Change in Thermal Conductivity Upon Low-Temperature Electron Irradiation: InSb†

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(Received 20 March 1964)

Measurements of the change in low-temperature thermal conductivity of high-purity single crystal InSb were made upon 2-MeV electron irradiation and annealing. For small defect concentrations, the additive thermal resistivity increases as the $\frac{3}{4}$ power of the time-integrated flux Φ rather than the first power as observed for GaAs. The defect thermal resistivity is not proportional to the absolute temperature as predicted by the strain and mass-difference scattering theories of Klemens and Ziman. Irradiation to 5×10^{17} e/cm^2 below 52°K gave $1/K-1/K_0=2.7\times10^{-13}\,\Phi^{\frac{3}{4}}$ cm-deg/W at 18°K. A similar result was obtained for measurements at 50°K. Isochronal anneals with measurements of thermal and electrical conductivity made near 18 and 50°K indicated almost complete recovery of the thermal conductivity by 395°K. The increase in thermal resistivity on bombardment at 50°K is orders of magnitude larger for InSb than for GaAs even though InSb and GaAs exhibit the same increase in lattice strain per 2-MeV e/cm^2 at 50°K. It would have been expected that GaAs would be more sensitive to point defects than InSb since GaAs is less limited by umklapp scattering at 50°K. The strain and mass-difference scattering theories of Klemens and Ziman can not explain the InSb data. The Keyes electron-phonon scattering theory based upon the large effect of strain on the defect electronic wave functions gives a plausible explanation. Annealing begins as low as 40°K for n-type InSb converted to p type on bombardment, whereas it has been reported that no annealing occurs below 80°K in unconverted n-type InSb. "Reverse anneals" occur in both thermal and electrical conductivity, implying the existence of at least two competing annealing processes. The activation energy for the anneal of thermal conductivity between 85 and 105°K agrees with that for the annealing of carrier concentration in p-type InSb, suggesting that the defects measured by electrical and thermal conductivity are the same.

I. INTRODUCTION

THE purpose of the present paper is to report the changes in thermal conductivity of InSb upon the low-temperature introduction and annealing of point defects produced by 2-MeV electron irradiation. In the preceding paper,[1] hereafter referred to as I, it was shown that the observed linear increase in low-temperature thermal resistivity of GaAs upon electron irradiation can be adequately related to the measured linear increase in lattice strain[2] through the theories of mass difference and strain field scattering of Klemens[3] and Ziman.[4] The very much larger increase in thermal resistivity of InSb cannot be related through the same theories to the observed increase in lattice strain.[2] Following a description of the experiments and presentation of the results, theoretical considerations are given to explain the quite different behavior of InSb as resulting from a phonon scattering mechanism, such as proposed by Keyes,[5] based on the large effect of strain on impurity-electron wave functions. Simultaneous isochronal annealing measurements of electrical and thermal conductivity, showing fine structure and "reverse annealing," are presented and compared to previous measurements of annealing in electron irradiated InSb.[2,6,7]

† This work was supported by the U. S. Atomic Energy Commission. Reproduction in whole or in part is permitted for any purpose of the U. S. Government.

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[3] P. G. Klemens, Proc. Phys. Soc. (London) **A68**, 1113 (1955); *Solid State Physics* edited by F. Seitz and D. Turnbull (Academic Press, Inc., New York, 1958), Vol. 7, p. 1.

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II. EXPERIMENTAL

Measurements of the change in thermal conductivity of InSb were made *in situ* without warmup at base temperatures of 18 and 50°K. The experimental arrangement and measuring apparatus were similar to that reported in the preceding paper. The sample was soldered at one end to a sample block which in turn was conduction cooled in an irradiation cryostat. Measurements of the change in thermal conductivity on 2-MeV electron bombardment were made using either the ionization heat input of the electron beam or the heat of a small wire heater attached to the end of the sample. Measurements of both electrical and thermal conductivity were made on annealing.

The sample was fabricated from single crystal material obtained from Ohio Semiconductor. The sample was initially n type with a carrier concentration of 1×10^{15} cm^{-3} and mobility of 3×10^5 cm^2/V-sec at 80°K. The bar-shaped sample was 0.17 cm wide, 0.0463 cm thick, and the irradiated length was 1.0 cm. The sample was irradiated in the ⟨111⟩ direction. The long dimension was the ⟨110⟩ direction.

For the beam ionization method, which was used only on bombardment, the increase in thermal resistivity is given by Eq. (1) of I. Measurements were made for beam-current densities of 0.91 and 1.36 μA/cm^2 corresponding to total beam currents of 1.0 and 1.5 μA, respectively. Heater measurements were made both on bombardment and annealing. Equation (3) of I gives the increase in thermal resistivity for the heater measurements. The fraction of the heater power which flowed through the sample was obtained by calibrating the heater at 50°K, using the value of $K_0=2.5$ W/cm-deg

[7] L. W. Aukerman, Phys. Rev. **115**, 1125 (1959).

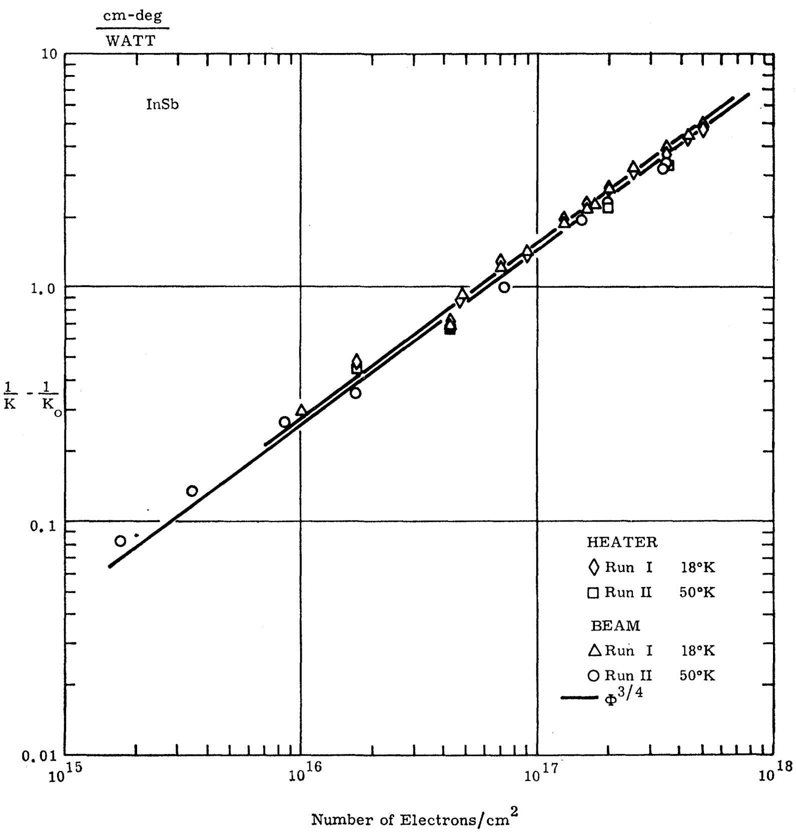

FIG. 1. Increase in thermal resistivity on bombardment for both the beam and heater measurements; run I 18°K, run II 50°K. The sample heater was calibrated for run II such that $K_0=2.5$ W/cm-deg at 50°K. The heater was calibrated at 18°K for run I using the ionization heat of the electron beam.

given by Holland.[8] The results obtained using the heater calibrated in this way agreed very well with those obtained independently using the beam heating method.[9] They imply that there is no large change in the thermal conductivity of InSb when ionization is introduced by the electron beam. The beam measurements give the best absolute measurements of the increase in thermal resistivity on bombardment. At 18°K the heater was therefore calibrated using the known heat input of the electron beam.

During the first irradiation the base temperature of the sample was near 18°K. The sample was irradiated with such a flux that the maximum temperature at the sample tip was never more than 52°K. Measurements of the increase in thermal resistivity were made periodically using either the electron beam or the sample heater. After a total flux of 5.0×10^{17} e/cm^2, 15-min isochronal anneals were performed in 10°K steps to 80°K with the heater measurements made near 18°K. At this point a new reference point of 50°K (pumped nitrogen) was established and 15-min isochronal anneals were performed in smaller steps to 395°K. By this temperature all the additive thermal resistivity measured at 50°K had annealed out within the accuracy of the measurements. This first irradiation and anneal will be referred to as run I. The sample was then cooled to 50°K and reirradiated to an additional flux of 3.5×10^{17} e/cm^2 with the maximum temperature kept below 80°K. The sample was then annealed in 15-min isochronal anneals to 410°K. This second bombardment and anneal will be referred to as run II. The sample was again cooled to 50°K and reirradiated (run III) to an additional flux of 4.47×10^{17} e/cm^2, keeping the maximum irradiation temperature below 80°K. Fifteen-min isochronal anneals were performed in 1°K steps to 124°K with measurements made again of both electrical and thermal conductivity.

III. RESULTS

The increase in thermal resistivity on bombardment is shown in Fig. 1. These data were obtained using the ionization heat input of the electron beam with $H=J(\mu\text{A/cm}^2)(\Delta E/\Delta x)(\text{MeV/cm})$. The average value used for $\Delta E/\Delta x$ for InSb was 10.8 MeV/cm obtained using the range energy formula of Katz and Penfold[10] and the known thickness of the sample. For all bombardment runs the additive thermal resistivity increased as the $\frac{3}{4}$ power of Φ, the number of electrons/cm^2 passed

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[9] Preliminary accounts of these data were reported in Bull. of Am. Phys. Soc. **8**, 209 (1963). The values of thermal resistivity reported there for the heater measurements are too low due to an over estimation of the heater input power.

[10] L. Katz and N. Penfold, Rev. Mod. Phys. **24**, 28 (1952).

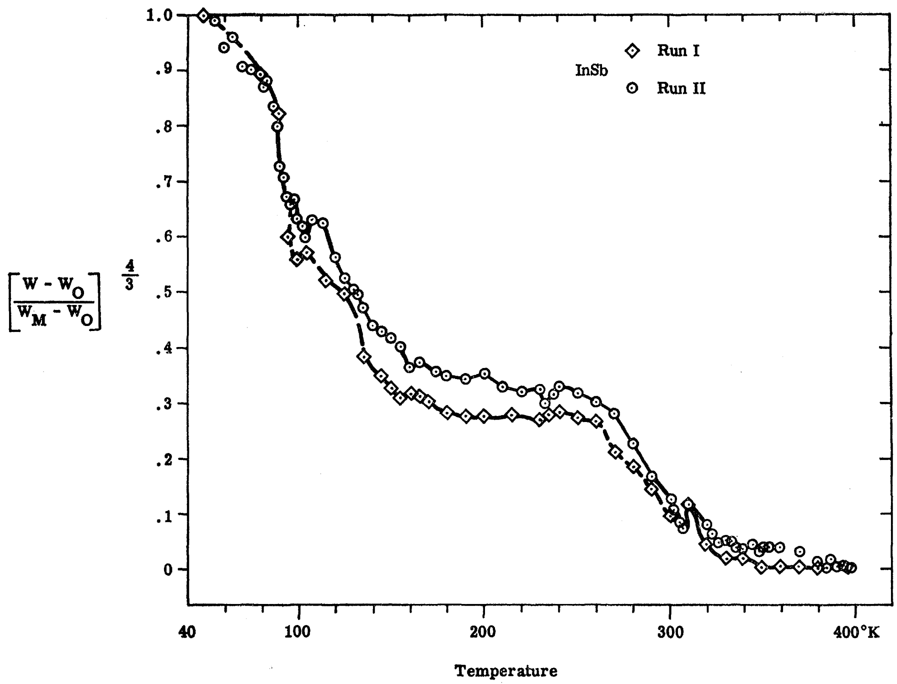

FIG. 2. Normalized 15-min isochronal anneals of thermal resistivity for runs I and II. All measurements made at 50°K.

through the sample. Using the above analysis, the beam data can be fit very well by the following equations:

$$\frac{1}{K}-\frac{1}{K_0}=2.7\times10^{-13}\Phi^{3/4}\text{ cm-deg/W for run I (18-K)},$$

where Φ is in units of 2.0-MeV electrons/cm^2,

$$\frac{1}{K}-\frac{1}{K_0}=2.5\times10^{-13}\Phi^{3/4}\text{ cm-deg/W for run II (50-K)}.$$

The increase in thermal resistivity on bombardment obtained using the sample heaters agreed with the above values obtained using the beam. These heater data also are plotted in Fig. 1. Although the beam data are most accurate on bombardment, simple annealing data can only be obtained using the relative changes provided by the heater measurements.

1. Annealing

The thermal resistivity increase is directly proportional to the $\frac{3}{4}$ power of the integrated flux which in turn is assumed to be proportional to the concentration of defects. If it is also assumed that annealing proceeds such that the thermal resistivity maintains this concentration dependence, then $[(W-W_0)/(W_m-W_0)]^{4/3}$ gives the fraction of the damage which is unannealed. Here W_m is the maximum thermal resistivity observed at the end of the bombardment and the start of the annealing. Annealing begins as low as 40°K. Approximately 5% of the added thermal resistivity as measured at 18°K anneals below 50°K. Figures 2 and 3 show the damage remaining at 50°K after 15-min isochronal anneals at higher temperatures for runs I and II. Figure 4 shows the corresponding simultaneous isochronal annealing curves for electrical conductivity for runs I and II. Figure 5 shows the detailed isochronal anneals of thermal and electrical conductivity for run III.

IV. DISCUSSION

1. Bombardment

Figure 6 compares the results reported in this paper for InSb with those reported in I for GaAs. The first observation is the much larger nonlinear thermal resistivity per 2-MeV electron/cm^2 observed for InSb than for GaAs.[11] This is in spite of the fact that at 50°K, InSb is more limited by umklapp scattering than GaAs[8] and should therefore be *less* sensitive to point defects than GaAs. In addition, InSb and GaAs exhibit the *same* linear increase in lattice strain[2] per 2-MeV electron/cm^2 at 50°K, indicating that a different scattering mechanism is applicable for InSb. The second observation is the dependence of the additive thermal resistivity on the three-quarter power of the defect concentration rather than the first power observed for

[11] A GaAs sample (G-2, similar to G-1 and G-3 of I) was simultaneously irradiated with the InSb sample. The large disparity in damage, however, made it impractical to investigate both InSb and GaAs in the same experiment. When the thermal resistivity of the InSb sample had increased to the point where the resulting temperature rise on bombardment was large, the change in GaAs was still very small. The measurements of G-2 were therefore discontinued.

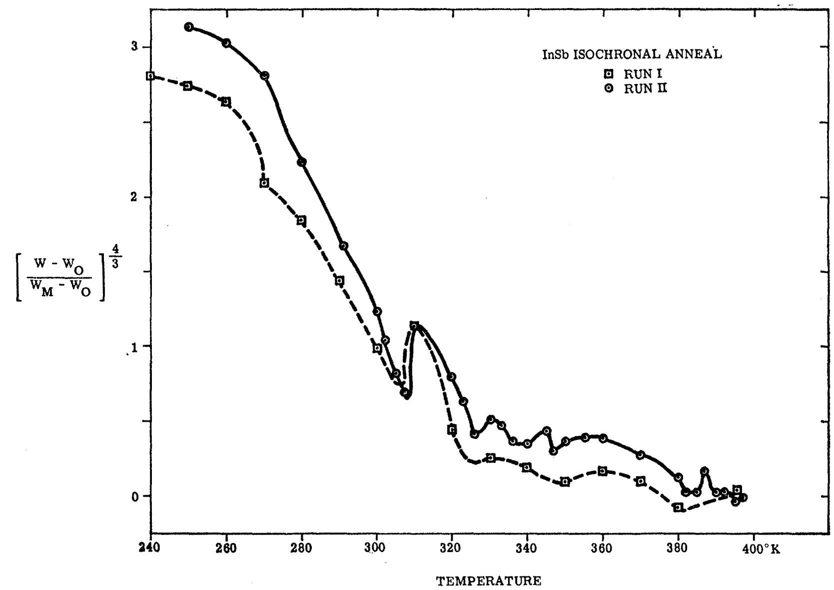

FIG. 3. Isochronal anneals of thermal resistivity near room temperature for runs I and II showing "reverse annealing." All measurements made at 50°K.

GaAs. The third observation is that the additive thermal resistivity, as measured at 18 and 50°K, was temperature-independent and not directly proportional to the absolute temperature as predicted by Klemens for point-defect scattering.

Low-temperature measurements of lattice strain[2] show that electron irradiations of InSb and GaAs produce approximately the same linear increase in length per 2-MeV electron/cm^2. Estimates of the average displacement threshold energies have been made from measurements of strain rate as a function of incident electron energy. These in turn provide a means by which the number of defects can be estimated using a displacement theory such as that given by Seitz and Koehler.[12] The results of such calculations are that the lattice strain per defect is approximately one atomic volume for both InSb and GaAs.

The increased thermal resistivity of GaAs could be explained on the basis of the Klemens' theory[3] which includes the effects of mass difference and increased lattice strain. A similar treatment for InSb, using the strain results, would predict a closely comparable defect thermal resistivity, linear with defect concentration, and directly proportional to the absolute temperature. The strong disagreement of these predictions with observation implies that there must be some other dominant scattering mechanism.

Strong scattering of phonons by small concentrations of donors and acceptors has been observed for germanium[13–15] and interpreted as evidence for the scattering of phonons by carriers contained either in an impurity band[14] or localized on the impurity centers.[5] Keyes used a localized model,[5] and treated the scattering of phonons in terms of the large effect of strain on the impurity electron wave functions. He was able to semiquantitatively account for many of the unusual features of the thermal resistance of n-type germanium,[15] including the large difference between the scattering powers of antimony and arsenic, the strong thermal conductivity temperature dependence between 1.3 and 4°K, the observation that the scattering depends on the number of occupied donors rather than the total number of impurities,[15] and the unusual sensitivity to strains.[16,17] In addition, the same model has been quite successful in explaining the large electronic effects in the elastic constants of germanium[18] and silicon.[19] Recently, Griffin and Carruthers[20] have treated the thermal conductivity of germanium in terms of the resonance fluorescence scattering of phonons by bound donor electrons. Apart from anomalous effects due to resonance scattering, their results are similar to the previous work by Keyes.

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[15] J. F. Goff and N. Pearlman, *Proceedings of the Seventh International Conference on Low Temperature Physics*, edited by G. M. Graham and A. C. Hollis Hallet (University of Toronto Press, Toronto, 1961), p. 284; also J. F. Goff Ph.D. thesis, Purdue University, 1962 (unpublished).

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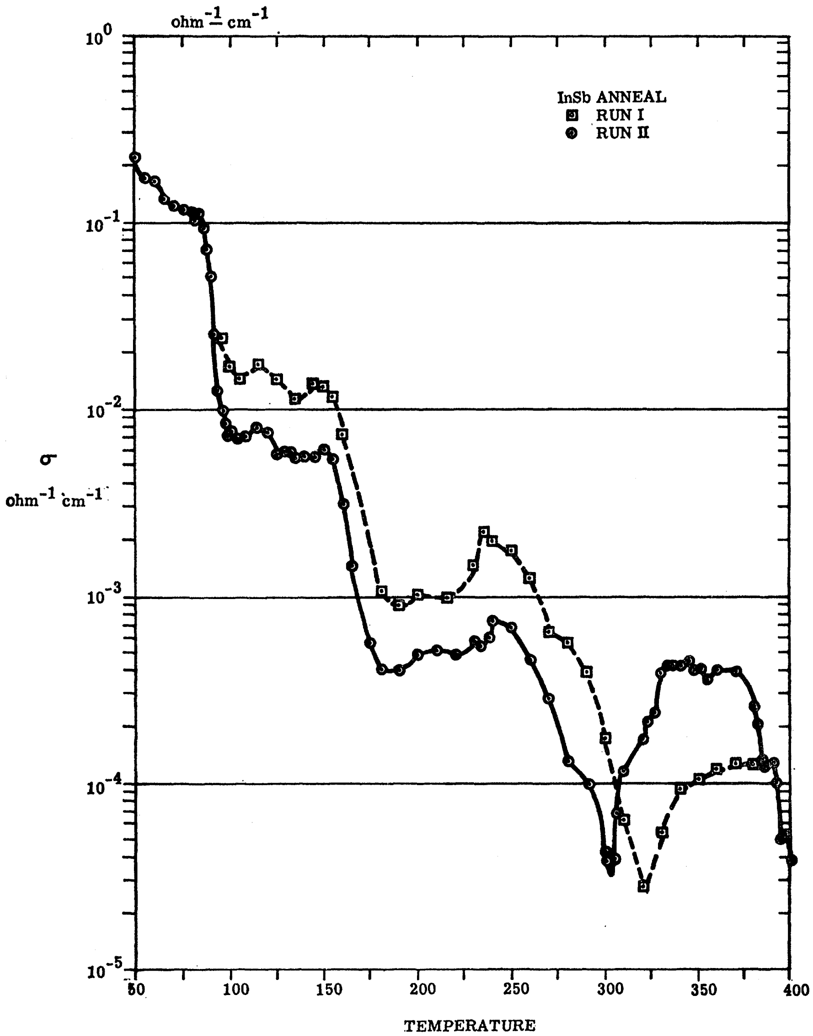

FIG. 4. Isochronal anneals of electrical conductivity for runs I and II. All measurements made at 50°K.

A necessary condition for the Keyes mechanism to be applicable is a large change in defect electron wave functions with strain. Keyes states that where there is a dependence of the electronic levels on strain, then the strain energy function of the crystal will depend on the occupation of the electronic levels. He further concludes that the interaction between the elastic strain and the occupied electronic levels is responsible for the scattering of phonons.

This condition would require a nonspherical band structure for a large strain dependence. The conduction band of InSb is spherical with a minimum at $k=0$.[21] Although there is no detailed quantitative information on the form of the valence band of InSb, the experimental results appear to be consistent with a valence band structure similar to germanium or silicon, i.e., warped spheres. Kane[22] used a perturbation method together with experimental results to derive a theoretical model for the band structure of InSb. Since the energy gap is so narrow in InSb, the interaction between the conduction and valence bands is not small compared with the energy separation between them. Kane treated the interaction of the conduction and valence bands directly. This interaction splits the sixfold degeneracy of the valence band into three twofold degenerate bands: a "heavy hole" and a "light hole" band degenerate at $k=0$, and a band split-off by spin-orbit interaction. The interaction with higher bands is treated by second-order perturbation theory. Near $k=0$ the remaining twofold degeneracy of the valence band is split by an amount proportional to k. Therefore, at $k=0$ there is a heavy-hole band degenerate with a light-hole band, and a further band split by spin-orbit interaction. The maxima of the heavy-hole band lie slightly away from the center of the zone. Elastoresistance measurements are consistent with this

[21] C. Hilsum and A. C. Rose-Innes, *Semiconducting III–V Compounds* (Pergamon Press, Inc., New York, 1961), p. 37.
[22] E. O. Kane, Phys. Chem. Solids **1**, 249 (1957).

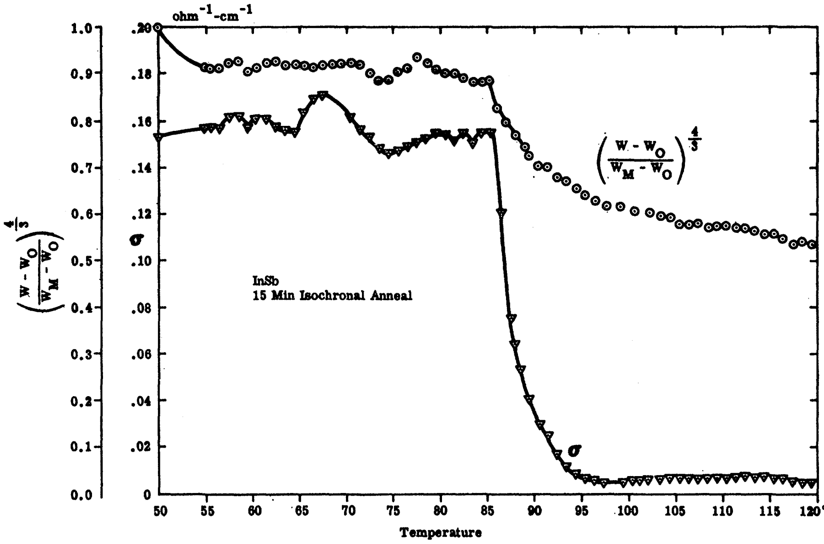

FIG. 5. Comparison of electrical and thermal conductivity annealing for run III. All measurements made at 50°K.

picture. The elastoresistance shear coefficients[23] for p-type InSb determined by the piezoresistance measurements of Potter[24] performed at 77°K are very large: $m_{44}=\pm 101.0$ and $(m_{11}-m_{12})/2=+21.0$ (for n-type InSb $m_{44}=-1.3$ and $(m_{11}-m_{12})/2=-1.1$).

The valence band of InSb is of particular interest, since the sample was rapidly driven p type before the first bombardment measurements were taken and remained p type until near room temperature on annealing. Since the defect levels are near the valence band which is known to be greatly strain-dependent, it is reasonable that the energy of the defect levels should be strain-dependent, thereby producing the large observed phonon scattering.

One may qualitatively understand why GaAs behaves differently from InSb. GaAs has a large energy gap rather than the very small one of InSb. On bombardment GaAs becomes intrinsic rather than p type as InSb does. The elastoresistance measurements of Sagar[25] at 300°K give $m_{44}=-1.4$ and $(m_{11}-m_{12})/2=0.5$ for n-type GaAs, consistent with a spherical conduction band having a small strain dependence. No measurements are available for p-type GaAs. There is no evidence therefore that the defect levels should be greatly strain-dependent. The present data agree with the tentative conclusion that they are not and that therefore the Keyes mechanism is not the dominant phonon scattering mechanism for GaAs.

Theoretical studies to explain the concentration dependence of isotope (mass difference) scattering have been made by several authors[3,4,26–29] and reviewed in I. Most theories predict that the defect thermal resistivity is directly proportional to the concentration of defects for small concentrations. Our data for InSb, on the other hand, exhibit a quite definite $\frac{3}{4}$-power dependence. Further support for the Keyes mechanism which we have discussed above is that it predicts[5] a less than linear concentration dependence which is close to 0.75. The data of Goff and Pearlman,[15] interpreted by them on the basis of the Keyes' theory, indeed show a closely $\frac{3}{4}$-power dependence of the phonon-electron thermal resistivity on the exhaustion carrier concentration becoming proportional to the 0.5 power at large concentrations. As a point of interest, a 0.75-power dependence has also been observed for x-ray irradiated KCl (0.6 for KBr) by Gebhardt.[30]

2. Annealing

Measurements of the changes in electrical conductivity and Hall coefficient of electron irradiated n- and p-type InSb have been reported by Eisen[6] and Aukerman.[7] Eisen found that the damage recovered in five well-defined stages with the recovery nearly complete at 320°K. There was evidence that the two lowest temperature recovery stages involved the annihilation of close interstitial-vacancy pairs and that interactions

[23] The elastoresistance coefficients m are defined in terms of the fractional change in electrical resistivity $\Delta=(\delta\rho)_{ij}/\rho$ and the homogenous strain $\boldsymbol{\varepsilon}$ by the equation $\boldsymbol{\Delta}=\mathfrak{m}\cdot\boldsymbol{\varepsilon}$. For cubic crystals the elastoresistance results can be reduced to two shear coefficients, m_{44} and $\frac{1}{2}(m_{11}-m_{12})$. The change in resistivity measured along a [110] direction, produced by a shear corresponding to an elongation along this direction and a contraction along the perpendicular [$\bar{1}$10] is m_{44}. Similarly $\frac{1}{2}(m_{11}-m_{12})$ is related to the [100] directions. See also Ref. 21, p. 38.

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[27] J. Callaway, Phys. Rev. **110**, 585 (1958); *Proceedings of the International Conference on Semiconductor Physics, Prague, 1960* (Academic Press Inc., New York, 1961), p. 627.

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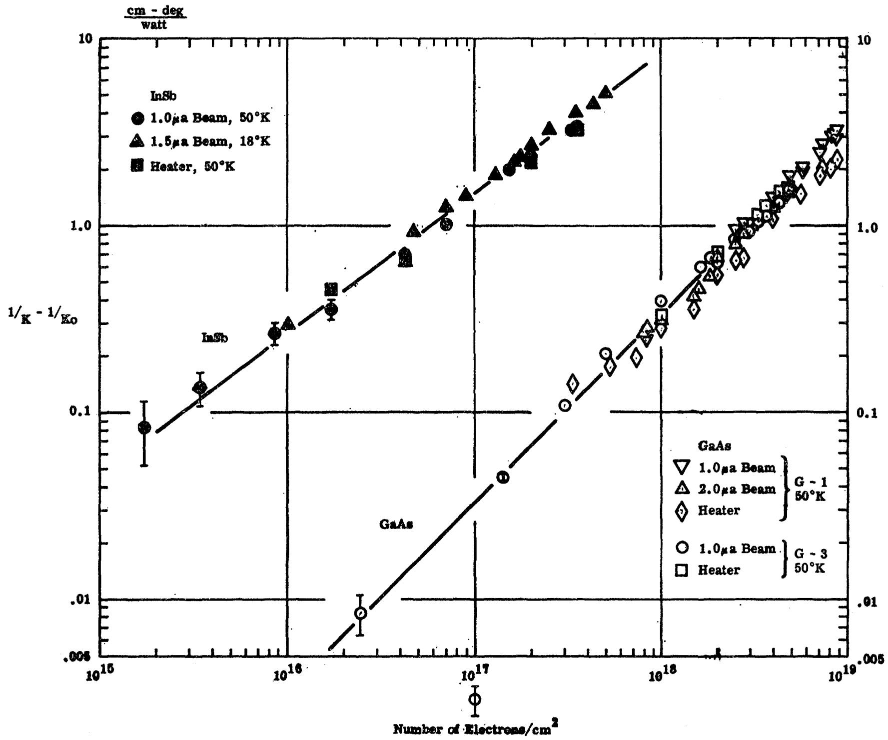

FIG. 6. Comparison of the change in thermal resistivity of InSb and GaAs upon 2-MeV electron irradiation.

of primary defects with impurities do not occur. However, since first-order kinetics expected for close-pair recovery was not explicitly observed, he proposed a mechanism involving the independent annihilation of two types of close pair configurations in the same stage with an electrostatic interaction between the interstitial and vacancy.

For *p*-type InSb, Eisen[6] found that following irradiation at 78°K, which increased the resistivity and decreased the Hall coefficient, recovery occurred in six regions with center temperatures at 87, 103, 157, 235, 305, and 385°K. Reversals in annealing of conductivity and Hall coefficient occurred at 87, 157, and initially at 235°K. The hole mobility, however, increased in all regions of recovery. Eisen found that his "stage II" recovery (80–105°K) gave activation energies between 0.25 and 0.28 eV depending on the initial hole concentration. Reversals were also seen by Aukerman[7] in the annealing of carrier concentration of electron irradiated *p*-type InSb. These reversals correlated very well with similar reversals in the annealing of length change measurements of *n*-type InSb which was converted to *p*-type by low-temperature electron irradiation.[2]

The electron irradiations reported here are fairly heavy compared with those of Eisen. On bombardment at 50°K the electrical conductivity increases following conversion from *n* to *p* type and saturates near 0.2 Ω^{-1} cm^{-1} for fluxes greater than 2×10^{17} e/cm^2 as the energy levels of the introduced defects pin the Fermi level. The thermal conductivity however continues to decrease without sign of saturation as phonon scattering continues to increase with the addition of neutral acceptors. Our thermal and electrical conductivity annealing results agree fairly well with the results of Eisen above 78°K for initially *p*-type samples. The data of run I show annealing beginning at about 40°K with a sharp drop beginning at 85°K. It is conceivable that with a lower irradiation temperature, annealing might begin even below 40°K in *p*-type InSb. Upon annealing as the thermal conductivity recovers and increases, the electrical conductivity decreases. Several reversals in annealing are shown in Figs. 2–4 for both thermal and electrical conductivity particularly near 105 and 310°K. The reversals imply the existence of at least two competing annealing processes and may be due to defect reordering or clustering as seen in I.

Using an analysis of isochronal anneals proposed by Aukerman,[31] it was found that the thermal conductivity annealing in run III between 85 and 105°K gives a roughly second order process with an activation energy of 0.26 eV. This agrees with the values given by Eisen for annealing of carrier concentration in *p*-type InSb in this temperature interval, and suggests that the defects measured by electrical and thermal conductivity are the same.

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ACKNOWLEDGMENTS

The very able assistance of J. H. Smalley in the development and performance of the experiments is gratefully acknowledged. The author has benefited from discussions with Dr. P. G. Klemens and Professor J. S. Koehler. He wishes to thank Dr. M. G. Holland for the use of his data on unirradiated InSb and GaAs prior to publication, and Dr. F. M. Smits and Dr. M. Moss for reviews of the manuscript.

PHYSICAL REVIEW VOLUME 135, NUMBER 6A 14 SEPTEMBER 1964

Cyclotron Resonance in Cadmium Telluride*

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(Received 27 March 1964)

Cyclotron-resonance experiments were carried out on cleaved samples of high-purity n-type CdTe at liquid-helium temperatures and a frequency of 70 Gc/sec. A single well-defined resonance line believed due to electrons was observed under photoexcitation. Rotation of the magnetic field in the (110) plane showed no evidence for anisotropy. An electron effective mass of $m_p=(0.096\pm0.005)m_e$ was obtained. The cyclotron-resonance photosensitivity-versus-wavelength spectrum was found to peak strongly in the vicinity of the direct exciton transition. These results provide data not only for the value of the effective electron mass, but also for additional empirical evidence demonstrating the single-valley behavior of the conduction band of CdTe.

I. INTRODUCTION

Cadmium telluride is generally believed to be a direct band gap material having a conduction band of standard form. As early as 1955, Herman[1] surmised that the band structure of CdTe is similar to that of InSb, which has been shown to be isotropic. Optical experiments have been described which support this point of view. For example, low-temperature luminescence has been observed[2,3] very close (<0.005 eV) to the direct exciton peak which occurs at 7778 Å (1.595 eV). Recently, absorption constant data on high-purity crystals have been unambiguously interpreted in terms of direct transitions.[4] An electron effective mass of the order of $m=0.1\ m_e$, where m_e is the free electron mass, has been used to analyze this optical work.

A similar value of conduction band mass was determined by analyzing certain free carrier and electron transport measurements. For example, Marple[5] was led to a value of $m/m_e=0.11\pm0.01$ from free carrier Faraday rotation experiments and from the contribution of these free carriers to the dielectric susceptibility. His analysis was based on the assumption of a simple band shape. Segall, Lorenz, and Halsted[6] also used a simple band shape and effective mass of the order of 0.1 to interpret Hall mobility data in the temperature range where optical mode scattering predominates. Theoretical estimates[7,8] of the conduction band mass have been based upon the assumption that the band minimum lies at $\mathbf{k}=0$. Again these calculations yield the result that $m/m_e=0.1$, although this is probably not a highly accurate figure. It would be interesting to have an independent and accurate determination of the mass such as provided by cyclotron resonance.

In spite of the evidence in favor of a direct band gap, there have been several attempts to interpret experiments on CdTe in terms of anisotropic bands and indirect transitions. In 1960, Davis and Shilliday[9] observed the spectral dependence of the optical absorption constant and concluded that indirect transitions occur, beginning at photon energies about 0.1 eV less than the direct transition energy. Similar results have appeared in the literature very recently.[10] It should be pointed out that these absorption measurements are not in agreement with the very careful work previously cited (Ref. 4). Magnetoresistance measurements on n-type CdTe [11] have been interpreted in terms of a many-valley conduction band with minima along the ⟨111⟩ directions. Such measurements are quite difficult to carry out at low temperatures and it is possible that contact problems influenced the results, as has been suggested by others.[6]

* Work supported in part by the Advanced Research Projects Agency.

† Work performed in partial fulfillment of the requirements for the Ph.D. degree.

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