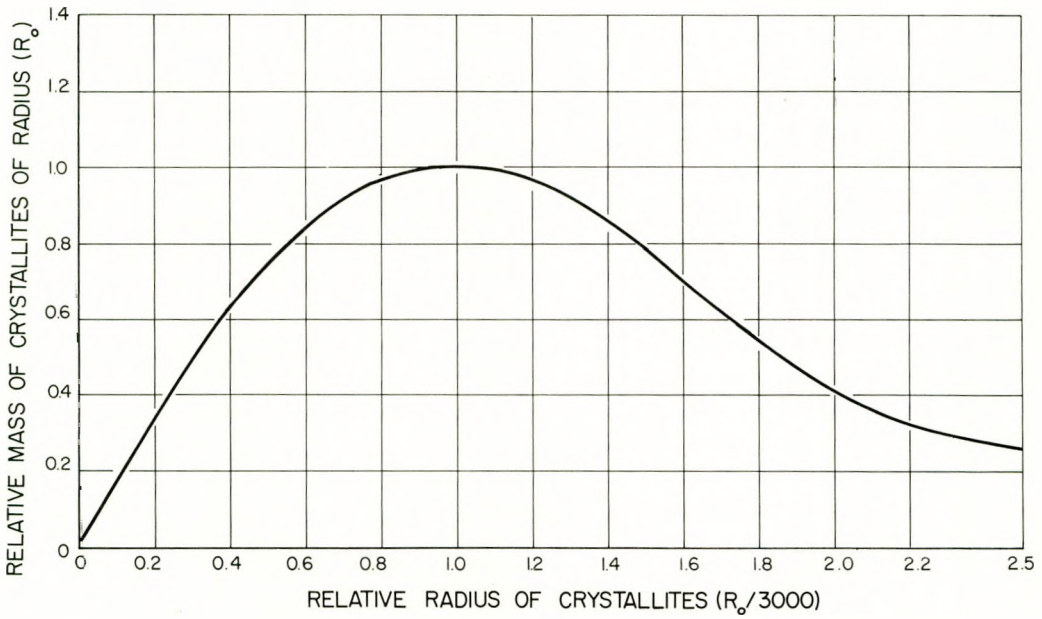

Fig. 8. Hypothetical Distribution of Crystallite Sizes Used to Compute Curve B Fig. 7

This equation and the function $N(r_0)$ (Fig. 8) yielded Curve B in Fig. 7. This also does not have the required peak breadth.

The value of the frequency factor for these calculations was taken to be 10^{13} The breadth of the annealing peak can be increased by decreasing the frequency factor, but this leads to other difficulties. For example, if $\nu = 10^{11}$, then $E = 0.36$ ev, and the temperature difference for the two graphite types is calculated to be 43° C, or about 50 per cent greater than observed.

Further exploratory calculations indicate that use of equation (6) cannot predict the breadth of the annealing peak with any seemingly reasonable distribution function $N(r_0)$ of crystallite size. From these considerations we might conclude that, if this diffusion process is valid, there must be, not only a distribution of crystallite size, but also a distribution of activation temperatures. From this point of view, the original hope of simpler processes being separable at low temperatures has not been realized.

If the annealing mechanism is governed by distribution of both particle sizes and activation energies, the calculation of the annealing behavior becomes quite

difficult unless there is independent knowledge of either or both variables. The calculations therefore have not been extended to include both of these.

The initial success of the simple hypothesized model in predicting the shift in temperature of the maximum annealing rate as a function of crystallite size encourages the hope that modification of the proposed model or inclusion of other data will make it possible to properly account for the observed breadth of the stored-energy release annealing peak.

V. SUMMARY AND CONCLUSIONS

The electrical resistivity annealing above 120° K and the reduction in c-axis spacing during annealing to room temperature are consistent with the Hennig-Hove model. It is uncertain whether the sudden increase in electrical resistivity between 80° K and 110° K is correctly explained by the model, and further special experiments will be necessary to demonstrate whether the increase is due to release of electrons from traps or to an increase of scattering centers.

Recent data shows that the temperature for maximum stored-energy release is dependent on crystallite size. This dependence is not explained by the Hennig-Hove model, nor is the assumption that interstitials migrate to the crystallite boundary consistent with the assumption of interstitial-vacancy integration. However, in lieu of another mechanism to account for the crystallite-size dependence, stored energy release calculations were made on the diffusion-to boundary process. These calculations show that, with such a process, it is necessary to assume not only a wide range of crystallite sizes in a given sample, but also a continuum of activation energies. If anything, this tends to make the proposed process more, but not completely, unacceptable.

There was an initial hope of finding, at low temperatures, property changes and, therefore, annealing processes which were uniquely separated in temperature from other changes or processes. It is noteworthy that the increase of electrical resistivity between 80° K and 110° K is bounded by temperature regions of little or no change, and that the rather large change occurs in a narrow temperature range. This, more than any other observation to date, seems to hold promise of finding a simple mechanism of radiation damage and annealing.

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