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Seebeck Effect in Germanium

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An apparatus for making measurements of the Seebeck effect between 20°K and 375°K is described. Experimental curves of the Seebeck effect are given for a series of germanium crystals in which various concentrations of acceptor and donor atoms have been incorporated. The Seebeck data are compared with Hall data obtained concurrently. Above 250°K the two effects can be correlated by means of existing theory. Below 250°K a marked discrepancy occurs between existing theory and experiment. The temperature dependence of this discrepancy and its dependence upon concentration of added atoms are given. The results are consistent with a new theory proposed by C. Herring which considers an interaction between the phonon and electron systems. A dependence of Seebeck voltage upon sample dimension which is predicted by Herring's theory has been found in the temperature region below 50°K.

A value of $(0.75\pm0.2)m$ has been determined for the mass parameter of both holes and electrons which enters into the "density of states" equation.

I. INTRODUCTION

THIS investigation[1] was undertaken to examine the Seebeck effect and its relationship to the Hall effect in the well-defined single crystals of germanium that have recently become available. It will be apparent that a major modification of existing theory is necessary to explain the results presented here and elsewhere[2] in order to account for the very large Seebeck voltages measured below room temperature. Measurements have been made between 20°K and 375°K on a series of p-type germanium crystals which have varying excess amounts of acceptor atoms and n-type germanium crystals which have varying excess amounts of donor atoms incorporated into the lattice. Near room temperature it is found that the results can be explained by existing theory.

II. THEORETICAL RELATIONSHIPS

The energy flux[3] relative to the electrochemical potential carried through the thermoelectric circuit per carrier of charge e is given by eQT, where Q is the Seebeck voltage per degree difference in junction temperature, or the thermoelectric power, and T the absolute temperature. The Wilson-Sommerfeld theory applied to a nondegenerate extrinsic semiconductor gives[4]

$$eQ_eT=\Delta E_F-\Delta E_T. \qquad (1)$$

The subscript e is used to emphasize that we are considering the contribution of current carriers only. The terms on the right-hand side refer to potential and kinetic energy respectively. ΔE_T is the average kinetic energy transported by the carriers and ΔE_F is their Fermi level, both measured relative to the energy of the edge of the band (conduction or valence) in which the carriers move. ΔE_F is the same parameter which enters into the equation for the density of mobile carriers in an extrinsic, nondegenerate semiconductor.

$$n=2N_v\left(\frac{2\pi m^*kT}{h^3}\right)^{\frac{3}{2}}\exp(-\Delta E_F/kT), \qquad (2)$$

where N_v is a statistical weight factor and m^* is an inertial effective mass. The density of mobile carriers, n, is also inversely proportional to the Hall constant and thus the n determined from Hall measurements can be related to the thermoelectric data by means of Eqs. (1) and (2). The data to be presented below yield a value for the product $N_v^{\frac{2}{3}}m^*$. This product is referred to as the "density of states" effective mass.

III. EXPERIMENTAL PROCEDURE

The crystals were grown by pulling from the melt by the method of Teal and Little.[5] In some cases, the samples were taken from the same crystals used by Debye.[6] We are indebted to J. P. Maita for some help with the sample preparation. Briefly, this is done as follows: A slab is cut perpendicular to the pulled direction of the crystal and lapped to approximately 0.13 cm thick. The [110] direction which is perpendicular to the developed faces is conveniently located. The long axis of the cutting die is lined up in the [110] direction and the sample shown in Fig. 1 cut out using the magnetostriction device developed by W. L. Bond.[7]

Two of the arms shown in Fig. 1 are spares. The two control arms, F, are used as Hall probes and the two outside arms, E, as electrical conductivity probes

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[1] Some results were presented at the Rochester meeting of the American Physical Society, June, 1953 [Phys. Rev. **92**, 857 (1953)].

[2] H. P. R. Frederikse, Phys. Rev. **92**, 248 (1953).

[3] C. Herring and M. H. Nichols, Revs. Modern Phys. **21**, 5506 (1949).

[4] This equation is discussed in detail in a forthcoming paper by C. Herring, Phys. Rev. (to be published). The theoretical investigation carried out by Herring and the experimental investigation reported here were carried out cooperatively and it will be convenient to make frequent reference to that paper.

[5] G. K. Teal and J. B. Little, Phys. Rev. **78**, 647 (1950).

[6] P. P. Debye and E. M. Conwell, Phys. Rev. **93**, 693 (1954).

[7] W. L. Bond, Phys. Rev. **78**, 646 (1950).

in the manner developed by Pearson.[8] All samples used, or samples cut from sections of the crystals near to the samples used are probed at room temperature at 0.05- or 0.1-cm intervals to insure uniform resistivity along the axis. The sample is gold-plated from aqueous solution and then tinned with an indium alloy solder a distance of 0.18 cm on the upper and lower surface of each end. In a few cases, the solder has been secured directly to the germanium surface using acid flux. Gold lead wires are welded to the arms. The sample is washed in distilled water and lightly sand-blasted using 180-mesh silicon carbide powder and soldered in the holder shown schematically in Fig. 1. The hollow heater *A* is sufficiently light to be supported by the sample. The thermal sink *B* is made from copper-filled lead in order to have sufficient thermal stability at the lower temperatures. The copper thermocouple blocks *C* are soldered to the upper surface of the germanium. To facilitate assembly, they are held in place by phosphor-bronze springs that are electrically and thermally insulated from the rest of the system. The thermocouples are constructed from No. 30 constantan and No. 36 copper wires. They are soldered in 0.06-cm holes drilled close to the lower inside corner of each block as indicated. It proved desirable to incorporate the differential thermocouples *G* in order to keep track of the thermal impedance across each junction. The whole system is surrounded by a radiation shield and placed in a container connected to a high vacuum system. The vacuum is usually maintained at 5×10^{-6} mm except at the higher temperatures where out-gassing causes the pressure to rise slightly above 10^{-5} mm.

IV. METHOD OF MEASUREMENT

The Hall and electrical conductivity measurements are made in the usual manner.[6] The Seebeck effect is measured in the steady state with a few hundredths of a watt dissipated at the heater *A*. For a period of about 10 minutes, the power dissipated at the heater, the thermocouple readings, and the Seebeck voltage across the two thermocouple copper leads are followed as a function of time. The system inside the container drifts upward about 0.02° per minute at room temperature with a gradient of 2° between the thermocouples when in a vacuum. The decreasing thermal capacity of the thermal sink and increasing thermal conductivity of the germanium cause the drifts to increase as the temperature is lowered. To maintain the 2° gradient between the junctions, the heat introduced causes a 0.05° drift at nitrogen temperatures and 0.3° drift per minute at 30°K. However, it is possible to stabilize the system between 20 and 30°K, and also in the liquid nitrogen range, by introducing a small pressure of helium into the container and utilizing the heat capacity of the liquid refrigerant surrounding the container. The system quickly reaches a steady state in which there is no drift of temperature with time.

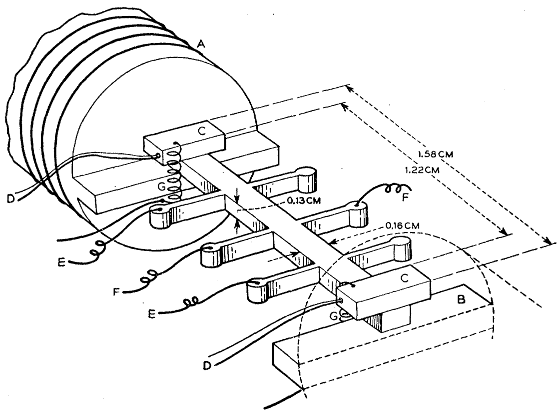

FIG. 1. Apparatus for making Seebeck and Hall measurements.

Although it is difficult to estimate the absolute accuracy of the measurements, we have made numerous checks and taken sufficient precautions to insure a reproducibility of better than 2 percent in the measured values of QT.

The chief source of error is in measuring the temperature exactly at the electrical junctions. The all-copper circuit which is used to measure the thermocouple voltages originally consisted of a type K potentiometer and a high sensitivity galvanometer. Parasitic emf's in the measuring circuit were measured and corrected for. Parasitic emf's introduced by rotation of the potentiometer drum were allowed to decay before taking drifts. Later measurements have been taken with a White double potentiometer and agree with the earlier results. The thermocouples have been calibrated at the boiling and triple points of hydrogen and nitrogen near the beginning and end of the measurements reported here and a temperature scale prepared from the table of Giauque *et al.*[9] The thermocouples are intercompared during each run.

It is possible to perform the following experiments without changing the measured QT value more than 2 percent.

(a) Vary the temperature gradient several fold.

(b) Make a drastic change in the container temperature (for instance from nitrogen temperatures to either hydrogen temperatures or room temperature) without changing the sample temperature. This shows that radiation and heat conduction losses are not affecting the results.

(c) Remove the sample, clean off all the contacts and remount. This indicates the thermocouples are measuring the temperature at (or reproducibly close to) the electrical junctions of the copper and the germanium.

(d) Have either 1 mm of the gas in the container

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[8] G. L. Pearson and J. Bardeen, Phys. Rev. **75**, 865 (1949).

[9] Giauque, Buffington, and Schulze, J. Am. Chem. Soc. **49**, 2343 (1927).

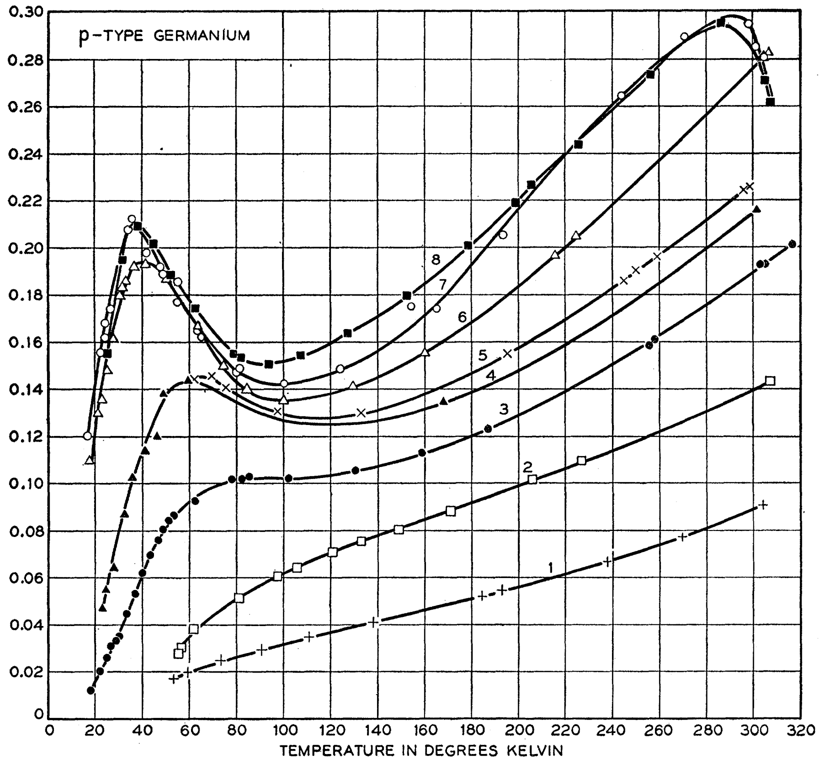

FIG. 2. Measured values of the QT product for eight p-type samples listed in Table I.

(no drift conditions, see above) or less than 10^{-5} mm residual pressure in the container. This shows the thermocouple blocks are in excellent thermal contact with the sample.

One can compare the ratio of the Seebeck voltages measured between the thermocouple blocks and between the arms E (Fig. 1) with the ratio of the distances between the blocks and between the insides of the arms E. At room temperature, the ratios are the same. In some samples, the Seebeck voltage ratio remains the same at low temperatures whereas in other samples it increases by as much as 10 percent. In a few cases, the ratio changed several hundred percent and in such cases the results have been discarded.

V. EXPERIMENTAL RESULTS AND ANALYSIS

The measured values of Q show a remarkable rise as the temperature is lowered below room temperature for a wide range of donor and acceptor concentrations. The QT product is plotted as a function of temperature between 20°K and 300°K for the series of p-type crystals in Fig. 2 and for the n-type crystals in Fig. 3. The room temperature resistivities of the samples are given in Table I. Also in Table I the excess donor or acceptor atom concentrations are given for each sample. These values are assumed equal to the number of mobile carriers determined by Hall measurements in the saturation temperature range. This assumption has been verified for the antimony-germanium system by Pearson, Struthers, and Theuerer using radioactive techniques.[10] By saturation temperature range is meant the temperature range over which the donor or acceptor atoms are almost completely ionized giving a constant number of mobile carriers which is much larger than the number of mobile carriers resulting from intrinsic ionization. In the purer samples the saturation range extends from below 50°K to room temperature. In the upper temperature range the measured QT values are almost proportional to T. The Wilson-Sommerfeld theory predicts this type of behavior throughout the saturation temperature range, i.e., from about 40°K to almost 300°K for the purer samples. It appears that this theory can be reasonably complete only above 250°K. For a given temperature the absolute value of QT decreases as the number of acceptors or donors increases as is also predicted by the Wilson-Sommerfeld treatment. The rapid increase in Q for the purer materials as the temperature decreases from 200°K to 35°K cannot be accounted for by this theory. It is postulated by Frederikse[2] and Herring[4] that there is an interaction between the anisotopic lattice vibrations, which exist of necessity in the thermal gradient, and the mobile charge carriers. The carriers are preferentially scattered toward the cold end of the sample by the nonequilibrium phonon distribution. The electrostatic field established within the semiconductor by the normal diffusion of the mobile carriers from hot to cold is thus enhanced by an interaction between

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[10] Pearson, Struthers, Theuerer, Phys. Rev. **77**, 809 (1950).

TABLE I. Room temperature resistivities and composition of samples.

Sample No.[a]	Resistivity at 300°K (ohm cm)	Excess No. of donor atoms per cc (from Hall measurements)	Sample No.[a]	Resistivity at 300°K (ohm cm)	Excess No. of atoms per cc (from Hall measurements)
1	0.0033	7.1×10^{18} gallium	*A*	0.0050	2.3×10^{18} arsenic
2	0.016	6.0×10^{17} gallium	*B*	0.032	1.08×10^{17} arsenic
3	0.066	8.0×10^{16} gallium	*C*	0.38	6.4×10^{15} arsenic
4	0.18	2.1×10^{16} gallium	*D*	2.5	7.8×10^{14} arsenic
5	0.23	1.5×10^{16} gallium	*E*	18.5	8.9×10^{13} arsenic
6	1.7	1.5×10^{15} gallium	*F*	46.0	8×10^{12}[b]
7	21.5	1.6×10^{14} gallium			
8	20.0	1.9×10^{14} indium			
9	34.5	8.0×10^{13} gallium			

[a] All samples are cut in the 110 direction from crystals pulled from the melt as described in the text except sample 8. Sample 8 is cut in some direction perpendicular to the 111 direction from a zone-leveled [see W. G. Pfann and K. M. Olsen, Phys. Rev. **89**, 322 (1953)] single crystal.
[b] Identity of excess donor not known.

the mobile carriers and the lattice. Indeed, the experimental values to be presented show that at the lower temperatures the latter effect must dominate almost completely.

The discrepancy between existing theory and experiment, which presumably is the result of the phonon-electron interaction, and will be referred to as such in the subsequent discussion, can be estimated by setting the measured QT product equal to two terms,

$$(QT)_{\text{meas}}=Q_eT+Q_pT, \tag{4}$$

where Q_eT is the contribution of the mobile carriers arising from the Wilson-Sommerfeld treatment, and Q_pT is the contribution arising from the phonon-electron effect. Combining Eqs. (1) and (2) and transforming to convenient units gives

$$\pm Q_eT=\left[\log 4.70\times10^{15}T^{\frac{3}{2}}N_v\left(\frac{m^*}{m}\right)^{\frac{3}{2}}-\log n\right]T/5037+AkT, \tag{5}$$

where A is a constant specifying the kinetic energy transported and which can be calculated, and n is the number of mobile carriers per cc. The theoretical limits of A in the final term of Eq. (5) are discussed by Herring.[4] Following him, A is taken as 2.0 for high purity n-type material and as 2.0 for high purity p-type material below 125°K. Between 125°K and 300°K, A for p-type material is assumed to decrease linearly from 2.0 to 1.7. It should be noted that the whole AkT term is only 15 percent of $Q_e\,T$ for the high purity samples. The number of mobile carriers per cc, n, is calculated from the Hall constant R_H using the equation[11]

$$n=\frac{\mu_H}{\mu_c}\frac{1}{R_He}. \tag{6}$$

The Hall to conductivity mobility ratio μ_H/μ_c, has been determined by Morin[12] and differs somewhat from $3\pi/8$ which is correct only when the mean free path is independent of energy. The Q_eT values are insensitive to small uncertainties in n since a 10 percent variation in n changes Q by only 10 microvolts.

In Figs. 4 and 5 are plotted isotherms of Q_pT determined from Eqs. (4) and (5) as functions of acceptor and donor atom concentrations, respectively. The unspecified effective mass parameter $N_v^{\frac{2}{3}}m^*$ occurring in Eqs. (2) and (5) is chosen as 0.75 of the free electron mass m for both n and p material on the basis of measurements in and near the intrinsic range which are discussed below.

It is apparent from Figs. 2 through 5 that the behavior of p- and n-type material is qualitatively similar. The dependence of Q_pT on donor or acceptor concentration is small until a concentration range is

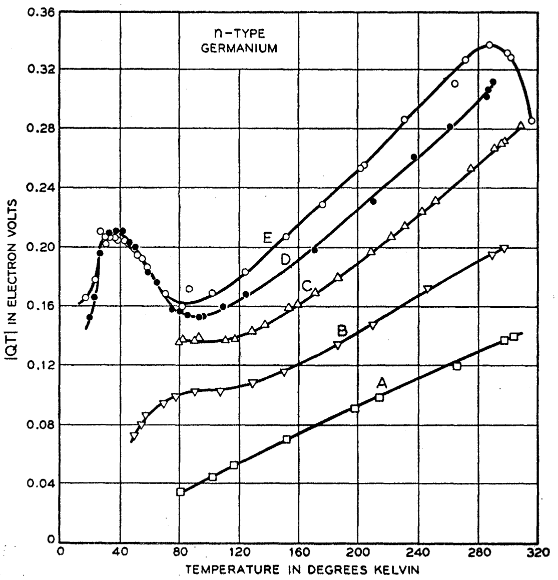

FIG. 3. Measured values of the QT product for five n-type samples listed in Table I.

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[11] W. Shockley, *Electron and Holes in Semiconductors* (D. Van Nostrand Company, Inc., New York, 1950), p. 215.

[12] F. J. Morin, Phys. Rev. **93**, 62 (1954).

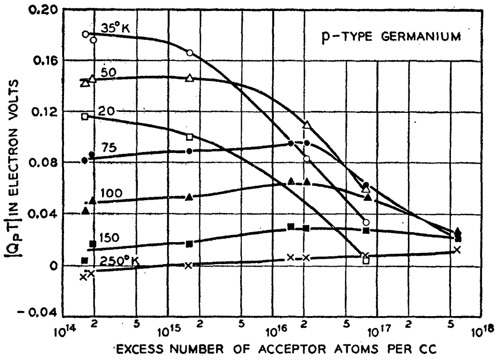

FIG. 4. Isotherms of Q_pT, Eq. (4), as function of concentration of excess acceptor atoms.

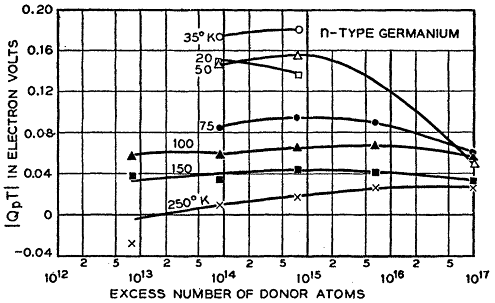

FIG. 5. Isotherms of Q_pT, Eq. (4), as function of concentration of excess donor atoms.

reached beyond which Q_pT falls rapidly. This concentration range becomes lower as the temperature decreases. Q_p becomes small when the concentration becomes large enough to approach Fermi-type degeneracy. The slightly positive slope between 10^{14} and 10^{16} which persists down to 75°K can be partly accounted for by a reasonable variation of the transport term AkT of Eq. (5) with carrier concentration.[4]

The temperature dependence of Q_pT is shown in Fig. 6 for both n- and p-type material. The plotted chords bracket variations in Q_p for samples of concentration less than 10^{+15} per cc down to 50°K and for concentrations less than 10^{+14} per cc below 50°K. The inverse temperature dependence is greater for the p-type material. Between 75°K and 150°K Q_pT for the p-type sample is proportional to about $T^{-2.2}$, whereas Q_pT for the n-type sample is proportional to about $T^{-1.4}$. The slopes of the linear portion of the curves of Fig. 6 correspond to the above temperature dependence. The effect becomes too small to measure at temperatures much above 150°K.

Herring's theory suggests that boundary scattering of phonons should be an important factor in describing the low temperature behavior in much the same way as it is important in low temperature thermal conductivity.[13] Reference to Fig. 6 shows that departure from the inverse temperature dependence given by the straight lines occurs at between 50°K and 70°K. This is a considerably higher temperature range than that at which phonon boundary scattering becomes important in thermal conductivity experiments. This is what one would expect if the low energy phonons predominantly are interacting with the mobile carriers. In order to determine whether boundary scattering can be used to explain the low temperature curvature in Fig. 6, and, in fact, to provide experimental corroboration of Herring's model,[4] the thicknesses of p-type sample 7 and n-type sample E were reduced by sand blasting and the samples remeasured. Sample 7 was reduced from 0.125 cm to about 0.069 cm for a distance of about 1.0 cm. Sample E was reduced from 0.148 cm to about 0.025 cm for a distance of about 1.0 cm.[14] In order to avoid breakage the thin portion of the sample was supported by a sheet of 0.005 in. mica using an epoxy-resin cement. The curves for the "thin" samples are within 2 percent of the originals shown in Figs. 2 and 3 down to 80°K. The results below 80°K are shown in Fig. 7 on a somewhat enlarged scale for both

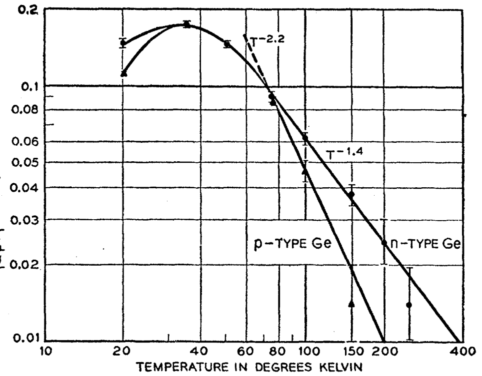

FIG. 6. Dependence of Q_pT, Figs. 4 and 5 on temperature for the purer samples.

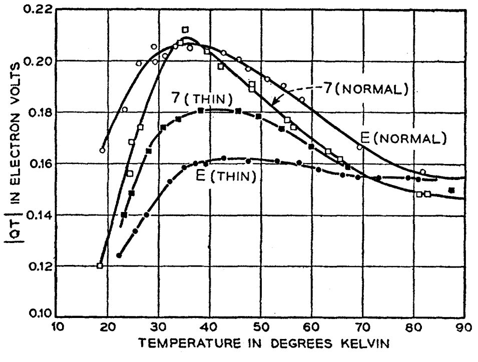

FIG. 7. Dependence of measured values of QT upon sample dimension.

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[13] R. Berman, Advances in Physics 2, 103 (1953).

[14] All samples except E were between 0.12 cm to 0.13 cm thick. However, breakage was encountered while measuring high purity n-type samples (for example, sample F, which did not break until after measurements shown in Fig. 8 were completed). Sample E was purposely made thicker in order to reduce the chance of breakage.

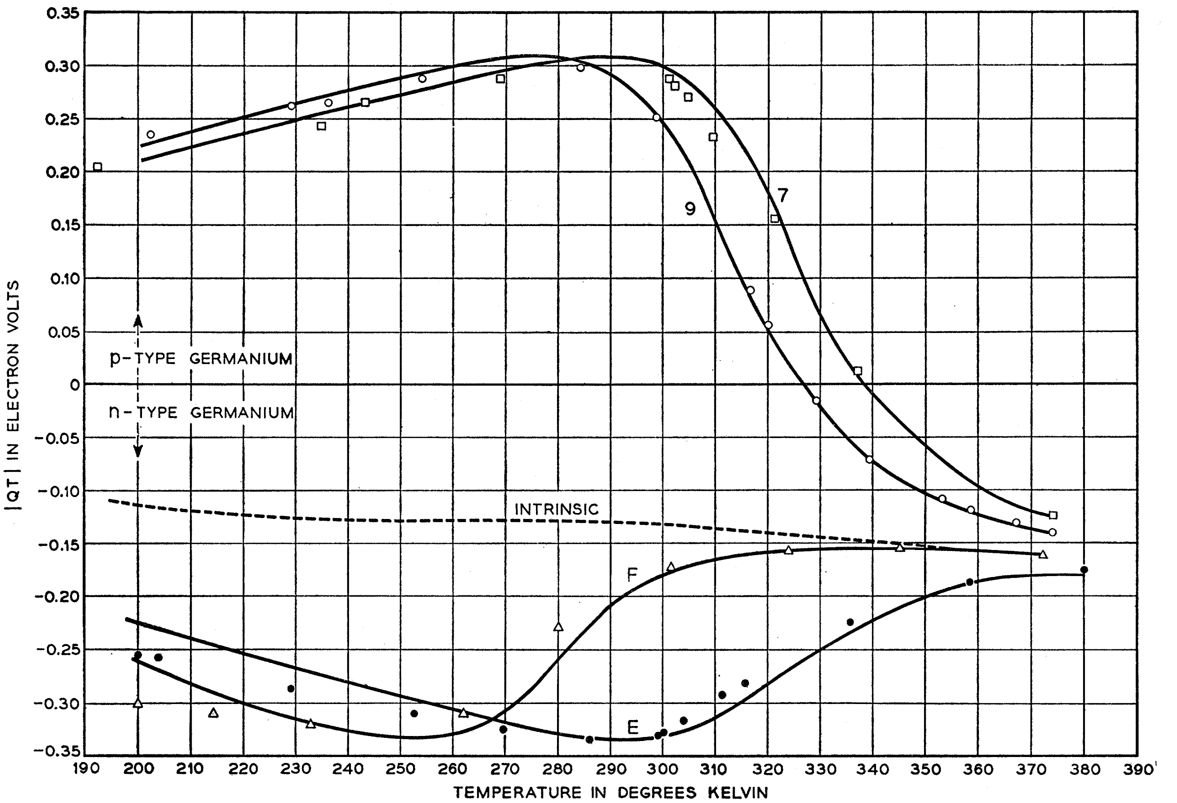

FIG. 8. Calculated values (curves) and measured values in the temperature range where the sample changes from extrinsic to intrinsic.

the normal (original) size and the "thin" size. It appears that the measurements below about 75°K are not typical of bulk germanium alone. The values are smaller for the smaller dimensions as is expected on the basis of Herring's model. The curvature in the low temperature portion of the curves of Fig. 6 is a property of the samples used, presumably much larger samples would follow the inverse temperature relations to lower temperatures. Similarly the maxima in Figs. 2 and 3 would shift to lower temperatures and higher values. The availability of large crystals of sufficient uniformity makes possible a more complete investigation of the effect of the sample dimensions. We hope to be able to report on this in the near future.

We now consider data near room temperature, where Q_p is small, in order to determine a value of $N_v^{2/3}m^*$ occurring in Eq. (5). It can be seen from Figs. 3 and 4 that the choice of $N_v^{2/3}(m^*/m)=0.75$ for both n- and p-type samples satisfies Eq. (1) to about ±0.01 ev for the purer samples at 250°K after making a small allowance for Q_p. It has already been noted that the positive slope of these curves, indicating a slow, possibly logarithmic variation with impurity concentration, can be partly explained by a variation of the transport term AkT of Eq. (5). Additional confidence in this value of $N_v^{2/3}(m^*/m)$ can be obtained by considering the results above room temperature where the purer samples become intrinsic. The purer samples have been measured to 375°K, and the results shown in Fig. 8. The curves are calculated using only the 1 adjustable parameter $N_v^{2/3}m^*=0.75m$ for both the n and p samples. To make this calculation, equation (5) must be generalized to include both positive and negative mobile carriers:

$$Q_eT=\frac{T(Q_+n_+\mu_++Q_-n_-\mu_-)}{n_+\mu_++n_-\mu_-}. \qquad (7)$$

The concentration product of holes, n_+, and electrons, n_-, at the temperature T, and the respective mobilities μ_+ and μ_- are taken from the data of Morin and Maita.[15] The quantities Q_+ and Q_- are the thermoelectric powers the mobile holes would have in the absence of electrons, and the mobile electrons would have in the absence of holes, respectively, calculated from Eq. (5). The higher temperature points where the samples are almost completely intrinsic fall on the curves within experimental error. A variation of $N_v^{2/3}m^*/m$ by ±0.15 for either holes or electrons separately would be sufficient to cause a systematic deviation of the points from the curves in the almost intrinsic region. The agreement is not so good in the temperature region where the sample is changing from extrinsic

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[15] F. J. Morin and J. P. Maita, Phys. Rev. (to be published).

to intrinsic, i.e., where the concentration of the minority carriers is changing rapidly with respect to the majority carriers. In order to expect agreement in this region, one must assume an unusually high degree of uniformity and perfection of the crystal. The extrinsic region, or lower temperature region of Fig. 8, has already been discussed. The value of $N_v^{\frac{2}{3}}m^*/m=0.75$ for both holes and electrons gives a reasonable fit at 300°K. An assumption of $N_v^{\frac{2}{3}}m^*/m=1.0$ would decrease the absolute value of the 300° points by 0.011 volt whereas an assumption of $N_v^{\frac{2}{3}}m^*/m=0.55$ would increase them by 0.015 volt.

The "density of states" effective mass parameter $N_v^{\frac{2}{3}}m^*/m$ is related to the inertial effective mass parameter, m^*/m, by the statistical factor which we have called $N_v^{\frac{2}{3}}$, N_v would be the number of equivalent valleys or edges in a band with no spin-orbit splitting. The theoretical problem is discussed in some detail by Herring.[4] It suffices here to point out that while our result is not directly comparable with a number of recent experiments[16] that determine inertial effective mass, a comparison of the values may give further information about the band structure.

We are indebted to our colleagues F. J. Morin, J. A. Burton and C. Herring for many helpful discussions.

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[16] Dresselhaus, Kip, and Kittel, Phys. Rev. **92**, 827 (1953); T. S. Benedict and W. Shockley, Phys. Rev. **89**, 1152 (1953); P. P. Debye and E. M. Conwell, Phys. Rev. **93**, 693 (1954).

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Structure of Gd, Dy, and Er at Low Temperatures*

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The crystal structures of gadolinium, dysprosium, and erbium metals have been determined over a wide temperature range. The metals are hexagonal close packed and the a_0 unit axis contracts nearly linearly with decreasing temperature, whereas the c_0 unit axis exhibits an abnormal expansion with decreasing temperature below magnetic transformations of the metals. The c_0 behavior is believed to be associated with the magnetic properties of the metals.

I. INTRODUCTION

MAGNETIC transformations in metals are usually accompanied by crystal structure changes which may be of different types.[1] In view of the fact that some of the rare earth metals have been reported to be ferromagnetic it was felt that an x-ray study of the structures of these over a wide temperature range should be made. This report covers such studies for gadolinium, dysprosium, and erbium.

Gadolinium has been reported[2–5] to have a ferromagnetic Curie point of 16°C. Dysprosium exhibits[6–9] a magnetic anomaly at about 175°K and a ferromagnetic Curie point near 90°K. Erbium has recently been found[10] to have a magnetic susceptibility anomaly of the dysprosium type near 80°K, and it is further reported that the susceptibility becomes strongly field-dependent below 55°K.

Dilatometric studies on gadolinium and dysprosium have been reported by Trombe and Foex.[11,12] For gadolinium they report an abrupt decrease in the coefficient of thermal expansion below the Curie point. A similar phenomenon was displayed by dysprosium below the magnetic anomaly near 175°K.

The electrical resistivities[4] of the three metals also exhibit peculiarities near the temperatures cited above, showing the influence of the transformations on the conduction process. This is also in evidence in the study of the Hall effect as reported by Kevane *et al.*[13]

II. EXPERIMENTAL METHODS

The metal samples used were prepared by methods reported earlier.[14,15] Metal filings annealed under vacuum at 550°C for about 30 hours were mixed with pulverized

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* Contribution No. 309 from the Institute for Atomic Research and Department of Physics, Iowa State College, Ames, Iowa. Work was performed in the Ames Laboratory of the U. S. Atomic Energy Commission.

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[3] F. Trombe, Ann. phys. **7**, 385 (1937).

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[5] Elliott, Legvold, and Spedding, Phys. Rev. **91**, 28 (1953).

[6] F. Trombe, J. phys. et radium **12**, 222 (1951).

[7] F. Trombe, Compt. rend. **221**, 19 (1945).

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[9] Elliott, Legvold, and Spedding, this issue, Phys. Rev. **94**, 1143 (1954).

[10] Elliott, Legvold, and Spedding, "Some of the Magnetic Properties of Erbium Metal" (to be published).

[11] F. Trombe and M. Foex, Compt. rend. **235**, 42 (1952).

[12] F. Trombe and M. Foex, Compt. rend. **235**, 163 (1952).

[13] Kevane, Legvold, and Spedding, Phys. Rev. **91**, 1372 (1953).

[14] Spedding, Voight, Gladrow, and Sleight, J. Am. Chem. Soc. **69**, 2777, 2786, 2812 (1947).

[15] F. H. Spedding and A. H. Daane, J. Am. Chem. Soc. **74**, 2783 (1952).