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SECURITY INFORMATION

DAVID SARNOFF RESEARCH CENTER

TWELFTH

INTERIM REPORT

INFRARED PHOTOCONDUCTORS

N6onr-23603

October 15, 1952 - January 15, 1953

CONFIDENTIAL
SECURITY INFORMATION

RADIO CORPORATION OF AMERICA
RCA LABORATORIES DIVISION
PRINCETON, N. J. April 3, 1953

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INTERIM REPORT

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I. Laboratory Equipment

A. Cryogenic Sample Holders

For purposes of clarification in these reports and as a matter of record, Table I below describes the sample holders now available or under construction for measurements at low temperatures. The numbered units are "external" cryostats and the lettered units are for use in the Collins.

TABLE I

Cryostat	Type	Original Use	Remarks
1	Small Pyrex	Spec. Resp.	He pot converted to metal (1/53)
2	Large Pyrex	Spec. Resp.	He pot converted to metal (1/53)
3	Metal & Pyrex	Abs. Resp.	For use with black body source
4	Small Metal	Hall Effect	Converted for spec. resp. (1/53)
5a, 5b	Large Metal	Spec. & Abs. Resp.	Under construction (1/53)
A	For Collins	Cond.	For glass slide samples
B	For Collins	Cond.	Enclosed chamber & heater
C	For Collins	Cond.	Flap for ΔI measurement
D	For Collins	Hall & Cond.	Under construction (1/53)

B. Cryogenic Sample Holders: External Cryostats

One of the runs during this period was made to demonstrate the spectral response of a germanium cell at low temperature using cryostat #2. This run was unsuccessful since the cell apparently cooled only to about 50°K. Operation of this cryostat previously resulted in a cooling of the cell to about 20°K. To eliminate this difficulty, the helium pots of cryostats #1 and #2 are being converted to metal to insure better thermal conductivity. The sample is to be soldered to the base of the helium chamber.

Cryostat #4, originally built for Hall measurements, is being converted for spectral response measurements with the Leiss. The beam will enter this unit through a window in its bottom. A disk type of sample will be soldered to the base of the helium chamber. Such optics are desirable to permit measurements at very long wavelengths to be made by the group at Ohio State.

C. Cryogenic Sample Holders: Collins

A new sample holder for the Collins, #D, is under construction. It will support a plug-mounted sample with conductivity and Hall probes at the center of a solenoid to permit measurements of carrier mobility and concentration over the range from 300°K to 2°K. The sample temperature is measured by a gas thermometer and a thermocouple. A flap will be provided to permit illumination of the sample with room temperature radiation.

The advantages of making Hall measurements directly in the Collins is the ease with which the sample temperature can be controlled. Conductivity measurements of the same sample would be made in the same run. Several conditions are imposed upon the magnet:

a) An air coil solenoid is used. The magnetic field through the center of the coil will thus be independent of any temperature-dependent properties of materials (except the electrical resistivity of the windings) and will depend only on the current through the coil.

b) The size of the coil must be such that it will fit in the Collins with the sample at its center.

c) A field of at least 1000 gauss is desired with sufficient uniformity over the sample.

d) The power dissipation must be small enough so that the sample will not be heated during a measurement.

D. Monochromator and Associated Equipment

The optical unit housing a thermocouple detector and focusing mirrors was installed at the exit slit of the monochromator and satisfactory performance was achieved in obtaining a final image of the exit slit in the two planes desired for photoconductive or absorption measurements.

In order to flush the monochromator system with dry nitrogen gas, rubber gaskets were installed at the exit and entrance slit flanges and two cocks were installed in the cover.

A motor driven drive for the monochromator wavelength drum was designed and built during this period. A manual change of gears permits either a free running drum or a drive at 2 or 0.5 RPM.

A motor driven chopper was designed to mechanically interrupt the beam between the source and the entrance slit at either 25 or 12.5 cps. The choice of frequency is made by a manual adjustment of the two chopping blades.

A control box for the Leiss Monochromator and Thermocouple Unit was constructed during this period. From this unit are controlled the chopper, the source, the prism heaters, wavelength marker, prism drum drive, and lights at the drum and entrance and exit slits.

E. Electrical Equipment

The battery box used for the main amplifier was revamped to provide voltages for the 12.5 cps band-pass amplifier and thermocouple preamplifier by the use of separate cables to these units. Additional changes also now provide negative voltages to 22-1/2 volts to be applied to the cell.

The thermocouple preamplifier was tested during this period. Considerable noise was found to come from the bias cell; cathode bias is now being used. The 12.5 cps band-pass amplifier was found to oscillate when the twin-T feedback was used. This trouble has not yet been remedied since ample signal from the thermocouple detector occurs using 25 cps chopped radiation.

F. Collins Liquifier

In run 81 during this period, the Collins reached a temperature of only 160°K after running six hours. The cryostat pressure was abnormally high and the engine inlet pressure decreased abnormally in cooling. These latter effects had been observed in the latest previous runs. Since a leak around the engines was suspected, a general overhaul of the engines and cryostat chamber was made. New engine gaskets and a new stuffing box were installed. The working parts of the Collins were apparently in good condition. At the same time, the oil in the compression crankcase was changed. Up to this run, the unit had been in operation for about 500 hours.

G. Crystal Pulling Assembly

In view of the apparently almost complete loss of zinc by volatilization in the first attempt at preparation of zinc-doped germanium, the crystal drawing apparatus was modified in such a manner as to permit addition of foreign atoms, stirring of the melt and drawing of the crystal all in rapid succession without the necessity of cooling or disassembling the furnace tube. This has been accomplished by mounting the graphite doping and stirring rod beside the crystal pulling rod in a manner such that the former can be raised or lowered relative to the latter from outside the furnace tube. The foreign element is introduced by lowering the graphite rod into the molten germanium while the crystal pulling rod is in a raised position. Stirring is accomplished by rotating the entire pulling rod assembly. When crystal growth is to be begun, the graphite rod is raised, the crystal pulling rod is lowered and the crystal is grown in the usual manner.

II. Temperature Dependence of Conductivity

The analysis of the log conductivity versus inverse temperature curve that was described in the last report has been extended to the case of helium- and lithium-like impurities, for example, zinc and gold, respectively. Equations have been derived giving the temperature dependence of the carrier concentration as a function of impurity content and binding energy of the carriers.

We will present here the theory for the case of helium-like donors, say selenium. See the last report for symbols not described here.

At any temperature, let n_2 be the concentration of unionized selenium which requires an energy E_2 to singly ionize it. Let n_1 be the concentration of singly ionized selenium which requires an energy E_1 to remove the last electron. Chemically,

$$\begin{cases} \mathrm{Se} \rightleftharpoons \mathrm{Se}^{+} + \text{electron} \\ \mathrm{Se}^{+} \rightleftharpoons \mathrm{Se}^{++} + \text{electron} \end{cases} .$$

Applying the law of mass action, where N is the concentration of selenium,

$$\begin{cases} n_1 n/n_2 = K_2 = N_c \exp(-E_2/kT) \\ (N - n_1 - n_2)n/n_1 = K_1 = N_c \exp(-E_1/kT) \end{cases} .$$

These equations can be solved for n_1 and n_2. The resulting form is similar, but not identical, to a Fermi-Dirac distribution. Applying electrical neutrality,

$$n + N_A = n_1 + 2(N - n_1 - n_2)$$

which becomes

$$n + N_A = N(n + 2K_1)/(n + K_1 + n^2/K_2) \ .$$

This equation corresponds to equation (1) in the last report if $n \gg p \ll K_A$, i.e., below room temperature.

The derivation for the lithium-like case runs similarly.

The resultant form of these equations at low temperatures is very dependent on the relative value of the n- to p-type impurity concentration. In attempting to fit curves with the equations derived in the last report, near balance of impurity types was found to be necessary. This poses the question of whether the introduction of a given amount of chemical impurity of one type requires the formation of an almost equal concentration of opposite type of center. For example, if v atoms of impurity enter the lattice substitutionally, do v atoms of the same impurity enter the lattice interstitially for stability? Or is there associated with each substitutional impurity a structural defect that acts as a center of the opposite type?

The general behavior of these derived equations is being studied in an attempt to analyze the data available in terms of the impurity content and binding energies.

III. Measurementsupon Germanium

A. Behavior of Contacts

The cooling curves of nearly all of the germanium cells described to date have been obtained from samples provided with two current contacts only. It is, of course, obvious that with such an arrangement, rather than with one employing potential probes, the resistance of the contacts is in series with that of the sample. This procedure has been adopted, in spite of the uncertainty which might be encountered in assigning observed effects to the bulk germanium on the one hand or to the germanium-metal contact on the other, since the cells when used as practical radiation sensitive elements will most likely be connected in this manner.

The theory of the behavior of germanium as a semiconductor is sufficiently well developed so that an estimate of the reliability of a given cooling curve can be made simply from a qualitative inspection of the shape of the curve. In most cases, the curves which have been obtained are in essential agreement with theoretical expectations. For some samples, however, curves which definitely appear anomalous have been obtained. It is desirable, for such, to determine whether the anomalies are due to the germanium-metal contact or to effects in the bulk of the germanium. In addition, it has not yet been determined to what extent the long wavelength infrared photoconductivity observed at low temperatures depends upon the nature of the contacts.

For these reasons, an investigation of the effect of the contacts upon the behavior of germanium cells has been undertaken. If the behavior is found to be independent of the kind of contacts employed, then it would be reasonably certain that bulk properties of germanium are being observed.

The contacts which have been used heretofore have been made by electroplating small areas of copper upon the freshly etched germanium sample. Fine copper wires were then fastened to the copper-plated areas by means of ordinary soft solder. Before any low temperature measurements are made upon a given sample, the quality of the contacts is determined by measuring the current-voltage characteristic at both room and at liquid nitrogen temperatures. If the contacts are not ohmic at these temperatures, the contacts are removed and reapplied. Satisfaction of this criterion is a necessary but not a sufficient condition to insure satisfactory low temperature behavior. Some samples which have been ohmic at liquid nitrogen temperatures have, nevertheless, given anomalous cooling curves.

The modifications in contact preparation which have been tried consist of the elimination of the copper-plated layer and of the use of materials other than lead-tin alloys as solder. Materials which have been tried include:

1) Indium; mp. 156°C
2) Indium-bismuth eutectic; 33 wt% Bi; mp. 72°C
3) Bismuth-tin eutectic; 57 wt % Bi; mp. 139°C
4) Indium-zinc eutectic; 96 wt % In; mp. 144°C

The initial set of experiments was made upon a number of doped samples of comparatively low resistivity which had previously been measured with copper-plated contacts. This set includes cells AA53, 70, 73, 74 and 95. Some of these samples had shown normal behavior with copper contacts while others had been abnormal. The impurity slopes and low temperature long wavelength photoconductive responses of these cells, both with copper-plated and with modified contacts are listed in Table II.

Cell AA73 may be taken as an example of one which appeared to show normal behavior with copper-plated contacts. The cooling curves obtained with these and subsequently with indium-bismuth contacts are illustrated in Fig. 1. The agreement between the two sets of measurements, both with regard to the shape of the cooling curves and with regard to the magnitude of the photoresponse, is essentially quantitative.

Outstanding examples of the effects of unsatisfactory contacts are to be found in cells AA70 and AA95. Although both of these cells with copper-plated electrodes satisfied the ohmicity criterion reasonably well, the cooling curves illustrated in Figs. 2 and 3 are highly abnormal. The same samples, when provided with modified contacts, gave the cooling curves, also shown in Figs. 2 and 3, both of which more or less exemplify normal behavior. The photocurrents were improved about one order of magnitude by the change in contacts.

TABLE II

Germanium Cells Measured

Cell	Added Impurity	Contact Type	Impurity Slope	Photoconductance at 4°K(a)
AA17	None	Cu-plate	0.040 ev.	0.11 µA/v
*AA53	Bismuth	Cu-plate	0.012	≈0.4
AA53	Bismuth	Bi-Sn	Contact opened during run	
*AA70	Bismuth	Cu-plate	0.012	0.046
AA70	Bismuth	In	0.012	0.30
*AA73	Indium	Cu-plate	0.0125	1.11
AA73	Indium	In-Bi	0.0120	1.12
*AA74	Indium	Cu-plate	0.0094	1.3
AA74	Indium	In	0.0085	7.
AA94	Gallium	Cu-plate	0.0090	3.3
*AA95	Arsenic	Cu-plate	No well-defined slope	0.10
AA95	Arsenic	Bi-Sn	0.012	3.0
AA118	None	Cu-plate	~ 0.006	0.10
AA120	None	Cu-plate	0.044	0.017
AA122	None	Cu-plate	0.043	0.0006
AA124	None	Cu-plate	No well-defined slope	0.14
AA141	Zinc	Cu-plate	0.011	0.087
AA144	Zinc	Cu-plate	0.013	0.038
AA145	Thallium	Bi-Sn	0.014	0.041
AA146	Thallium	In	0.015	0.20

(a) For one volt applied to cell.
* Data from a previous run.

The results of the study of contact behavior up to the present might be summarized by the statement that, although it is possible to obtain satisfactory copper-plated contacts, it has been found to be generally much easier to obtain modified contacts which show reasonable behavior. Usually, the first attempt at application is successful in the case of a modified contact, whereas it has frequently been necessary to reapply copper-plated contacts several times to a given sample before satisfactory behavior has been achieved. The next step in the investigation of contact behavior is the study of the behavior of very high purity samples. This is currently under way.

One possible difficulty with the modified contacts which was anticipated but which has apparently not been encountered is the following. Suppose one had, for example, a p-type germanium sample. It might be expected that the behavior of this sample would be different depending upon whether the contact material was one which acted as an n-type or as a p-type impurity in germanium. In the former case, a p-n junction might be established at the contact. If this occurred, the measured properties of the sample would largely be characteristic of the junction rather than of the bulk germanium. There have been, thus far, no effects observed with modified contacts which can be ascribed to this cause.

B. Current-Voltage Characteristics

For practically all of the samples whose current-voltage characteristic has been determined at liquid helium temperature, deviations from ohmicity of one type or another have been observed. For samples with copper-plated electrodes, it was felt that these deviations might be due to the contacts. The generally more satisfactory behavior of samples with modified contacts has made possible a re-examination of the problem of the dependence of current upon voltage at very low temperatures.

No attempt has been made to examine in detail the current-voltage characteristic at intermediate temperatures (between 77°K and 4°K), since this would require the precise maintenance, for considerable intervals of time, of constant temperature at the samples in the Collins Cryostat. However, during the course of the determination of conductance as function of the temperature, the voltage applied to the cells is increased as the sample resistance becomes greater at lower temperatures. Any appreciable non-ohmicity would be apparent as a discontinuity in the cooling curve at those points where the voltage has been changed.

Although copper-plated samples have occasionally shown such discontinuity, samples with modified contacts (with one exception) have shown none over the entire temperature range. However, at 4°K, these samples have shown rather remarkable deviations from ohmicity. Typical examples of this behavior are illustrated in Fig. 4 for photocurrents in cells AA70, 95 and 146. At low voltages, the current-voltage characteristic approaches linearity. As the voltage is increased, deviations from linearity become more pronounced until at a farily well deviations from ohmicity of one type or another have been observed. For samples with copper-plated electrodes, it was felt that these deviations might be due to the contacts. The generally more satisfactory behavior of samples with modified contacts has made possible a re-examination of the problem of the dependence of current upon voltage at very low temperatures.

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At voltages below the breakdown, the conductance is independent of the polarity of the applied voltage in every case. If the non-linear current-voltage characteristic were determined by the nature of the contacts, this reversibility would require that the contact area at both contacts on each sample be identical. Since this is rather unlikely, the phenomenon is probably a bulk effect. It may be noted that this type of non-linearity has also been observed in samples having copper-plated contacts. In every case, the cells exhibiting this behavior have had fairly low resistivity and have had impurity slopes in the neighborhood of 0.01 ev.

A consequence of the existence of this non-linear behavior is a dependence upon voltage of the photoconductance. An idea of the magnitude of this dependence is given in Table III in which the photoconductance expressed in microamperes per volt is given at several voltages below breakdown for the same cells whose current-voltage characteristics are illustrated in Fig. 4. Because of this effect, the comparison of the photoconductive response of different cells is somewhat arbitrary.

TABLE III

Dependence of Photoconductance upon Voltage

Voltage	Photoconductance		
	AA70	AA95	AA146
1.0 volt	0.30 µA/v	3.0 µA/v	0.20 µA/v
2.0	0.61	8.8	0.28
3.0	1.20	20.0	0.43
4.0	2.22		0.70
5.0	4.3		1.57

C. Doped Samples

Much of the work done during this period on samples containing deliberately added impurities has already been discussed in Section III (A). Samples containing impurities (hydrogen-like) from either column III or V of the Periodic Table have all shown slopes in the neighborhood of 0.01 ev. The slopes for the two thallium-doped samples, AA145 and 146, are somewhat larger than for the others but this may not be significant.

The photoconductances of the samples with hydrogen-like impurities are among the largest which have been observed to date in this work. The impurity concentrations in these samples are large enough so that, upon cooling from room temperature, the dark conductance rises. This implies that the concentrations are somewhere in the neighborhood of 10^{15} cm^{-3}. The optimum photoconductivity will apparently be exhibited by material containing a higher impurity concentration than was previously thought desirable.

The theory outlined in Section II indicates that germanium doped with helium- or lithium-like impurities might exhibit larger activation energies than does germanium containing hydrogen-like impurities.

There is some experimental evidence available to indicate that this is the case. Such materials, if their absolute photosensitivity is comparable to that of the samples with hydrogen-like impurities, will be more useful as photoconductors in the 8 to 15 micron range than are thosewith a 0.01 ev activation energy.

One attempt has been made to obtain a germanium crystal which has been doped with zinc, a helium-like impurity. Because of the volatility of zinc at high temperatures, it is believed that essentially none remained in the grown crystal. The two cells, AA141 and 144, from this crystal, therefore, are believed not to be characteristic of zinc-doped germanium. Another attempt at zinc doping, making use of the modified crystal pulling apparatus described in Section I(G), is planned.

IV. Measurements upon Other Materials

A. Silicon

A sample of single crystal silicon (0.8 ohm cm at room temperature) was obtained from another group in the Laboratories. This sample has been measured in order to obtain some idea of the relative long wavelength photoconductive response of silicon as compared with germanium. Two needle-shaped polycrystalline samples of Johnson & Matthey silicon were also measured. The behavior of these three silicon samples is summarized in Table IV. Cooling curves for two of these samples is given in Fig. 5.

TABLE IV

Silicon Samples

Cell	Material	Contact Type	Impurity Slope	Photoconductance at $4^{\circ}K$
AD4	J.& M.	Cu-plate	≤ 0.03 ev.	3×10^{-5} μA/v
AD5	Single Crystal	Cu-plate	0.046	2.2×10^{-3}
AD7	J.& M.	Cu-plate	0.034	9×10^{-6}

It is interesting to note that the photoconductive response of the single crystal sample is about one hundred times larger than that of the polycrystalline samples. Also of interest is the fact that the response of the single crystal silicon sample is considerably smaller than that of most of the germanium samples which have been measured.

B. Zinc Antimonide

A sample, H11, having the nominal composition Zn_3Sb_2 and for which measurements to 100°K have been reported in the Eleventh Interim Report (N6onr23603), has now been measured to helium temperatures. The resulting cooling curve is shown in Fig. 6. The complete independence of conductance upon temperature between 30°K and 4°K indicates that there is no condensation of carriers into impurity centers upon cooling to low temperatures. One would expect, therefore, to find no impurity photoconductivity at low temperatures. None was observed. Further investigation of this material seems unprofitable at present.

C. Cadmium Antimonide

Measurements have now been made, over the range from room temperature to about 100°K, upon crystals, of nominal composition CdSb, grown by the Kyropolous technique as described in the Eleventh Interim Report. A crystal as grown gave the cooling curve illustrated in Fig. 7(A). The conductance increased with decreasing temperature over the range covered. This result, which is quite different from that exhibited by polycrystalline CdSb (See Fig. 7 of the Eleventh Interim Report), may have been due (1) to composition changes which occurred because of sublimation from the melt during growth of the crystal or (2) to incomplete compound formation in the solid because of too rapid cooling during growth (see Tenth Interim Report).

In order to determine, if possible, which of these factors was responsible for the shape of the observed cooling curve, a sample of the grown crystal was annealed in vacuum at about 350°C for about 92 hours. This sample gave the cooling curve of Fig. 7(B) which shows a region of decreasing conductance below about 170°K. This indicates that (2) is responsible, in part at least, for the behavior of the unannealed crystal.

Rough spectral response measurements have been made upon several cadmium antimonide preparations cooled to liquid nitrogen temperature. Samples having compositions in the neighborhood of CdSb showed a measurable response with a long wavelength threshold lying at approximately 3 microns. This appears to be the intrinsic response since a value of about 0.5 ev has been reported in the literature for the intrinsic gap of CdSb.

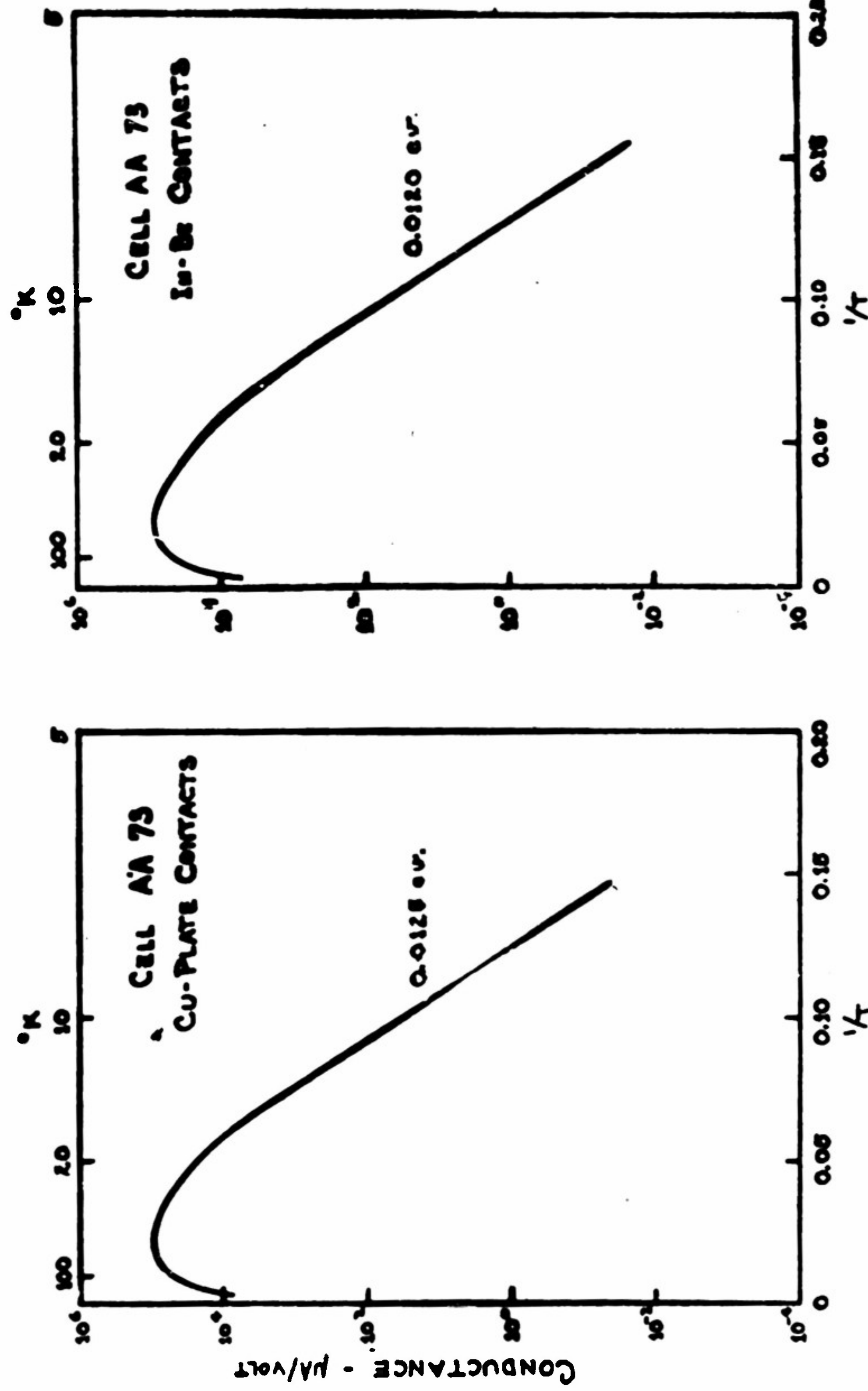
A very slight response to the unresolved radiation from an infrared source has been obtained with Cd_3Sb_2 preparations. This response was too feeble to permit spectral response measurements. It may be due to the presence of small amounts of CdSb resulting from the decomposition of metastable Cd_3Sb_2. It is not clear why there was apparently no response of sufficient intensity to be considered characteristic of Cd_3Sb_2 in spite of the fact that this compound shows clear-cut semiconductor behavior insofar as the temperature dependence of conductivity is concerned.

D. Lead Iodide

Three bulk samples of PbI_2, two of which were polycrystalline and one which was single crystal, have been measured. Lead iodide is a fairly good insulator ($\approx 10^9$ ohm cm at room temperature) and shows a considerable intrinsic photoresponse with a threshold in the visible portion of the spectrum.

The dark conductance of the samples was followed upon cooling from room temperature to a point where the currents became too small to measure conveniently. The resulting cooling curves are given in Fig. 8. The slopes of the three curves near room temperature are 0.36, 0.14 and 1.5 ev. This great variation of slope indicates that the samples are in the impurity range at room temperature. The fact that one of the slopes is as small as 0.14 ev indicates that it may be possible to introduce a sufficient concentration of impurity having an activation energy of about 0.1 ev to produce a useful photoconductor in the 8 to 15 micron range.

eb
3/26/53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Conductance - μA/volt

Fig. 1

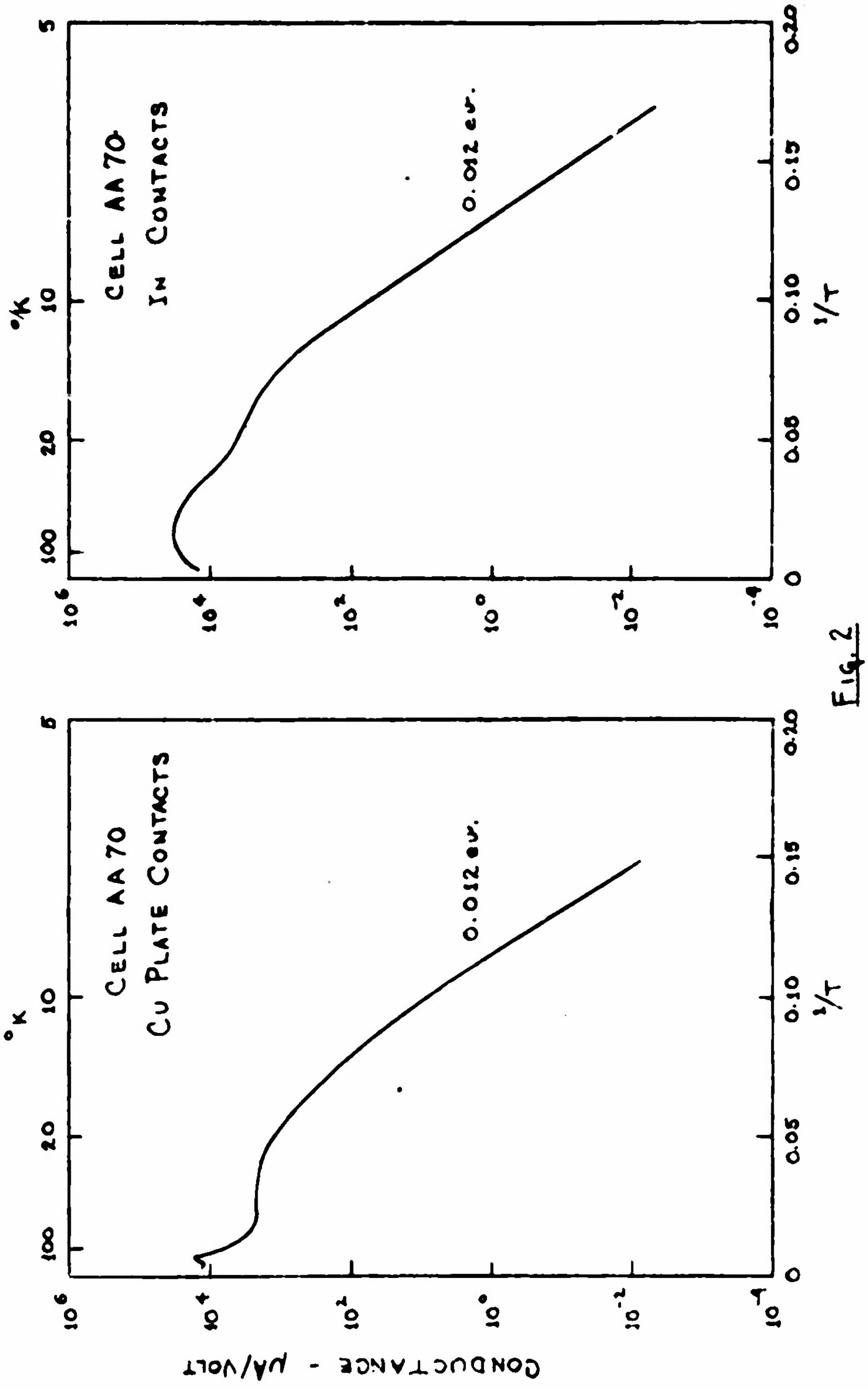
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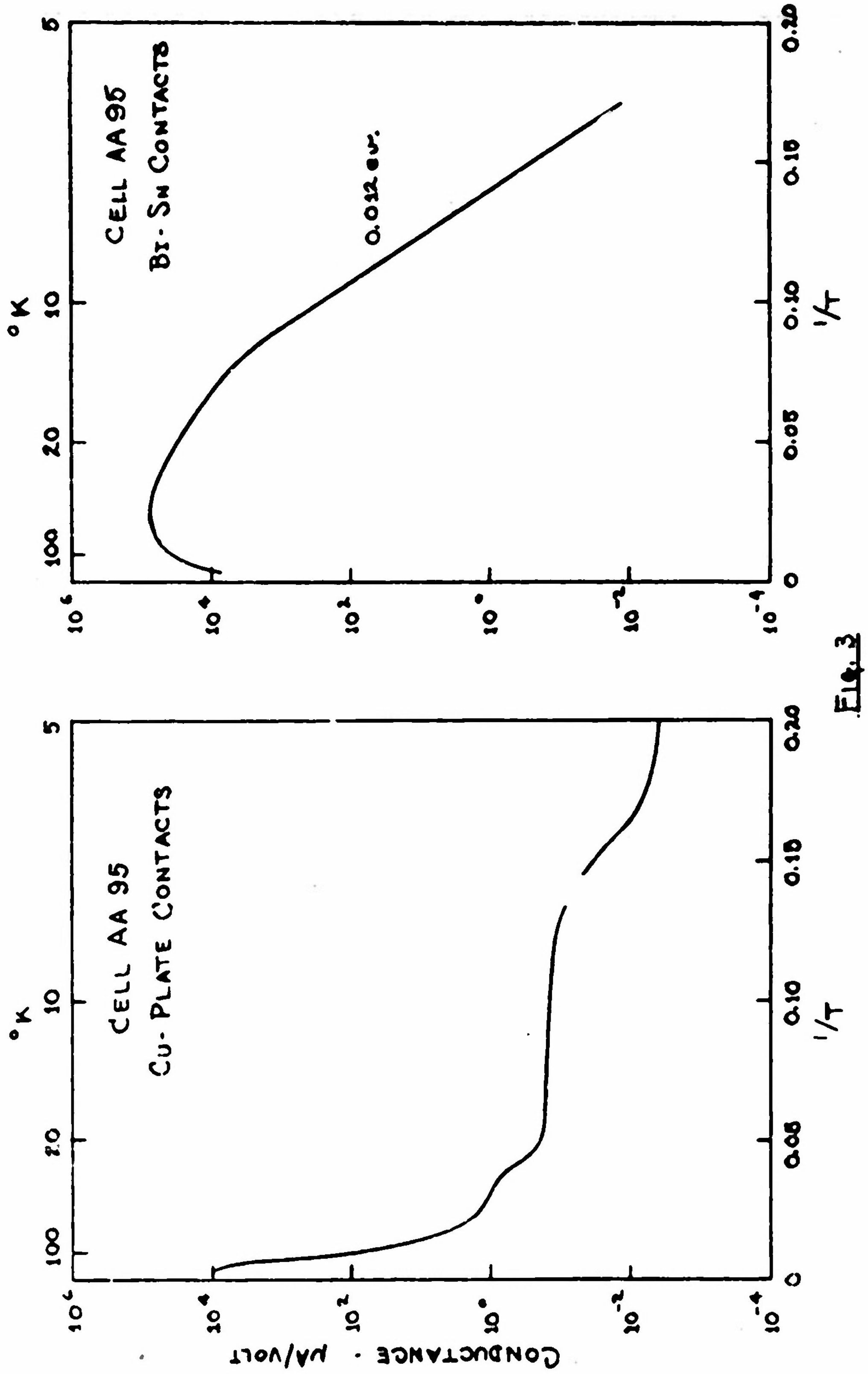
Fig. 2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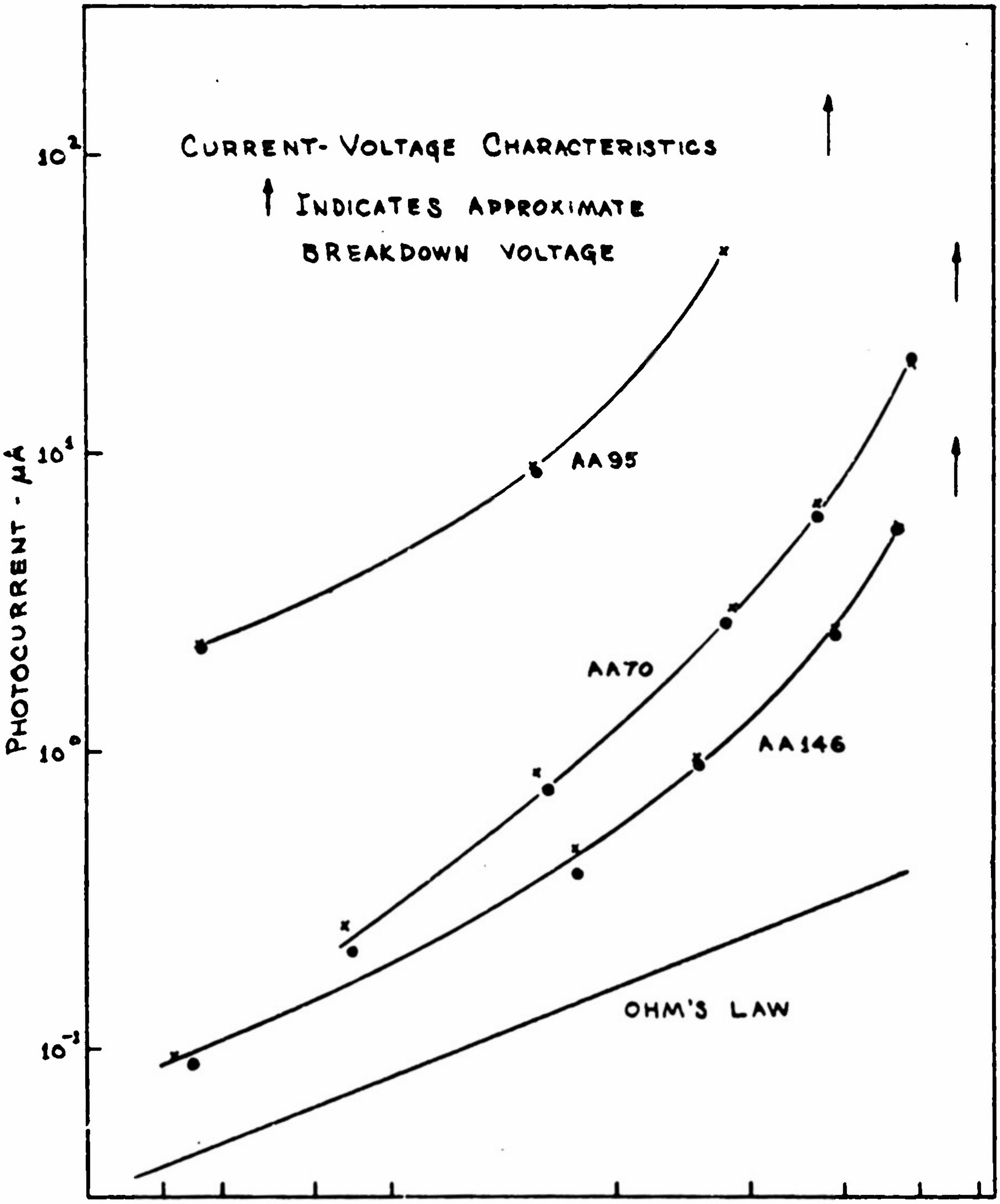
CONDUCTANCE · μA/VOLT

Fig. 3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10^1
10^0
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AA95
AA70
AA146
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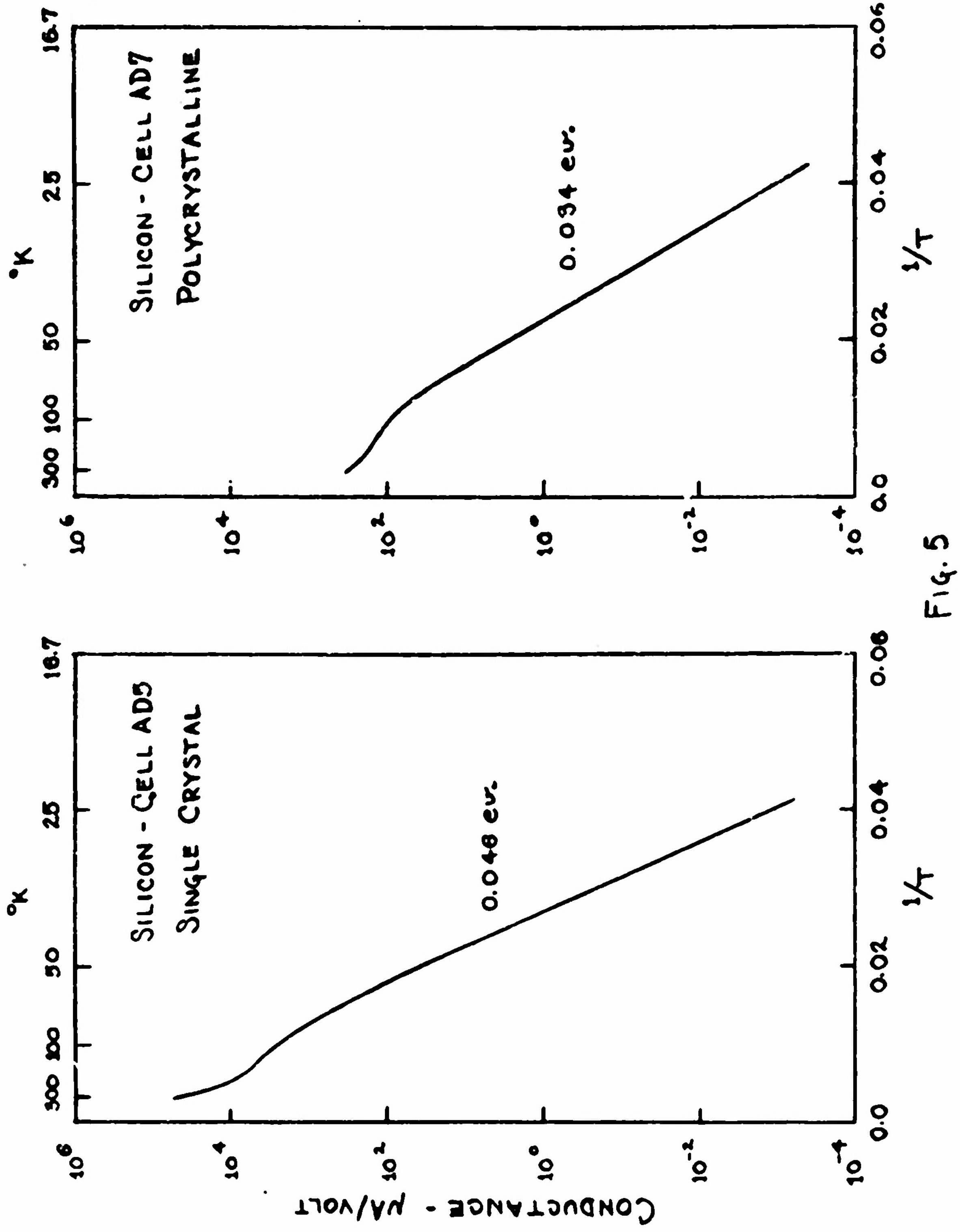
1
2
3
4
5
6
CELL VOLTAGE

Fig. 4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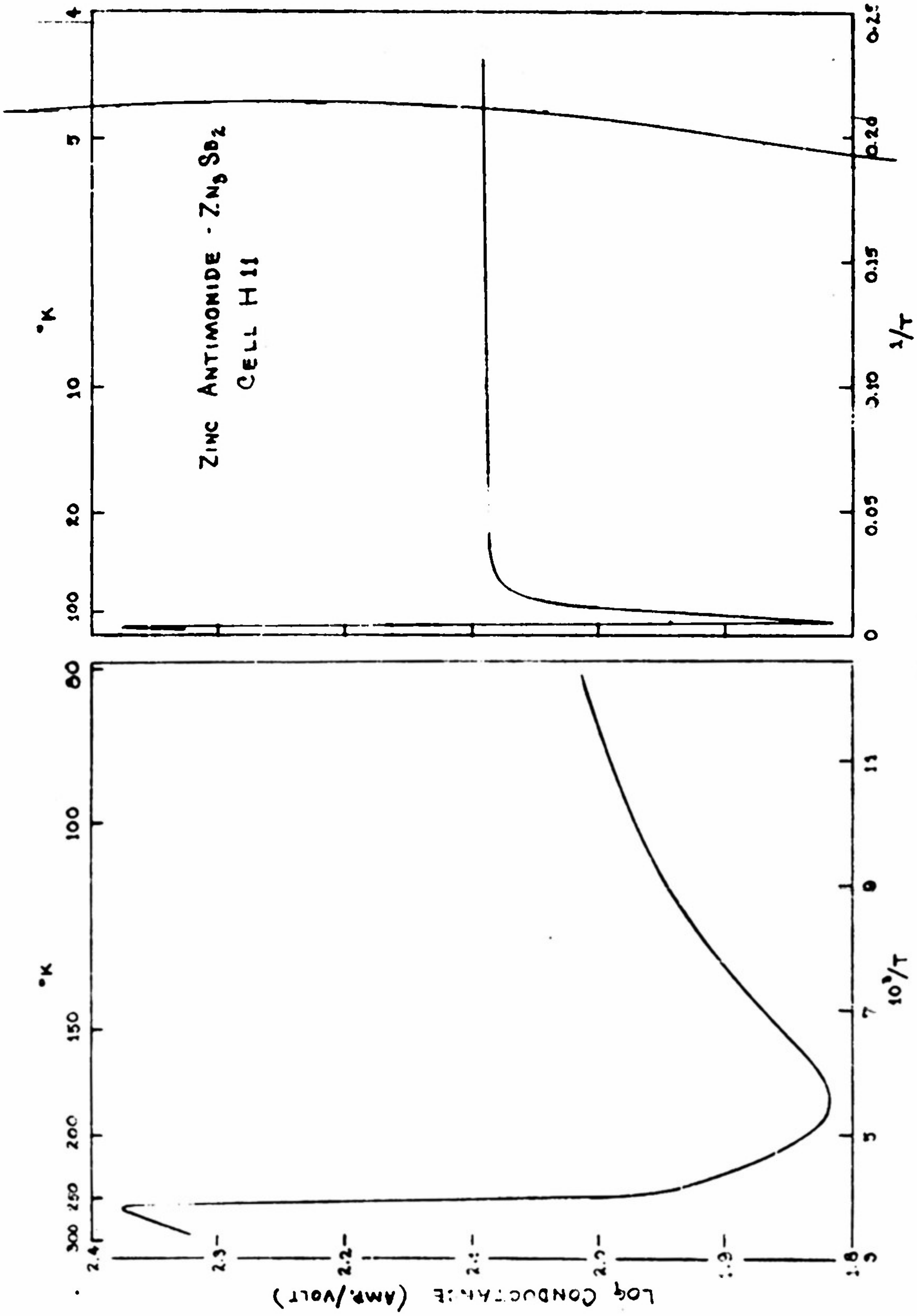
Conductance - µA/volt

Fig. 5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7
5
3
2.4
2.3
2.2
2.1
2.0
1.9
1.8
Log Conductance (amp./volt)

Fig. 6

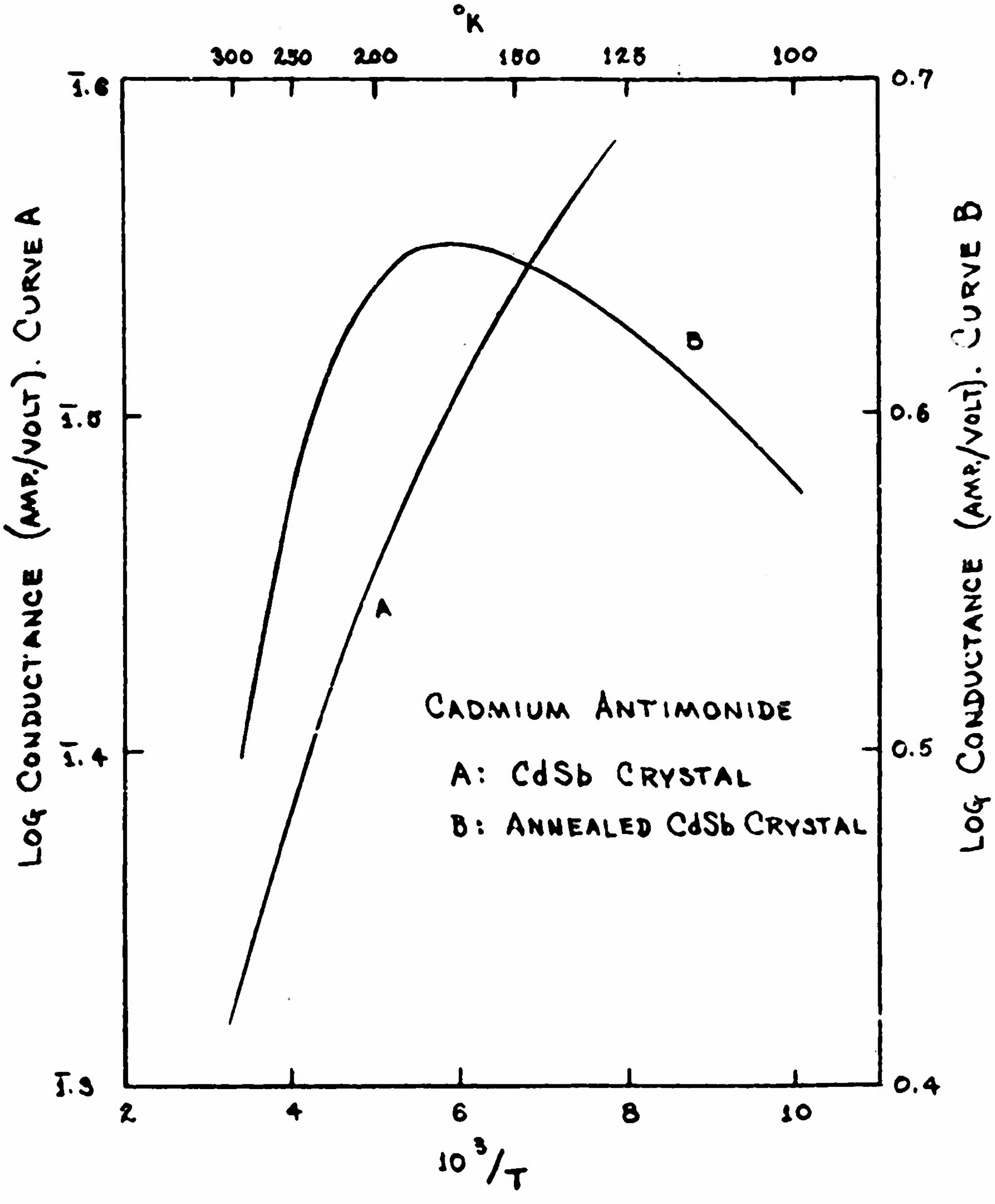
°K
300
250
200
150
125
100
$\bar{1}.6$
$\bar{1}.5$
$\bar{1}.4$
$\bar{1}.3$
0.7
0.6
0.5
0.4
Log Conductance (Amp./Volt). Curve A
Log Conductance (Amp./Volt). Curve B
A
B
Cadmium Antimonide
A: CdSb Crystal
B: Annealed CdSb Crystal
2
4
6
8
10
$10^3/T$

Fig. 7

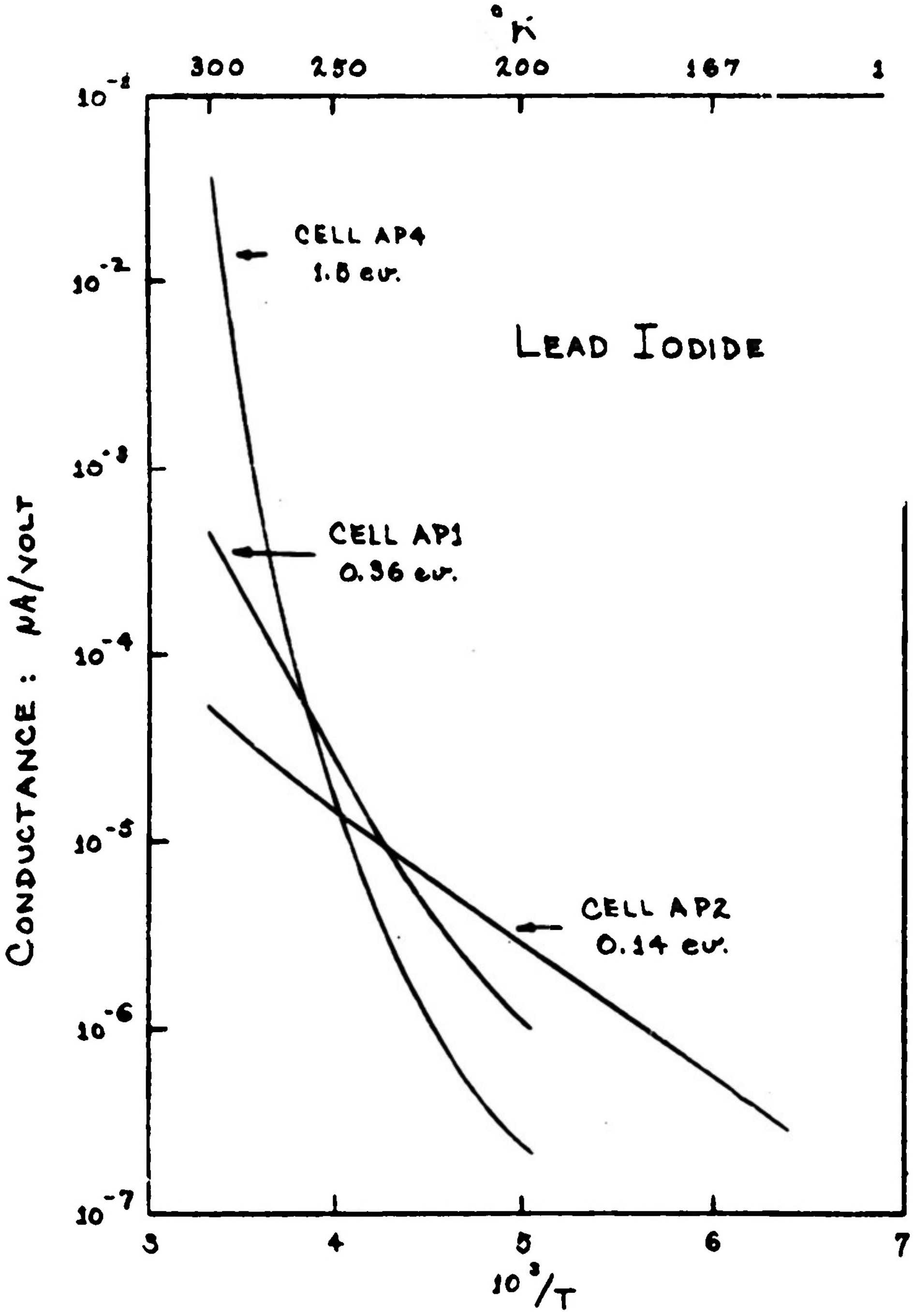
°K
300
250
200
167
1
10^-1
10^-2
10^-3
10^-4
10^-5
10^-6
10^-7
CONDUCTANCE : μA/VOLT
CELL AP4
1.8 ev.
LEAD IODIDE
CELL AP1
0.36 ev.
CELL AP2
0.14 ev.
3
4
5
6
7
10³/T

FIG. 8