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Electrical Properties of Tellurium at the Melting Point and in the Liquid State

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The electrical resistivity, the Hall coefficient, and the thermoelectric power of spectroscopically pure tellurium decrease abruptly on melting. The Hall coefficient and the thermoelectric power remain positive above the melting point up to approximately 575°C where they reverse their sign and above which they remain negative up to the highest temperatures measured. The resistivity continues to decrease with rising temperature in the liquid until it approaches a constant value at about 650°C. The experimental results indicate that semiconducting properties persist in the liquid state and that a gradual transition to metallic conduction takes place at higher temperatures.

INTRODUCTION

Most metals crystallize in either face-centered cubic or body-centered cubic lattices with coordination numbers of 12 or 8 except in the case of some of the so-called "semimetals" like bismuth. Upon melting, the structure is essentially disordered and in most cases the electrical conductivity decreases as predicted by the theory of Mott.[1] However, in the case of bismuth the conductivity in the liquid is larger than in the solid, contrary to the simple theoretical expectations but in agreement with the anomalous behavior of the solid. It looks as if the number of carriers in the liquid is an order of magnitude larger than in the solid. Recently a similar behavior has been observed in antimony[2] where the conductivity increases on melting and the Hall coefficient decreases.

The following investigation was prompted by the fact that it is known from the x-ray investigations of Hendus[3] that liquid germanium has a coordination number of 8 as compared to the coordination number of 4 in the solid and that the density of the liquid is greater than the density of the solid. Therefore, one might expect that liquid germanium should behave like a metal and not like a semiconductor, with the resistivity increasing with increasing temperature.[4]

On the other hand, there are semiconductors such as selenium and tellurium which have structural elements—spiral chains—which might be preserved even in the amorphous and the liquid state. In amorphous selenium it has been found[5] by x-rays that the number of nearest neighbors is 2, just as in the crystalline solid, and that the distance between nearest neighbors is preserved. Electrical investigations of liquid selenium[6] indicate that it remains a semiconductor in the liquid state with predominant hole conduction.

It was of particular interest to investigate the electrical properties of liquid tellurium since it was possible to produce extremely pure tellurium ingots and to observe the solid-liquid transition in this material. The Hall coefficient, the thermoelectric power, and the resistivity of tellurium over a wide range of temperatures were investigated.

A brief summary of the structural and electrical properties of tellurium will be helpful for discussing the experimental results.

TELLURIUM

Tellurium crystallizes in a hexagonal structure[7,8] as shown in Fig. 1 with the atoms arranged in spiral chains, each sharing covalent bonds with its two nearest neighbors in the chain. The chains are held together by forces of Van der Waals type. Every third atom is directly above another atom in its chain, so that the projection of the chain on a plane perpendicular to the c axis is an equilateral triangle. The hexagonal lattice is achieved by locating a chain at the center and at each of the six corners of a hexagon. The relatively weak bonds between chains generate planes of easy cleavage parallel to the c axis. The

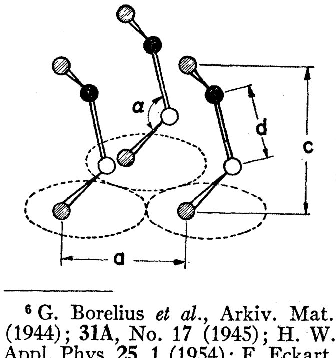

Fig. 1. Chain structure of tellurium. Similarly shaded atoms are in the same plane in neighboring chains and above each other in the same chain. $a=4.44$ A; $c=5.92$ A; $d=2.86$ A; $\alpha=102.6°$

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‡ Supported by Signal Corporation Contract.

[1] N. F. Mott, Proc. Roy. Soc. (London) **A146**, 465 (1934).

[2] G. Busch and O. Vogt, Helv. Phys. Acta **27**, 241 (1954).

[3] H. Hendus, Z. Naturforsch. **2a**, 505 (1947). For a discussion of the change in structure, density, and electrical properties of elements on melting see also references 9 and 16.

[4] This was actually found by A. S. Epstein and independently by R. W. Keyes, Phys. Rev. **84**, 367 (1951).

[5] K. Lark-Horovitz and E. P. Miller, Phys. Rev. **51**, 380 (1937).

[6] G. Borelius *et al.*, Arkiv. Mat. Astron. Fysik **30A**, No. 14 (1944); **31A**, No. 17 (1945); H. W. Henkels and J. Maczuk, J. Appl. Phys. **25**, 1 (1954); F. Eckart, Ann. Physik **14**, 233 (1954).

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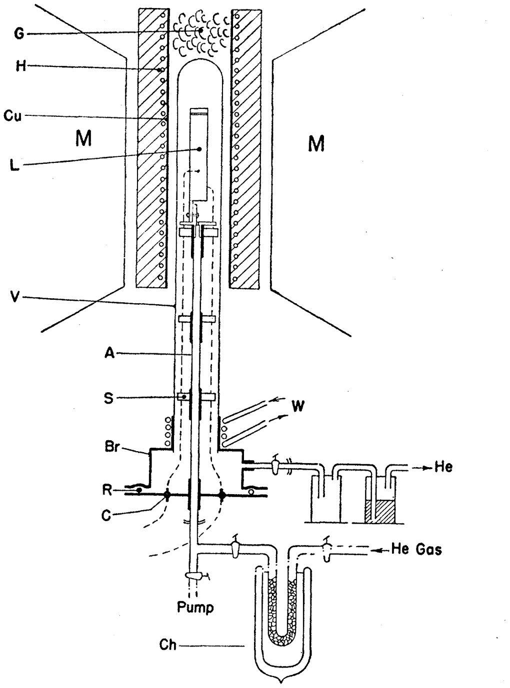

FIG. 2. Arrangement for measuring Hall coefficient, resistivity, and thermoelectric power of liquid tellurium. L = Lavite container, M = magnet poles, H = heater, Cu = copper shield, G = glass wool, V = Vycor tube, A = Alundum tube, S = Lavite spacer, Br = brass base, W = cooling-water connections, R = O-ring seal, C = Kovar to glass seal, Ch = liquid-nitrogen cooled charcoal trap.

melting point is 445°C[9] and the entropy of fusion is given as 5.80 cal/g-atom deg.[10]

Several investigators[11] have studied the electrical properties of single-crystal tellurium of various degrees of purity. The results show that tellurium is always a *p*-type semiconductor at low temperatures. Other elements added to the tellurium in small concentrations may introduce acceptors; none has been found so far which acts as a donor. As tellurium becomes intrinsic at higher temperatures, the sign of the Hall coefficient reverses and becomes negative, indicating a mobility of the electrons larger than that of holes. The forbidden energy gap is about 0.35 ev.

The sign of the Hall coefficient reverses back to positive at about 230°C in the intrinsic range. This second reversal temperature is independent of the impurity concentration. This anomalous second sign reversal of the Hall coefficient of tellurium has been interpreted[12] as evidence of the existence of a second narrow band inside the conduction band.[13] At higher temperatures an appreciable concentration of electrons will then be excited into this low-mobility conduction band, thus decreasing the average mobility of the electrons below that of the holes so that the sign of the Hall coefficient reverses to positive. According to another interpretation,[14] the valence band contains another high-mobility band into which holes are excited at high temperatures, thus increasing the average mobility of the holes above that of the electrons and causing the Hall coefficient to become positive.

The measurements of optical absorption of tellurium seem to be in better agreement with this interpretation[15] than with one previously given[16] which relates the positive Hall coefficient above 230°C to an increase in hole concentration due to an increase of the number of lattice defects with rising temperature.

The electrical properties of tellurium are strongly anisotropic. The resistivities of intrinsic tellurium at 20°C are $\rho_{\parallel} = 0.26$ ohm cm and $\rho_{\perp} = 0.51$ ohm cm, parallel and perpendicular to the *c* axis, respectively. The ratio of the resistivities in these two directions is approximately independent of temperature at high

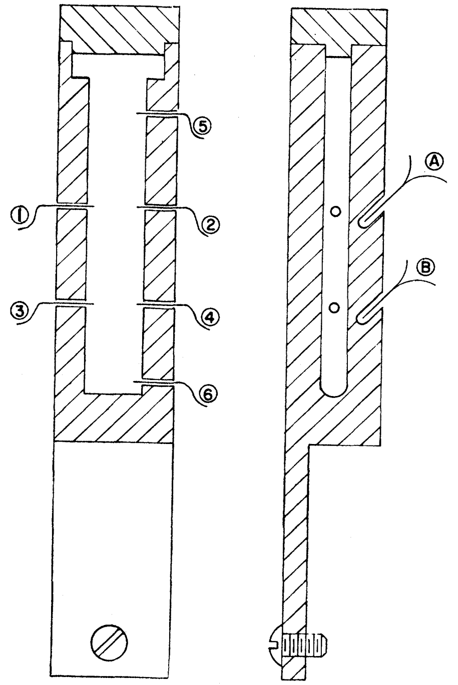

FIG. 3. Lavite container L for Hall and resistivity measurements. 1–4 voltage probes; 5, 6 current electrodes; A, B Chromel-Alumel thermocouples.

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[13] Regarding this interpretation, see the comments of G. Dresselhaus, Phys. Rev. **105**, 138 (1957).

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[16] W. Schottky (private communication to K. Lark-Horovitz, 1950); H. Fritzsche, Science **115**, 571 (1952); S. Tanuma, Sci. Repts. Research Inst. Tohoku Univ. **6**, 159 (1954).

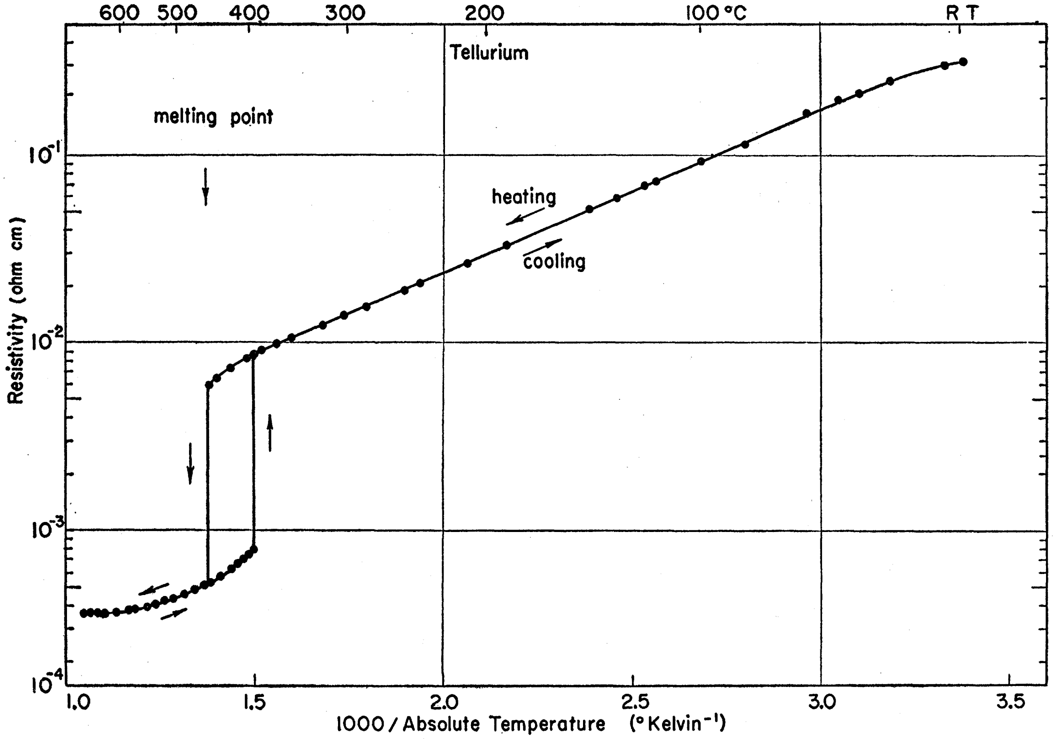

FIG. 4. Temperature dependence of the resistivity of liquid and solid tellurium measured in a Lavite container with long, narrow bore.

temperature (intrinsic range). The anisotropic properties of the Hall coefficient have not been investigated systematically. Measurements of the thermoelectric power with temperature gradients parallel and perpendicular to the *c* axis will be presented below.

EXPERIMENTAL PROCEDURE

The electrical measurements were carried out using the apparatus shown in Figs. 2 and 3. Container L, which held the tellurium, was made of Lavite and baked at a temperature of 900°C. Tungsten wires or graphite probes were put through the openings at 1 to 6 in good contact with the tellurium. Tungsten, Lavite, and spectroscopically pure graphite do not react noticeably with tellurium.

All measurements were performed with doubly distilled spectroscopically pure tellurium in a stream of helium at atmospheric pressure.

The potential differences were measured with a conventional dc compensation method. The current-voltage characteristic was found to be ohmic up to 9 amperes/cm^2. The Hall voltage was proportional to the dc magnetic field up to 4200 oersteds, and the thermoelectric voltage was proportional to the temperature gradient up to 6 degree/cm. These were the largest values used for the respective quantities.

EXPERIMENTAL RESULTS

(a) Resistivity

A typical graph of the temperature behavior of the resistivity of tellurium is shown in Fig. 4. In the solid the resistivity decreases exponentially with increasing temperature according to an intrinsic energy gap of approximately 0.34 ev. In a limited interval before melting, the resistivity decreases faster with increasing temperature. At the melting point the resistivity of this particular ingot drops discontinuously by a factor of 11.7. If one goes to higher temperatures in the liquid, the resistivity continues to decrease until it slowly approaches the constant value of 3.6×10^{-4} ohm cm at about 625°C, and remains constant up to 670°C, above which temperature it slowly rises.

Tellurium can be supercooled sometimes to a temperature of 400°C. The resistivity-temperature curve in the supercooled region is a smooth continuation of that in the normal liquid region.

After solidification the resistivity will depend on the average orientation of the polycrystalline ingot. In the case of Fig. 4, reproducibility of the average orientation of the crystallites and hence of the resistivity was obtained by using a long Lavite container 2 mm in diameter.

After the measurements were completed, the Lavite containers were dissolved in hydrofluoric acid in order

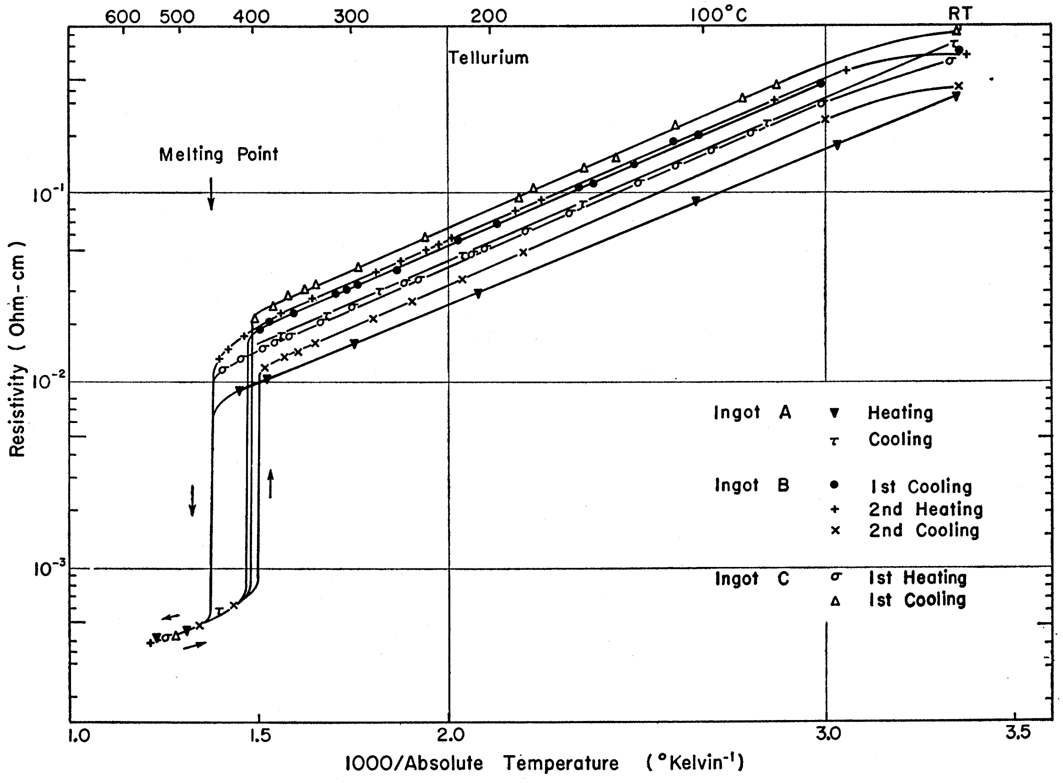

FIG. 5. Same as Fig. 4 using Lavite containers with large cross sections.

to investigate the orientation of the crystallites by x-rays.[17] In long, narrow containers the *c* axes of the crystallites were inclined by angles of 0° to 15° away from the long axis of the bore which was the direction of current flow.

The result of measurements carried out with containers of larger cross sections is shown in Fig. 5. The resistivity in the liquid is reproducible for all ingots of various cross sections. A great number of measured points have been omitted on this graph for clarity. In the solid the resistivity curves are shifted parallel to each other because the over-all resistivity depends on the average orientation of the crystallites which is not reproducible for ingots with larger cross sections. Values between 12 and 20 were obtained for the ratios of the resistivity of the solid to that of the liquid tellurium at the melting point.

One ingot with the largest resistivity and another with the smallest resistivity were investigated by x-rays.[17] From this a factor of 11±0.5 and a factor of 21±0.5 were obtained as the best estimates for the resistivity ratios at the melting point for orientations parallel and perpendicular to the *c* axis, respectively.

(b) Hall Effect

The temperature dependence of the Hall coefficient of solid and liquid tellurium is shown in Fig. 6 for two measurements. In the solid the sign of the Hall coefficient reverses to positive with increasing temperature at about 230°C as discussed above, and retains its positive sign to the melting point. The magnitude of the Hall coefficient at the melting point ranges from $+0.2$ cm^3/coulomb to $+0.4$ cm^3/coulomb depending on the orientation of the crystallites. Because of the small dimension of the ingot in the direction of the magnetic field the *c* axes were in all cases perpendicular to the direction of the magnetic field. The large Hall coefficient corresponds to the *c* axis oriented more nearly perpendicular to the current flow, the smaller Hall coefficient corresponds to the *c* axis oriented more nearly parallel with the current flow.

At the melting point the Hall coefficient shows a discontinuous decrease by a factor varying between 10 and 20 to a value in the liquid of $+0.02$ cm^3/coulomb. The Hall coefficient of the liquid continues to decrease with increasing temperature until its sign reverses again to negative at a temperature of about 575°C.

The Hall curve is found to be retraceable in the liquid and it continues smoothly into the supercooled region. After solidification one again finds different values due to the anisotropy of the Hall coefficient.

To check the validity of the results in the liquid, the dependence of the Hall voltage on the current, the magnetic field, and the thickness of the sample was tested. The Hall voltage was found to vary linearly with current and magnetic field and was inversely

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[17] We are grateful to I. Geib and W. Binnie (Purdue University) for the x-ray investigations.

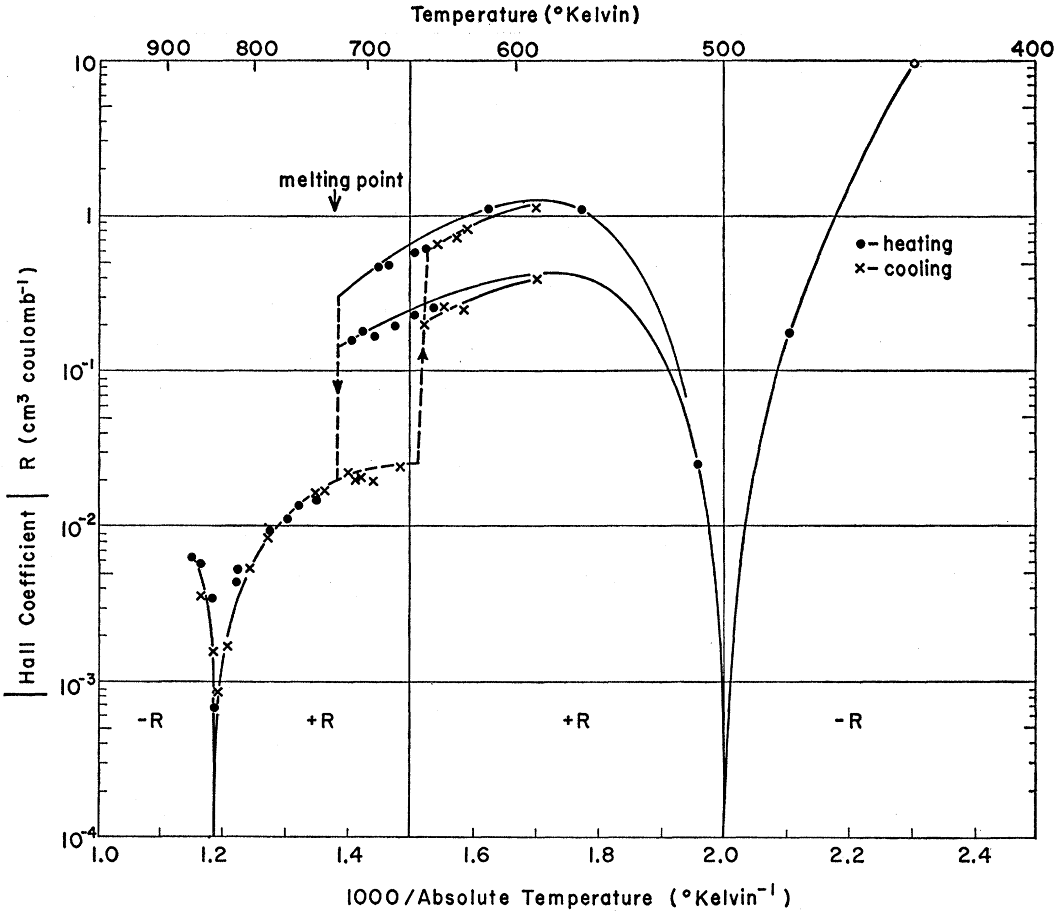

FIG. 6. Two measurements of the Hall coefficient of liquid and solid tellurium as a function of $1/T$.

proportional to the thickness. The thickness was changed by a factor of 2.

(c) Thermoelectric Power

Figure 7 shows some typical curves of the thermoelectric power of tellurium against tungsten. Above 403°K the thermoelectric power increases with increasing temperature and assumes positive values above 453°K. A spread in the magnitude of the thermoelectric power for different samples from about +120 microvolts/degree to about +300 microvolts/degree is observed at higher temperatures. On melting there is a discontinuous decrease to +9 microvolts/degree in the liquid. The value in the solid at the melting point varies from +100 microvolts/degree to +260 microvolts/degree.

The proximity of the temperatures at which the signs of the Hall coefficient and of the thermoelectric power of the liquid reverse might be a coincidence since the correction which has to be applied to obtain the absolute thermoelectric power of liquid tellurium may not be negligible. A vertical shift of the thermoelectric power curve will in turn change its reversal temperature considerably.

The large spread in magnitude of the thermoelectric power in the solid can be attributed to the anisotropy of tellurium. Figure 8 shows the result of thermoelectric power measurements on a single crystal of tellurium. For a temperature gradient parallel to the c axis the thermoelectric power reaches a value of +320 microvolts/degree, for a temperature gradient perpendicular to the c axis a value of +135 microvolts/degree at 623°K.

DISCUSSION

In discussing the electrical properties of liquid metals and alloys one usually tries to apply the theories of electric conduction of the crystalline state to the liquid state by treating the liquid as a highly disordered solid.[1] This approach yields quite satisfactory results in the case of true liquid metals. In these the concentration of free carriers is constant and the temperature dependence of the resistivity can be explained by the increase of scattering with rising temperature. Germanium also seems to show metallic conduction

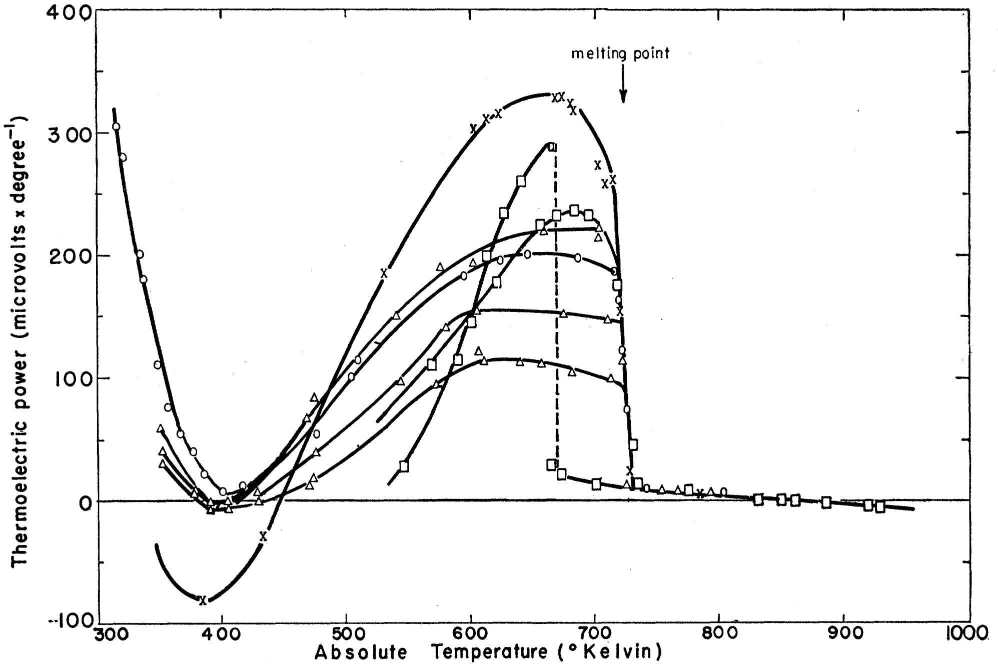

FIG. 7. Thermoelectric power of several tellurium ingots measured against tungsten.

with a temperature-independent carrier concentration in the liquid state.

In contrast to these are the liquid semimetals Bi and Sb, with rather strongly covalent bonds. The temperature dependence of the resistivity of these liquids can be understood as a combination of two competing processes: one is the normal resistivity increase with temperature, due to increasing scattering, and the other is the transition in the liquid from covalent bonding into metallic bonding, a decomposition of aggregates of the original crystalline type of binding which still exist in the liquid state. This will consequently result in an increase of free-carrier concentration and a decrease of the resistivity with rising temperature if one makes the plausible assumption that the electrons used for covalent or heteropolar bonding do not contribute noticeably to the electrical conduction.

The last assumption, however, seems not to hold in the case of liquid tellurium. The strongest evidence for a different behavior is the reversal of the sign of the Hall coefficient in the liquid. The Hall coefficient can become zero only if there are two kinds of carriers of opposite sign contributing to the conduction at the same time. A noticeable contribution of tellurium ions to the conduction can be excluded because their mobility is smaller than that of the electrons by several orders of magnitude. Hence one is led to the conclusion that electrons and holes participate in the conduction of liquid tellurium. Hole conduction in amorphous and liquid selenium also indicates that the existence of holes is not restricted to periodic crystalline structures.

The results of x-ray investigations[18] on tellurium slightly above the melting point have shown that not only the coordination number but also the nearest-neighbor distance of the crystalline state is preserved in the liquid. This indicates the presence of covalent bonds which orient and bind a certain fraction of the

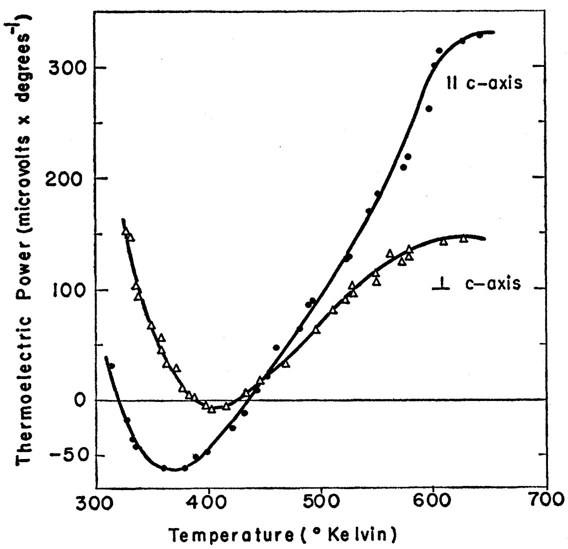

FIG. 8. Thermoelectric power of a tellurium single crystal as measured with temperature gradient respectively parallel and perpendicular to the *c* axis.

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[18] Buschert, Geib, and Lark-Horovitz, Phys. Rev. **98**, 1157 (1955).

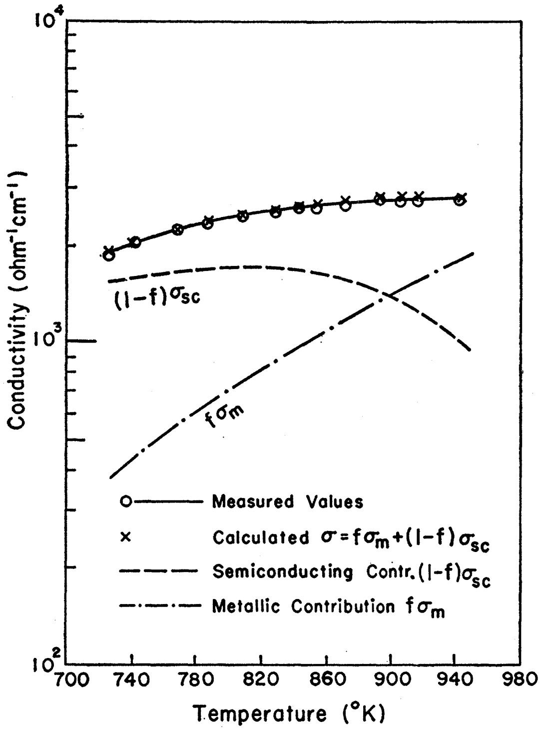

FIG. 9. Semiconducting and metallic contributions to the conductivity of liquid tellurium (after V. A. Johnson[19]).

tellurium atoms in chains. This fraction will be larger at temperatures near the melting point than at very high temperatures where chains of appreciable length will become increasingly improbable.

A possible explanation for the observed electrical behavior may be the following. In general one can assume tellurium ions, free electrons, and tellurium atoms bound by covalent forces into chains of various length to be present in liquid tellurium. The chains will be ionized giving off free electrons which leave behind holes, i.e., missing covalent bonds, which in turn are able to migrate within the chains.

At temperatures immediately above the melting point the chain structure of the liquid seems to be so predominant that conduction by holes dominates conduction by free electrons due to the larger mobility of the holes. As a consequence the Hall coefficient and the thermoelectric power have positive signs. As the temperature increases, the concentration of tellurium ions will increase and the number of covalent bonds and, therefore, of holes will decrease until at very high temperatures the liquid will be composed solely of tellurium ions held together by the same number of free electrons. In this way one might understand that the Hall coefficient and the thermoelectric power reverse their sign and become negative at higher temperatures. The two reversal temperatures do not have to coincide because the condition for sign reversal is in general different for the two quantities.

Johnson[19] succeeded in expressing the conductivity of liquid tellurium as a sum of a metallic conductivity σ_m and a semiconducting conductivity σ_{sc}, each weighted by the probability of finding the tellurium atoms ionized or bonded in chains, as

$$\sigma = (1-f)\sigma_{sc} + f\sigma_m,$$

where f is a Boltzmann factor $e^{-W/kT}$. Since the tangent of the $\log\rho$ *vs* $1/T$ curve in the liquid at the melting point corresponds to the forbidden energy gap E_G of solid tellurium, σ_{sc} can be found from $\sigma_0 \exp(-E_G/2kT)$ with σ_0 determined from the conductivity of the liquid at the melting point. The metallic conductivity σ_m is assumed to be inversely proportional to the absolute temperature, $\sigma_m = A/T$, with A determined from the conductivity maximum. Figure 9 shows that good agreement with the experimental results is obtained with an energy W of approximately $(0.48 - 4.8 \times 10^{-4} T)$ ev.

The interpretation of the discontinuous decrease at the melting point of the resistivity, the Hall coefficient, and the thermoelectric power is complicated by the presence of two kinds of carriers in the liquid as well as in the solid. Thus one must consider possible changes in the concentration of electrons and of holes and a change of their respective mobilities to take place during fusion.

Since the slopes of the resistivity curves in the solid and the liquid are approximately equal at the melting point, the energy necessary to dissociate an electron from the filled octet shells of the tellurium chains seems not to change greatly on fusion. The density of states, however, a parameter which depends sensitively on the lattice structure must be expected to change at the phase transition this causing a change in carrier concentration. It is difficult to estimate the effect of melting on the effective mass of the holes since the existence of the holes depends on the existence of the covalent bonds which are still intact in the liquid.

The mobilities of the charge carriers will decrease on melting due to the higher degree of disorder in the liquid. Furthermore any change of the effective masses will also change the mobilities.

The anisotropy of the solid which seemingly disappears upon melting can probably be observed even in the liquid if one succeeds in aligning the tellurium chains parallel to each other. This could perhaps be achieved by producing a laminar flow of liquid tellurium through narrow containers but such measurements have not yet been performed.

Evidence in the liquid for the presence of aggregates of nonmetallic binding, which resembles closely the type of binding in the corresponding crystalline states,

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[19] V. A. Johnson, Purdue University Report, 1954 (unpublished); Phys. Rev. 98, 1567 (1955).

has been found in many liquid alloys which belong to the compound systems of Sauerwald.[20]

In conclusion, one may say that the assumption of chains in the liquid and a gradual change of predominantly metallic bonding at very high temperatures makes it possible to understand the semiconducting properties of liquid tellurium and the gradual transition to metallic conduction at high temperatures.[21,22]

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[20] F. Sauerwald, Z. Metallkunde **35**, 105 (1943); **41**, 97 (1950); **41**, 214 (1950).

[21] After this work was completed we learned that there are a number of Russian papers[22] which discuss liquid semiconductors. However, these papers do not contain any Hall effect measurements and are not carried out with material of the purity which we have been able to produce.

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Electrical Properties of Boron Single Crystals*

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The electrical resistivity, Hall coefficient, and thermoelectric power of microscopic boron single crystals have been measured as a function of temperature. Rectification and photoconductivity effects were observed at room temperature. The large majority of these single crystals had a high resistivity (about 1.7×10^6 ohm cm at 300°K) which decreased by a factor of nearly 10^{10} as the temperature was increased from 200°K to 1000°K. A few crystals, however, had a much lower resistivity at 300°K (less than 100 ohm cm) and at this temperature their resistivity decreased much more slowly with increasing temperature. Such behavior is attributed to donor impurity levels. From data in the high-temperature range (800°K to 1000°K) the energy gap between the filled band and conduction band was calculated to be 1.55 ± 0.05 ev. Hall, thermoelectric, and rectification studies indicated that hole conduction predominated for all crystals at high temperatures but that electron conduction predominated for the low-resistivity crystals near 300°K. The mobilities of the charge carriers at 300°K were found to be the order of one cm^2 volt^{-1} sec^{-1} with the hole mobility the larger. Non-ohmic conduction was observed and shown to arise from Joule heating.

I. INTRODUCTION

OF the semiconducting elements, boron is perhaps the least understood with regard to the electrical properties of single crystals. Theoretical calculations of the electronic structure are difficult, owing to the extremely complicated crystal structure. Hoard, Geller, and Hughes[1] find 50 atoms in a tetragonal unit cell having $a_0=8.73\pm0.02$ A and $c_0=5.03\pm0.02$ A. Forty-eight atoms are at the vertices of four nearly regular icosahedra which are centered tetrahedrally and linked so that each boron atom has six bonds to the corners of a pentagonal pyramid. The mean distance to the six neighbors is between 1.75 and 1.80 A. The remaining two atoms form only tetrahedrally directed bonds to complete the network. Experimentally, adequate resistivity and Hall measurements on single crystals have not been reported because of the lack of large specimens.

The electrical resistivity of polycrystalline boron was first measured by Weintraub[2] and later by Warth,[3] Freymann and Stieber,[4] and others; a summary has been written by Moss.[5] More recently, the thermoelectric power and other electrical properties of polycrystalline samples have been reported by Lagrenaudie,[6] by Uno, Irie, Yoshida, and Shinohara,[7] and by Jacobsmeyer, Friedrich, and Badar.[8] These investigations show that boron is a semiconductor with hole conductivity predominant at high temperatures.

The present investigation[9,10] is the first in which single crystals have been studied. This work was made possible by the development of micromanipulative techniques previously reported.[11]

The single crystals were grown by Huibregtse and

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* This work was performed in the Ames Laboratory of the U. S. Atomic Energy Commission.

† Present address: Naval Ordnance Test Station, China Lake, California.

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[7] Uno, Irie, Yoshida, and Shinohara, J. Sci. Research Inst. Tokyo **47**, 216 and 223 (1953). A typographical error seems to exist in Fig. 3, p. 218. The activation energy 0.44 ev should probably read 1.44 ev.

[8] Jacobsmeyer, Friedrich, and Badar, Office of Ordnance Research Project No. 627, Final Report, St. Louis University, October 15, 1954 (unpublished); Phys. Rev. **91**, 492 (1953); **94**, 808 (1954).

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[11] Shaw, Hudson, and Danielson, Rev. Sci. Instr. **26**, 237 (1955); W. C. Shaw, Rev. Sci. Instr. **26**, 238 (1955).