

where  $R$  is the excitation rate of free charge,  $n_t$  is the density of trapped electrons, and the other terms have been defined earlier. Equations (4) are nonlinear simultaneous differential equations. As a first approximation let us assume that the retrapping term  $k_2n(N_T - n_t)$  is small during the decay. Then with  $R=0$  at  $t=0$ , Eqs. (4) may be rewritten as

$$dn/dt = -k_1cn + b_2n_t, \quad (5a)$$

$$dn_t/dt = -b_2n_t. \quad (5b)$$

A solution of these equations is

$$n_t = n_{t_0}e^{-b_2t}, \quad (6a)$$

$$n = n_0 \left[ \left( 1 - \frac{b_2k_2N_T}{(k_1c - b_2)(k_2n_0 + b_2)} \right) e^{-k_1ct} + \frac{b_2k_2N_T}{(k_1c - b_2)(k_2n_0 + b_2)} e^{-b_2t} \right], \quad (6b)$$

where  $n_0$  and  $n_{t_0}$  are the values at  $t=0$  and the relationship between them is obtained from setting (4b) equal to zero for the steady-state condition (8).

The assumption that the retrapping term can be neglected is valid if

$$k_1cn \gg k_2n(N_T - n_t), \quad (7a)$$

$$b_2n_t \gg k_2n(N_T - n_t). \quad (7b)$$

Equation (7a) may be readily satisfied at low temperatures since then  $n_{t_0} \approx N_T$  and  $n_t$  changes slowly with time during the decay. Equation (7b) is certainly not valid at the start of the decay because at equilibrium

$$b_2n_{t_0} = k_2n_0(N_T - n_t). \quad (8)$$

However, at low temperatures  $n$  decreases much faster than  $n_t$  during the decay, so that (7b) will be satisfied for times shortly after the start of the decay.

By considering the temperature dependence of the time,  $t_1$ , for  $n$  to decay to some constant value  $n_1$ , it can be shown that  $t_1$  has a maximum as a function of temperature at temperatures such that

$$k_1c > b_2. \quad (9)$$

Equation (9) is satisfied at low temperatures.

## Length and Resistivity Changes in Germanium upon Low-Temperature Deuteron Irradiation and Annealing\*†

FREDERICK L. VOOK‡ AND R. W. BALLUFFI  
University of Illinois, Urbana, Illinois

(Received August 19, 1958)

Simultaneous measurements of the length change and resistivity of high-purity germanium single crystals were made upon irradiation and annealing. The specimens were initially irradiated at 25°K with deuterons of average energy 10.2 Mev to an integrated flux of  $6.2 \times 10^{16}$  deuterons/cm<sup>2</sup> and annealed to room temperature. The specimens were then irradiated at 85°K to an additional flux of  $9.2 \times 10^{16}$  deuterons/cm<sup>2</sup> and annealed to 364°K. The specific length expansion for both bombardments was  $\Delta L/L = (1.5 \pm 0.3) \times 10^{-21}/(\text{deuteron/cm}^2)$ . The annealing after both irradiations showed a gradual recovery of the expansion which was observable only after warming to above 200°K. The precise determination of the onset of annealing was limited by the accuracy of the expansion measurements ( $\sim 20\%$ ). Recovery was  $\sim 50\%$  complete by 300°K and  $\sim 85\%$  complete by 360°K.

### I. INTRODUCTION

**R**ADIATION damage produced in semiconductors by high-energy particle irradiation has been the subject of considerable interest in recent years.<sup>1-6</sup>

\* A portion of this paper was submitted by Frederick L. Vook to the Graduate College of the University of Illinois in partial fulfillment of the requirements for the degree of Doctor of Philosophy in May, 1958.

† This work was supported by the U. S. Atomic Energy Commission.

‡ Now at Sandia Corporation, Albuquerque, New Mexico.

<sup>1</sup> F. Seitz and J. S. Koehler, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press, Inc., New York, 1956), Vol. 2, p. 305.

The resistivity measurements agreed generally with previous results. A large increase in resistivity occurred during the low-temperature bombardments. The specimens became  $p$ -type after bombardment and annealing. On the first warmup after the bombardment at 25°K the resistivity decreased irreversibly by a factor of  $10^8$  between 65°K and 300°K with a large part of the decrease coming apparently between 150°K and 225°K. On the warmup from the second bombardment at 85°K an irreversible resistivity increase occurred on warming to 140°K. The resistivity then decreased irreversibly by a factor of  $10^3$  between 150°K and 200°K. Extensive resistivity annealing, therefore, occurred in an earlier stage of the recovery process than did the experimentally observable length change annealing.

Electrical measurements have been the prime tool in the great majority of investigations because of their sensitivity to lattice defects. Unfortunately, electrical

<sup>2</sup> J. W. Glen, in *Advances in Physics*, edited by N. F. Mott (Taylor and Francis, Ltd., London, 1955), Vol. 4, p. 381.

<sup>3</sup> H. Brooks, in *Annual Review of Nuclear Sciences* (Annual Reviews, Inc., Stanford, 1956), Vol. 6, p. 215.

<sup>4</sup> H. Y. Fan, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press, Inc., New York, 1955), Vol. 1, p. 283.

<sup>5</sup> H. Y. Fan and K. Lark-Horovitz, in *Report of the Bristol Conference on Defects in Crystalline Solids, July, 1954* (The Physical Society, London, 1955), p. 232.

<sup>6</sup> G. J. Dienes and G. H. Vineyard, *Radiation Effects in Solids* (Interscience Publishers, Inc., New York, 1957).

measurements alone do not lead to an unambiguous interpretation of the defect state (see the following paper<sup>7</sup>). Of major interest is the structure of the damaged material, and further experiments with other physical properties which yield information about the structural nature of the damage in germanium are, therefore, required. The present experiment was designed to (1) make simultaneous measurements of both volume changes and electrical resistivity changes produced by deuteron irradiation near helium temperature, and (2) study the annealing processes by making similar measurements during warming after irradiation. Measurements of volume changes under irradiation have been made for copper<sup>8</sup> and yield valuable information about the damaged state. The simultaneous measurement of resistivity was planned to allow a direct correlation of the two types of measurement on similar specimens subjected to the same treatment. Irradiation was carried out at low temperature in order to retain the damage. Many previous experiments at higher temperatures are difficult to interpret since unknown amounts of annealing may have occurred. In addition, high-purity germanium was used in an effort to eliminate possible complications in the irradiation and annealing caused by impurities. In several previous investigations the concentrations of chemical impurities have been comparable to the expected defect concentrations introduced by irradiation. Experiments with heavily doped material do give sensitive effects, however, since defect levels introduced by the damage occur near the Fermi levels for these materials.

## II. EXPERIMENTAL

The length and electrical resistivity measurements were made on several thin, bar-shaped single crystals mounted side by side in a helium temperature cryostat. The irradiation and annealing studies were carried out with the specimens in place, and all specimens were subjected to essentially the same treatments. The measurement of length changes upon cyclotron irradiation at low temperatures involves considerable difficulty since the fractional changes are small ( $10^{-4}$  in the present case). The rather specialized techniques used in these measurements are, therefore, described in the following.

### 1. Apparatus

The irradiation was performed in vacuum using a slightly modified form of the helium cryostat which has been employed previously by the University of Illinois radiation damage group.<sup>9</sup> The cryostat consists essentially of a liquid helium vessel surrounded by a

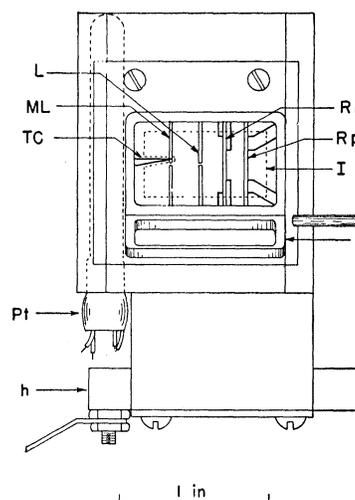


FIG. 1. Specimen block assembly.  $L$ =length specimen,  $ML$ =monitor length.  $TC$ =thermocouple,  $R_n$ =resistance specimen  $R_n$ ,  $R_p$ =resistance specimen  $R_p$ ,  $I$ =beam intensity outline,  $d$ =open door on block,  $Pt$ =platinum resistance thermometer,  $h$ =heater.

liquid nitrogen jacket. Four single-crystal specimens of the same orientation and similar geometry were mounted on a heavy block at the bottom of the helium Dewar in line with the deuteron beam (Fig. 1). One specimen was used for the length measurement ( $L$ ), two specimens were used for the resistivity measurements ( $R_n$  and  $R_p$ ), and the fourth specimen ( $ML$ ) was used as a monitor for measuring the temperature. The lower half of  $ML$ , not containing the thermocouple, was removed at an intermediate stage of annealing in Run II ( $T \leq 308^\circ\text{K}$ ) for x-ray lattice parameter measurements.<sup>10</sup> The upper half of  $ML$  was removed at the end of the present experiments for further lattice parameter measurements. The block and specimens were in good thermal contact with the liquid helium reservoir and were conduction cooled. The specimen block assembly was immediately enclosed by a copper thermal radiation shield which was conduction cooled to liquid nitrogen temperature. A final outer manifold (or "sump") maintained the vacuum. A set of ports and doorways was constructed to accommodate the deuteron beam and to allow the length measurement. A top view of the entire arrangement is shown in Fig. 2. The collimated deuteron beam entered the sump, passed through the specimens, and was stopped in the radiation shield. The cryostat Dewars and the radiation shield were electrically insulated from the sump and formed a Faraday cage which was used to measure the deuteron flux during bombardment. The charge accumulated on the cage was leaked to ground through a standard resistor. The potential across the resistor was recorded

<sup>7</sup> F. L. Vook and R. W. Balluffi, this issue [Phys. Rev. **113**, 72 (1959)].

<sup>8</sup> R. W. Vook and C. Wert, Phys. Rev. **109**, 1529 (1958).

<sup>9</sup> D. E. Mapother and F. E. L. Witt, Rev. Sci. Instr. **26**, 843 (1955).

<sup>10</sup> These measurements were carried out by Simmons; see the following paper [R. O. Simmons, Phys. Rev. **113**, 70 (1959)] for further details.

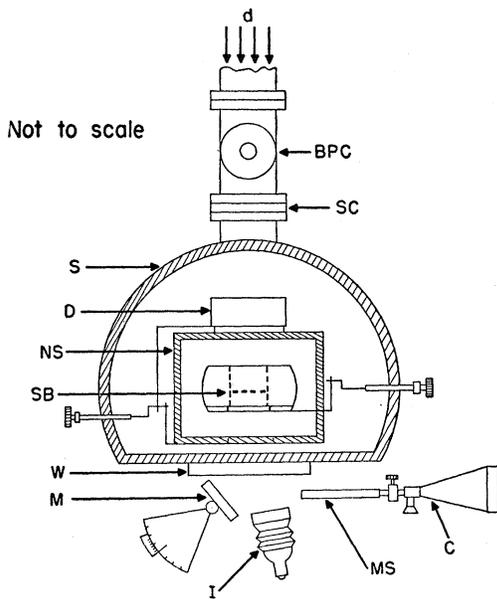


FIG. 2. Top-view schematic diagram of apparatus.  $d$ =deuteron beam, BPC=beam port collimator used in aligning specimens, SC=beam collimator for sump, S=sump, D=door closed during alignment to prevent specimens from being pre-irradiated at room temperature, NS=nitrogen shield, SB=specimen block, W=window, M=front surfaced mirror, I=30 watt AO illuminator, MS=microscope, C=camera.

on a Leeds & Northrup recording potentiometer giving a record of the cyclotron beam current.

Specimen alignment in the cyclotron beam was a critical problem, since the irradiated specimen length was comparable to the half-width of the vertical intensity profile of the beam. In order to measure the beam profile and place the specimen in the optimum position, the entire apparatus was mounted on a telescoping stand that could be moved up or down while the cyclotron was in operation.

## 2. Length Measurement

Since the length change was expected to be small, a refined measuring technique was necessary. The change in length was observed directly by measuring changes in distance across a gap cut transversely through the mid-point of the length measurement specimen as shown in Fig. 1. In this arrangement the two halves are free to expand and the change in length may be measured by observing only the narrow gap region. The optical technique described by Vook and Wert<sup>8</sup> was adopted. This method consists of photographing the gap region with a Leitz Makam Camera through an adapted Gaertner creep test microscope having a long working distance. This optical system had a magnification of 125 $\times$ . Distances on the photographic plates were then measured by means of a coordinate comparator. This technique allowed length change measurements to  $\pm 2 \times 10^{-5}$  cm on virtually strain-free crystals.

The arrangement is shown schematically in Fig. 2. The length measurement crystal and temperature monitor were obliquely illuminated through a port in the sump and open doors in the nitrogen and helium radiation shields. The gap was photographed in a front-surface mirror set at 45° to the specimen and open doors. The distance across the gap was measured between reference marks near the adjacent ends of the two half-crystals. The crystal surfaces were first polished to a mirror-like surface, and the reference marks were then lightly scratched into the surface parallel to the gap. With the oblique illumination the polished surface of the crystal appeared dark since most of the light intensity was reflected out of the viewing direction. However, small irregularities in the scratches produced bright diffraction spots against the dark background, and a number of these spots were used for measuring purposes. A reproduction of a typical photographic plate is shown in Fig. 3. The spots numbered are those used in the measurements. The coordinates of each spot were read with a Gaertner  $x$ - $y$  comparator which reads to 1 micron in both the  $x$  and  $y$  directions. The length change was calculated from the relative motions of the spots. The  $x$ - $y$  coordinate positions of the six spots were determined relative to a common coordinate system having its origin at spot 1 and  $y$  axis passing through spot 2. Changes in the length of the specimens were, therefore, given by changes in the  $x$  coordinates. The  $y$  coordinates served as a check on possible lateral movement of one half of the crystal relative to the other.

In order to separate irradiation and temperature dilation on annealing, the gap spacing as a function of temperature before irradiation was determined. The temperature dependence of this spacing is a function of the differential expansion of the germanium crystal and the block material. Molybdenum was used for the block since its thermal expansion closely matches that of germanium. The unirradiated thermal background curve is shown in Fig. 4. In the annealing studies this background curve was subtracted from the measured

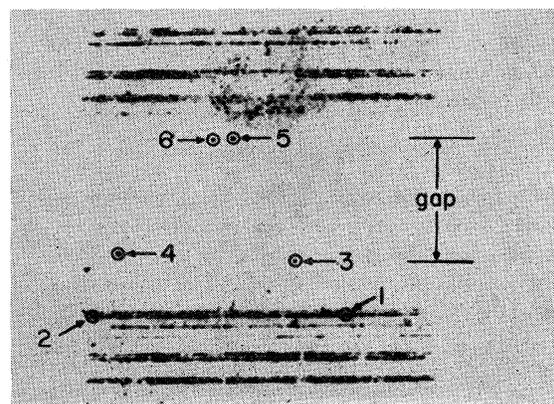


FIG. 3. Reproduction of typical photographic plate.

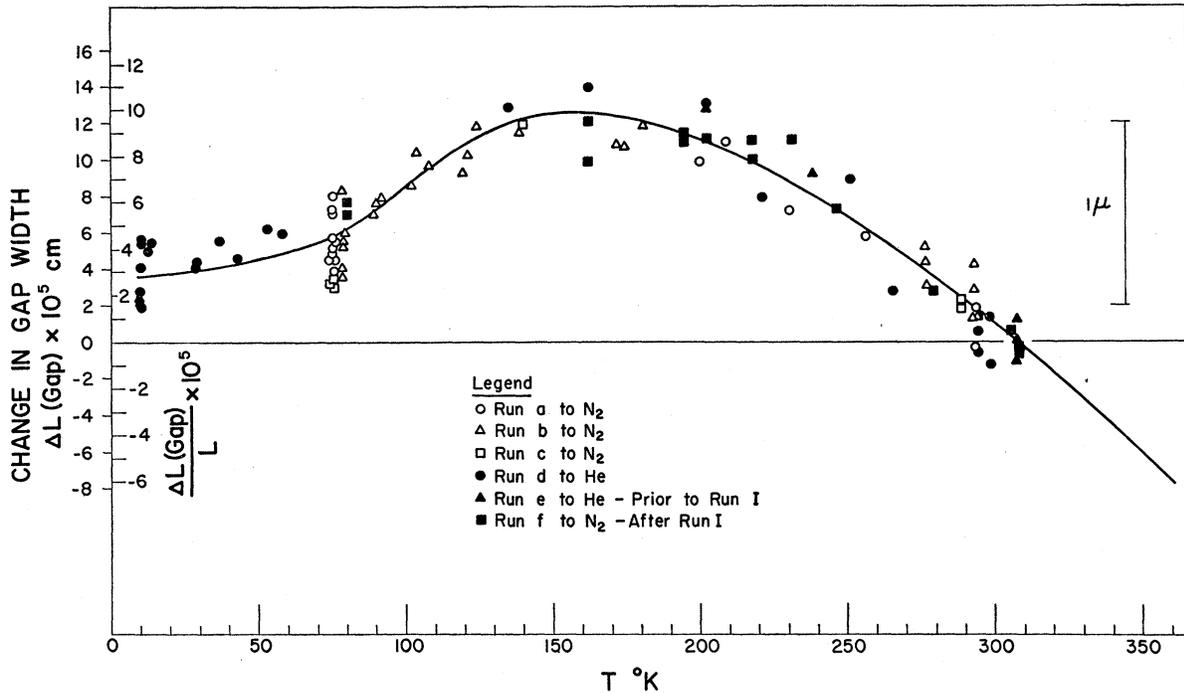


FIG. 4. Change in gap width vs temperature (Nonirradiated).

effects to give the actual length changes associated with the annealing.

Unless suitable precaution was taken, it was possible to condense sufficient vapor on the specimens during the runs to obscure the reference marks. The condensation problem was particularly difficult during annealing, since pressure bursts occurred during the warmups from low temperature. These bursts were associated with the liberation of condensed vapors from the cold metal surfaces when certain critical evaporation temperatures were reached. To prevent rapid condensation on the specimen during bursts and to prevent slow condensation over long periods at low temperature, it was necessary to surround the crystal with the cold radiation shields at the block temperature and at liquid nitrogen temperature shown in Figs. 1 and 2. The doors in these shields were opened only to make optical measurements when the cryostat pressure was  $<5 \times 10^{-5}$  mm of Hg. Several other precautions were taken. A 0.001-inch-thick Dural foil was used to separate the cryostat vacuum from the cyclotron thereby keeping out vapors from the cyclotron pumps and cyclotron arc. High-speed pumps were also used on the cryostat to reduce the pressure bursts.

### 3. Resistivity Measurement

The resistance was measured by a standard four-pole potentiometric method using field strengths of  $<5$  volts/cm. The resistances of two specimens designated  $R_n$  and  $R_p$  were measured on bombardment and during subsequent annealing. Small-diameter Kovar wires

were used as leads and soldered to the specimens. In the case of the  $R_n$  specimen, an antimony-doped tin solder was used. In the case of the  $R_p$  specimen, pure indium solder was used. The purpose of the different solders was to insure ohmic contact to at least one of the specimens at all times. However, it was found later that the difference in the type of solder had no effect on the resistance measurements. Although the specimens before irradiation were intrinsic at room temperature, they were  $n$ -type at low temperatures with approximately  $5 \times 10^{13}$  donors/cm<sup>3</sup>. Upon bombardment and annealing the specimens were converted to  $p$ -type. The sign of the majority carrier was determined by the sign of the photo emf between the tip of the temperature monitor specimen and the block. Because of the expected conversion, the electrical contacts were placed in the irradiated section of the specimen in order to eliminate internal barriers. Satisfactory ohmic contacts were obtained at low temperatures before irradiation. Upon irradiation and during subsequent annealing the contacts became excellent for both specimens at all temperatures.

### 4. Crystal Preparation and Mounting

The small range of the 12-Mev deuterons in germanium precluded the use of massive samples since homogeneously damaged specimens were desired. Specimens were cut from a germanium single crystal obtained from the Eagle Picher Company. They were ground, mechanically polished, and cut plane and parallel to a bar shape approximately 2.3 cm long,

0.046 cm wide, and 0.011 cm thick. The surfaces were then polished to a mirror finish. The resistance specimens were then given a light etch in CP-4 (without the bromine) to remove a possible disturbed surface conducting layer. The length specimens were left unetched to protect the polished surface and reference marks. X-ray diffraction line profiles were obtained from these unetched surfaces, and the resulting line widths appeared to be consistent with a high crystal perfection.<sup>10</sup> The specimen arrangement is shown in Fig. 1. The deuterons were incident normal to the specimen surfaces along the  $\langle 211 \rangle$  crystal direction. The longitudinal and lateral directions of the specimens were  $\langle 110 \rangle$  and  $\langle 111 \rangle$ , respectively. Because of the fragility of the length specimen it was impractical to measure the temperature by direct attachment of a thermocouple. Therefore, a temperature monitor specimen having the same geometry as the actual length specimen was mounted in an adjacent position (Fig. 1). The block was first annealed and lightly nickel plated and the two specimens were then attached with pure indium solder insuring good rigidity and excellent thermal contact. After soldering, the 0.015-cm-wide transverse gaps were cut through by means of a micro-sandblaster. An annealed copper-constantan thermocouple was then soldered to the tip of the temperature monitor with pure indium. The tip of the monitor containing the thermocouple was shielded from the deuteron beam by a small copper shield. The two resistance specimens were mounted on the block and insulated by applying a thin coat of Krylon, then a 0.0005-inch-thick sheet of mica, and then attaching the specimens with another thin Krylon coat.

### 5. Temperature Measurement

The annealed copper-constantan thermocouple on the temperature monitor was calibrated in place *vs* a Leeds & Northrup platinum resistance thermometer which was completely imbedded and glued into the molybdenum block. This platinum resistor was previously calibrated against a similar one calibrated by the National Bureau of Standards. All temperatures listed in this paper were obtained from this thermocouple. It is possible that the temperature of the resistance specimens was a few degrees higher than the temperature monitor, since the thermal contact of the soldered monitor was somewhat better. These differences were not judged to be of any importance. The temperature of the monitor was a minimum with the beam off and both doors closed. When the beam was turned on the temperature rose a few degrees. Before the reference marks were photographed, the beam was turned off, the doors were opened, and the illuminator was turned on. During photographing the temperature rose several degrees relative to the beam-off, doors-closed temperature. In addition to these effects, the various temperatures increased gradually with increased irradiation as a result of the decreased thermal con-

ductivity of the specimen. These perturbing effects were greater in the lower-temperature, Run I. The maximum temperature reached at any point before the warmup in Run I was 26.6°K. The corresponding maximum temperature in Run II was 85.0°K.

During the irradiation and annealing the block containing the specimens formed a completely closed radiation shield all at essentially the block temperature. The section of the shield through which the deuterons entered was a 0.0005-inch-thick aluminum foil. The opposite exit section of the shield was a 0.0005-inch-thick copper foil. The heat flow to the block at low temperatures with the beam off was primarily down the thermocouple wires, the platinum resistance thermometer leads, and the resistance specimen leads. To minimize this heat flow, small-diameter wires were used for leads and cooled to liquid nitrogen temperatures before being run to the block. The leads were also cooled to the block temperature before being led to the specimens. A heater was attached to the bottom of the block and was used during the annealing to give a uniform heating rate and to allow warming above room temperature.

### III. RESULTS

Length change and resistance measurements were made on the same set of specimens during two successive runs. In Run I liquid helium was used in the cryostat, and the specimens were bombarded at temperatures between 10°K and 25°K. Annealing was then carried out at temperatures up to 305°K. In Run II liquid nitrogen was put in the cryostat and the same specimens

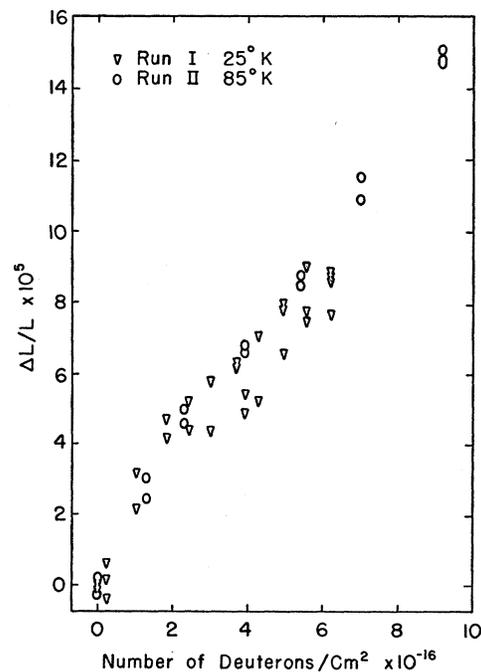


FIG. 5. Radiation expansion of germanium on bombardment.

were bombarded again at temperatures maintained between 80°K and 85°K. Annealing studies were then made up to 364°K.

1. Length

The specimens were observed to expand upon bombardment, and the specific length changes,  $\Delta L/L$ , during bombardment in Runs I and II are given in Fig. 5. The expansion appears to vary linearly with flux within experimental error, and the expansion per unit flux is the same for both runs. A value of  $\Delta L/L = (1.5 \pm 0.3) \times 10^{-21}$  per (deuteron/cm<sup>2</sup>) was obtained from the slopes of the bombardment curves.

After the Run I bombardment the specimens were annealed to room temperature (305°K). A constant heating rate of 5°/hr was used up to 85°K. Above 85°K the rate was increased to 10°/hr. Continual length change measurements were made during the warmup and are plotted in Fig. 6. Gaps in the length change data occur in temperature regions where pressure bursts prevented opening of the protective doors in the radiation shields. After reaching room temperature the specimens were cooled back to 10°K for

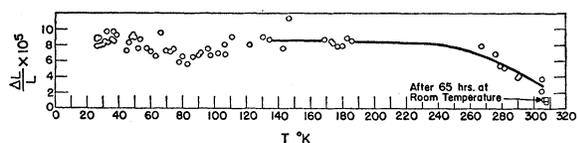


FIG. 6. Thermal recovery of irradiation-induced expansion (Run I, 25°K).

low-temperature measurements which confirmed the annealing observed in the warmup. The samples were then again warmed to room temperature and remained there for about 60 hours before being cooled to liquid nitrogen temperature for Run II. During this period 20% more annealing took place. The annealing procedure after Run II was somewhat more complicated. Warming and cooling rates of 10°/hr were used throughout. The specimens were warmed to 143°K, cooled back to 85°K, warmed to 364°K, and cooled back to 300°K. After being held for 1/2 hr at 300°K, the specimens were cooled back to 85°K for final low-temperature measurements. The annealing curve is shown in Fig. 7 and is quite similar to the Run I curve. Annealing is 50% complete at room temperature and 85% complete at 364°K (the temperature limit of our system).

In an ideal experiment of this type the specimens would be uniformly irradiated throughout and would be free to expand in all directions. It was impossible to meet these requirements exactly because of the finite thickness of the specimens, the restraints imposed by the clamping at the ends, and the nonuniformity of the cyclotron beam. These inhomogeneous effects are discussed in the following and are shown to be of minor importance.

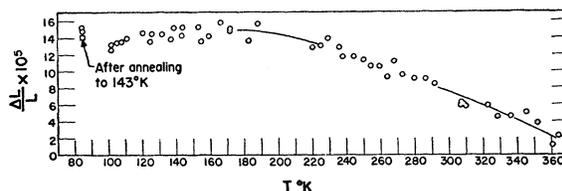


FIG. 7. Thermal recovery of irradiation-induced expansion (Run II, 85°K).

The energy of the deuterons dropped from 11.4 Mev to 9.0 Mev upon traversing the specimen. If the damage varies as  $1/E^{1.11}$  then the specimens should have become bent<sup>11</sup> due to the differential expansion between front and back. The error in the length measurement due to this effect is estimated to be  $7 \times 10^{-7}$  cm and is, therefore, negligible. The effects due to the constraints at the clamped ends were also very minor. The specimens were free to expand in the measuring direction, and any elastic strains would have been localized at the clamped regions which constituted only a small fraction of the total length. The nonuniformity of the cyclotron beam was determined before bombardment, and it was found that the specimen length was approximately equal to the beam width at one-half maximum intensity. The gap between the two crystal halves was then positioned at the intensity maximum. The variations in the damage resulting from the nonuniformity of the beam along the specimen length would produce

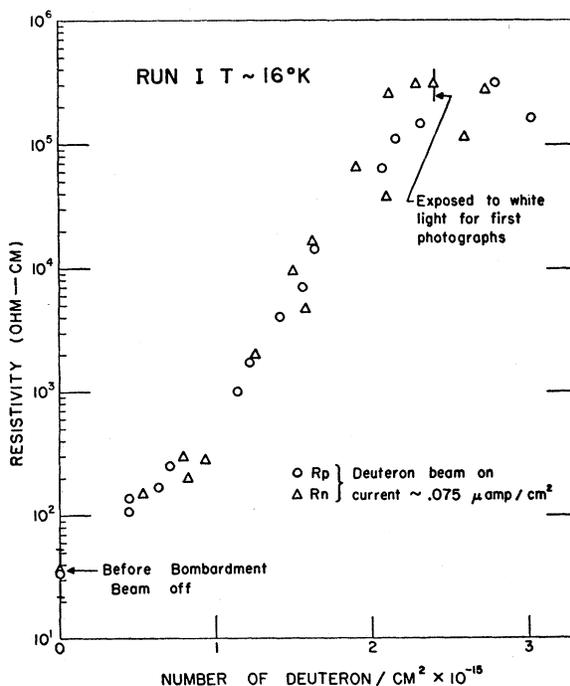


FIG. 8. Resistivity increase on bombardment (Run I, T ~ 16°K).

<sup>11</sup> R. O. Simmons and R. W. Balluffi, Phys. Rev. 109, 1142 (1958).

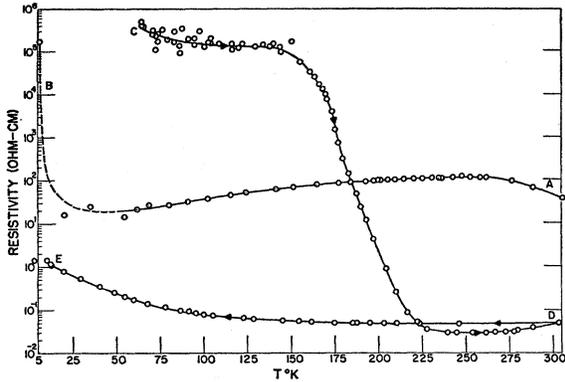


FIG. 9. Thermal recovery of resistivity (Run I,  $R_p$ ).  $AB$ =resistivity prior to irradiation,  $CD$ =initial warmup,  $DE$ =cool-down after irradiation.

only internal strains which should not have made any appreciable contribution to the integrated length changes.

## 2. Resistivity

The resistivity increase during the Run I irradiation is shown in Fig. 8. The resistance increased very rapidly and soon became too high to measure with the available apparatus and our requirement that the field strength be less than 5 volts/cm. Upon annealing to about 65°K the resistivity became measurable, and the annealing curve for the  $R_p$  specimen is shown in Fig. 9. The curve obtained by cooling back to the low temperature is also indicated and shows that a large irreversible resistivity annealing took place during the warming. The resistivity of the  $R_n$  specimen showed identical behavior. The photovoltage was first measured during the warming at 180°K, and the specimens were found to have been converted to  $p$ -type. Subsequent measurements indicated that the specimens remained  $p$ -type from then on. The corresponding curves for Run II are shown in Figs. 10 and 11. The resistance rose rapidly during bombardment and reached a value which seemed

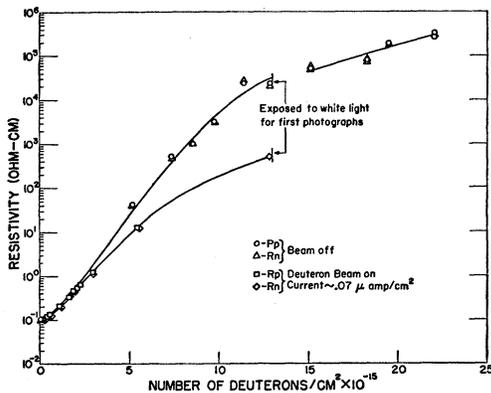


FIG. 10. Resistivity increase on bombardment (Run II,  $T \sim 80^\circ\text{K}$ ).

to barely exceed measuring capabilities. An appreciable difference was observed between the resistance measured with the cyclotron beam on and the beam off. This effect was particularly noticeable for the higher resistivities and is shown in Fig. 10. The resistivity became readily measurable soon after bombardment upon annealing near 80°K.

The lowest-temperature resistance annealing observed in the present work was found in the irreversible resistance increase obtained in the Run II cycling between 85°K and 140°K (see Fig. 11). Upon further annealing the resistivity decreased irreversibly by a factor of  $10^3$  between 150°K and 200°K.

## IV. DISCUSSION

The result that germanium undergoes a length expansion during low-temperature deuteron bombardment is in qualitative agreement with the work of Wittels<sup>12</sup> where a lattice parameter expansion was

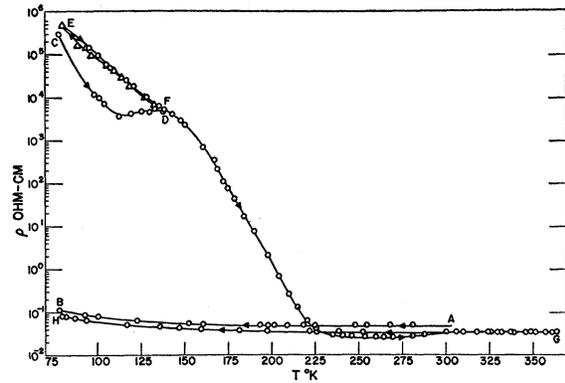


FIG. 11. Thermal recovery of resistivity (Run II,  $R_p$ ).  $AB$ =resistivity after Run I and annealing to room temperature,  $CD$ =initial warmup,  $DE$ =cool down 140°K–80°K,  $EF$ =second warmup 80°K–140°K,  $FG$ =continuation of warmup,  $GH$ =final resistivity of specimen after Run II anneal.

found after heavy reactor irradiation at 40°C and 140°C. However, it should be noted that length changes and lattice parameter changes are not necessarily equal (or even of the same sign).<sup>8,11</sup> The failure of Kleitman and Yearian<sup>13</sup> to observe an irradiation-induced length expansion in germanium was probably due to the relatively small magnitude of the effect and the fact that their measurements were made at room temperature. The irradiation-induced length change in germanium is generally similar to that reported for the close-packed metal copper with regard to the sign and magnitude of the effect and is quite different from results reported for III-V compounds having the diamond structure. Copper shows a length expansion<sup>8</sup> which is about twice as large as the present result for germanium. The III-V compounds reportedly contract

<sup>12</sup> M. C. Wittels, J. Appl. Phys. 28, 921 (1957).

<sup>13</sup> D. Kleitman and H. J. Yearian, Phys. Rev. 108, 901 (1957).

under certain conditions, and the length changes are larger by at least an order of magnitude.<sup>13,14</sup>

Possible anisotropy in the expansion is not excluded by the present experiment, since only the expansion in the (110) direction was measured. A variation in damage with bombardment direction in germanium has been found for electrons near the threshold energy.<sup>15</sup> However, these variations disappeared on room-temperature irradiation at higher energies. It appears likely that such effects were small in the present experiments. The bombardment curves appear linear within probable experimental error.

The annealing curves indicate that, within the experimental error of about 20%, no annealing of the irradiation-induced expansion occurs below 200°K. The bombardment curves are in agreement with this result since irradiation at the two different temperatures produced the same expansion per unit flux within this experimental error. Above 200°K very gradual annealing becomes perceptible and continues to above room temperature. At room temperature a small residual expansion remains. X-ray lattice parameter measurements, discussed in the following paper,<sup>10</sup> also reveal a small residual lattice parameter expansion at this temperature. It is likely that an appreciable annealing out of the length change corresponds to an annealing out and annihilation of irradiation-induced defects. In the case of copper it is currently believed<sup>8,11,16</sup> that the annealing of the irradiation-induced length expansion is due to the mutual annihilation of vacancies and interstitials. A detailed discussion of possible models for the radiation damage in germanium is presented in the following paper.<sup>7</sup>

The resistivity measurements agree generally with previous results.<sup>5</sup> The initially *n*-type specimens were converted to *p*-type upon bombardment and annealing, and the resistivity increased by several orders of magnitude on bombardment. The resistivity increase was unusually large in the present work because of the relatively high purity of the undoped specimens, and it was not possible to measure the resistance throughout the entire experiment. Unfortunately, the gaps in our resistivity data do not allow us to conclude whether resistivity changes occurred in the annealing range between 26°K and liquid nitrogen temperature. Other

workers have reported measurements in this range after other types of bombardment. Cleland and Crawford<sup>17</sup> have observed comparatively little resistivity annealing below 90°K after fast-neutron bombardment of chemically doped *n*-type germanium. Gobeli<sup>18</sup> has lightly irradiated degenerate *n*- and *p*-type germanium at 4.2°K using 3.73-Mev alpha particles and has observed two distinct annealing processes which occur in the vicinity of 32°K and 66°K. At 78°K recovery of the carrier concentration was 25% complete for *n*-type and 22% complete for *p*-type. These results, however, are not necessarily in contradiction as pointed out in a footnote added to reference 16, since the results may be sensitive to the doping and to the type and amount of irradiation used. The resistivity increase due to annealing in the temperature range 85°K to 140°K and the resistivity decrease in the range 150°K to 200°K have been observed by others.<sup>5</sup>

Of major interest is the correlation of the resistivity changes with the length changes and with the structure of the bombarded and annealed material. A result of interest is the observation that irreversible resistivity changes occur during annealing at temperatures ( $T < 135^\circ\text{K}$ ) which are considerably lower than the temperatures at which appreciable length change annealing occurs ( $T > 200^\circ\text{K}$ ). Further discussion of these phenomena and a proposed model of the damage are given in the following paper.<sup>7</sup>

#### ACKNOWLEDGMENTS

The stimulating discussions, helpful advice, and suggestion of the problem by Professor J. S. Koehler are gratefully acknowledged. Particular thanks are due to Dr. Richard W. Vook for helpful advice and discussions in the preparation of the experiment. Dr. R. O. Simmons and Mr. T. Nilan provided invaluable assistance in the performance of the experiment. Members of the Illinois radiation damage group and the Illinois cyclotron staff aided generally in carrying out the experiment. F. L. Vook held a fellowship supported by the United States Steel Corporation during the major portion of the experiment, which also received support from the U. S. Atomic Energy Commission.

<sup>14</sup> U. Gonser and B. Okkerse, Phys. Rev. **109**, 663 (1958); Bull. Am. Phys. Soc. Ser. II, **2**, 157 (1957).

<sup>15</sup> W. L. Brown and W. M. Augustyniak, Bull. Am. Phys. Soc. Ser. II, **2**, 156 (1957).

<sup>16</sup> Magnuson, Palmer, and Koehler, Phys. Rev. **109**, 1990 (1958).

<sup>17</sup> J. W. Cleland and J. H. Crawford, Jr., J. Appl. Phys. **29**, 149 (1958).

<sup>18</sup> G. W. Gobeli, Bull. Am. Phys. Soc. Ser. II, **2**, 355 (1957); Phys. Rev. **112**, 732 (1958).

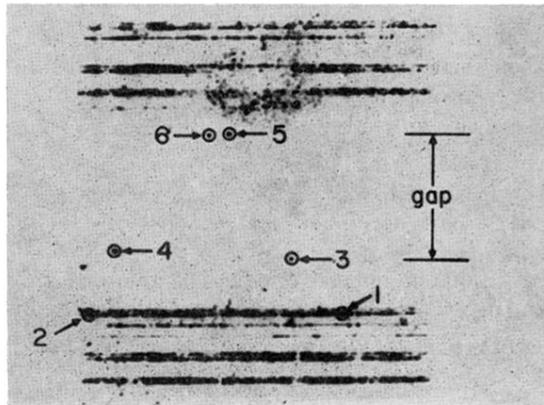


FIG. 3. Reproduction of typical photographic plate.