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Research Report

Electrical Resistivity of Semiconducting Diamond

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ELECTRONIC MATERIAL SCIENCES LABORATORY PROJECT 5621

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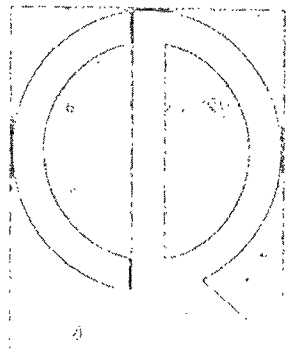

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Abstract

Semiconducting diamond with its wide energy gap, favorable current carrier mobility, and good mechanical properties holds promise of being a useful high temperature semiconductor material.

The data covered includes electrical resistivity measured from room temperature to 500° C in an inert atmosphere. Over the lower temperature ranges, below approximately 250° C, the temperature coefficient of resistivity is high, negative, and nonlinear. At the higher temperatures the coefficient rapidly becomes much smaller. A resistivity minimum is observed between 350 and 400°C. The results reported here constitute part of a program involving studies of relationships between defects in the diamond crystal structure and its semiconducting properties.

Acknowledgments

The authors wish to express appreciation to Dr. E. R. Czerlinsky, Chief of the Properties and Phenomena Section, for helpful discussions and suggestions during the course of this work.

Appreciation is expressed for the loan of the semiconducting diamond sample from Dr. J. F. H. Custers via Dr. C. J. Rauch of the Lincoln Laboratories.

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Electrical Resistivity of Semiconducting Diamond

1. INTRODUCTION

Semiconducting diamond holds promise as a material for use at elevated temperatures for military, as well as other applications. The wide energy gap, favorable current carrier mobilities, high chemical stability, good mechanical properties, and relative simplicity of atomic structure encourage detailed examination of its semiconduction properties.

In the low temperature ranges, the high thermal coefficient of resistance coupled with a high thermal conductivity (larger than for any metal) combine to make this material well suited for thermistors.

Most diamonds found in nature are insulators with resistivities in the order of 10^{14} to 10^{16} ohm-cm. These are classified as Types I and IIa.[1] A few, however, are semiconductors at room temperature. These have been classified as Type IIb by Custers.[2] Their room temperature resistivities have been reported as ranging from 25 to 10^{8} ohm-cm. Other classifications have also been proposed.[3, 4, 5]

In order to obtain information pertaining to the physical nature of defect structures and their influence on semiconduction phenomena in diamond, the need arises for interrelated measurements of diamond properties. With this in mind, the report is directed to a study of electrical resistivity as a function of temperature.

1.1 Objectives

(1) To obtain data (electrical resistivity as a function of temperature) which, when combined with results of other investigations, will contribute to a further understanding of the physical nature of those imperfections which cause semiconduction in diamonds.

(2) To examine techniques for making stable and reproducible electrical contacts to semiconductor diamonds.

(3) To compare results of the d-c technique and a contactless microwave technique.

1.2 Measurement Problems

In making these measurements, complications can arise from several sources, such as space charge effects in the diamond, rectification at the contacts (avoided by using potentiometric techniques), thermoelectric effects (although the very high thermal conductivity of diamond helps to decrease this source of error), photoelectric effects, and nonhomogeneity of samples. The scarcity of Type IIb diamonds decreases the opportunity of selecting samples. Small sample size sometimes necessitates extreme care in attaching and precisely locating electrical contacts or probes. One is limited also as to how high in temperature he can carry experiments for fear of graphitization of diamond.[6, 7, 8] Special precautions must be taken to avoid errors due to the large thermal coefficient of resistance over a wide temperature range.

2. HISTORICAL BACKGROUND

This section contains a few notes abstracted from the literature to indicate the materials used, problems encountered in making electrical contacts to semiconducting diamonds, temperature ranges covered, acceptor activation energies, and electrical resistivities obtained. A few notes are included on atomic models, as suggestions, to account for semiconduction in Type IIb diamonds.

2.1 The Work of Austin and Wolfe

Using "standard d-c methods," Austin and Wolfe[9] measured resistivity of a Type IIb sample from -100°C to +600°C. Electrical contacts to their sample were made by pressing graphite blocks against the ends of the sample and graphite or

copper probes to its edges. Their contacts were "variable and non-ohmic," but of lower resistance for probes placed on edges than on faces of the sample. For rectification measurements, they used a combination of one indium contact and one sharp tungsten contact under pressure.

Austin and Wolfe obtained a resistivity minimum of 1.5 ohm-cm at about 500°C. At 20°C the resistivity was 270 ohm-cm. In the Low temperature range, below -50°C, they obtained a straight line with slope corresponding to an energy of 0.38 ev from a plot of k log ρ vs. 1/T (°K), where k = Boltzmann's constant, and ρ = electrical resistivity.

Their suggestion as to the nature of the imperfections is this: a dissolved impurity such as trivalent aluminum acts as an acceptor (E_{ACT} = 0.38 ev), and these acceptors are partly compensated by donors of an unknown nature. Further, they suggest that although vacancies or other crystal imperfections can also act as acceptor centers, associated energy levels would be much deeper than those measured.

Several workers obtained room temperature activation energies of about 0.35 ev from graphs of resistivity versus temperature. However, as Austin and Wolfe pointed out, resistivity versus temperature curves are straight only below approximately -50° C in which region they obtained the 0.38 ev activation energy. They found that their electrical contact resistances were affected by heat cycling, decreasing by more than a factor of ten after the diamond had been heated and returned to room temperature. However, the use of potential probes resulted in "consistent" resistivity measurements. Leivo and Smoluchowski[10] on measuring resistivity for Type IIb diamonds between room temperature and 425°C found a minimum at about 360°C. At "low temperatures" they obtained an activation energy of 0.35 ev from the slopes of these curves.

2.2 The Measurement of Custers

Custers[11] measured electrical resistivity of a Type IIb sample from about 292 to 323°K. For contacts, thin layers of silver were deposited on two opposite sides and the sample clamped between two silver electrodes. A constant voltage was applied and the sample placed in a furnace. Complications arose since, on applying the voltage, the current would first rise to a maximum and then decrease a few percent. This appeared to be due to polarization, or to a space charge effect. He also observed a hysteresis, in that the resistivity values for increasing and for decreasing temperatures were different. Custers suggested that this was a bulk property of the diamond involving a delay factor for electrons to fall back to lower energy levels after thermal excitation. Austin and Wolfe, however, felt that this

was due to changes in contact barriers rather than the bulk of the diamond, since their potential "probe" method gave consistent results over a temperature range.

2.3 Other Measurement Procedures

Dyer and Wedepohl[12] measured the resistance of a Type IIb sample from 200 to 1040°K. They found log r plotted against 1/T to be linear in the lower temperature ranges from 200 to 290°K. They also found that the resistance became a minimum at about 590°K.

Rodgers and Raal[13] in reporting on semiconducting diamonds as thermistors show a resistance versus temperature plot similar to that of Dyer and Wedepohl. Their current leads consisted of platinum wires, secured by a titanium-silver-copper alloy bonded to the diamond.

Mutch and Raal, [14] in examining effects of heat treating a Type IIb diamond, made electrical contact to a surface which was roughened with 8 to 12 μ diamond powder, using small colloidal silver dots baked in vacuum for 20 minutes at 400°C. They found the contact resistance to be "negligible."

Mutch and Raal favored an atomic model in which acceptors consisted of substitutional group III atoms, and "donor centers" were attributed to displaced carbon atoms. At the temperatures used, thermal energy was not sufficient to overcome the threshold energy required to move the substitutional acceptors. However, the temperatures were high enough for annealing which would allow interstitial carbon atoms to move to vacant lattice sites, decreasing the donor density and the compensation. To substantiate their views, they observed decreases in resistivity with heat treatment.

Brophy[15] used indium solder and silver paint for electrical contacts. However, neither was entirely ohmic at all temperatures. The range of temperatures covered was limited by contact instability at low temperatures. On examining homogeneity of the sample, using floating potential probe measurements with a tungsten probe, he found experimentally reproducible variations in potential from point to point. This nonuniformity of the specimen precluded determination of the semiconductor parameters.

Wedepohl[16] used suspensions of colloidal graphite in water for contacts to Type IIb diamonds for electrical measurements, from about 200 to 800°K. External contact to the graphite was made by spring-loaded copper probes or plates. His high temperature measurements were carried out under vacuum to prevent oxidation of the diamond. The scatter in his measurements was greater than could be accounted for by experimental error. This was attributed to inhomogeneity in the distribution of the imperfection centers responsible for semiconduction. Wedepohl found an

acceptor activation energy of about 0.34 ev. He referred to a model containing acceptors consisting of trivalent impurity atoms, and donors.

Pogodaev[17] used vacuum-deposited silver electrodes on diamonds from 50 to 350°C. He pointed out that diamonds are inhomogeneous in regards to electrical conductivity. Investigations of conductivity versus temperature for Types I and IIa showed considerable evidence of space charge effects within diamonds.

Bate and Willardson[18] made contacts with silver paint to a polished diamond surface which had first been roughened by spreading Number 600 Carborundum powder on the surface and rubbing with a needle. They found such contacts to be ohmic and of very low resistance. Contact was made to the silver paint dots with spring-loaded probes. The strong temperature dependence of resistivity necessitated very precise (within ±0.05°C) temperature control of the diamond. Bate and Willardson found that in some cases the Hall voltage varied considerably, depending on the location of the probes. This was related to the inhomogeneous distribution of impurities.

Nordlin, Leef, and Schelhorn[19] have measured electrical resistivity and Hall coefficient of Type IIb diamonds over a wide temperature range at the I.T.T. Federal Laboratories.

Austin and Wolfe,[20] Wedepohl,[21] and others refer to the use of a hydrogen-like model for a substitutional impurity atom for obtaining an approximation in which the activation energy is given by

$$E = \frac{2\pi^2 m^* e^4}{k^2 h^2}$$

where

m^* = effective mass of electron
e = charge on electron
k = dielectric constant 5.7
h = Planck's constant

The approximation leads to an ionization energy of 0.4 ev for a singly ionized impurity atom in diamond when m^* equals the free electron mass. This figure is close to those obtained experimentally, and encourages the suggestion that acceptor centers may consist of trivalent impurity atoms such as aluminum. However, as Wedepohl cautions, if some other value is taken for the effective mass the activation energy differs from that given above. As indicated in a summary by Mitchell,[22] the hydrogenic approximation appears to be extremely rough in view of more recent information on the complex nature of the valence bands (Kemmey,[23] Rauch[24]) in diamond.

3. EXPERIMENTAL PROCEDURE

Resistivity measurements were made on a semiconducting diamond sample measuring 2.24 x 1.01 x 0.97 mm by passing a d-c current through it and measuring voltage drops potentiometrically, as shown in Figures 1 and 2. Initially a Leeds and Northrup Type K-3 potentiometer and an Electro Instruments Digital Voltmeter were used for measuring voltages. In later runs, the voltages were recorded with Sargent and with Bristol recorders, as indicated in Figure 1.

In order to evaluate simultaneously the performance of the end contacts over the same temperature range, two sets of IR drops were observed. (See V_T and V_P in Figure 2.)

The readings at V_T include the drops across two contact resistances, as well as across the diamond's bulk resistance. The readings V_P at the probes, on the other hand, exclude the two end contact resistances. These can be used to calculate the resistivity of a homogeneous sample

$$\frac{V_P}{I} = R_P = \rho \frac{L_P}{A}$$

$$\rho = \frac{V_P A}{I L_P}$$

where

V_P = voltage drop between two probes (volts)
I = current through sample (amperes)
R_P = resistance of that section of sample located between probes (ohms)
ρ = resistivity of sample (ohm-centimeters)
L_P = spacing between probes (centimeters)
A = cross section area of sample (square centimeters)

If the sample is homogeneous, the resistance of the two end contacts can be obtained by subtracting the resistance of the sample (extrapolated from resistivity measurements at the probes) from the total resistance of sample plus contacts. Thus

$$R_C = R_T - R_S$$

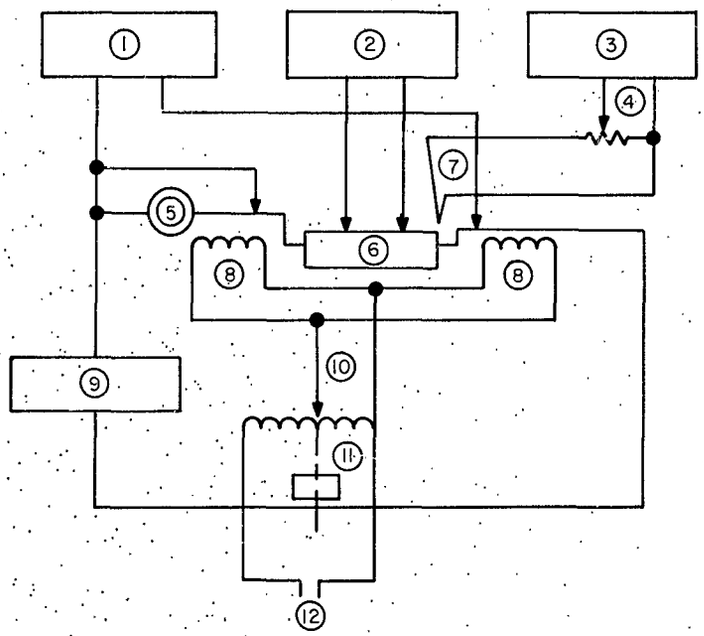

Figure 1. Schematic Diagram for resistivity Measurements

① Sargent Recorder-Model MR

② Sargent Recorder-Model MR

③ Bristol Recorder-Model 560

④ Helipot-Model A 20K

⑤ Sensitive Research Inst. Corp. Model S, D-c Milliammeter

⑥ Diamond Sample

⑦ Chromel-Alumel Thermocouple

⑧ Two 47-Watt Ungar Soldering Tips

⑨ Electronic Inst. Constant Current Generator-Model C62

⑩ Powerstat Type 116

⑪ 1 R. P. H. Timer

⑫ 115 Volt-60 Cycle Supply to Heaters

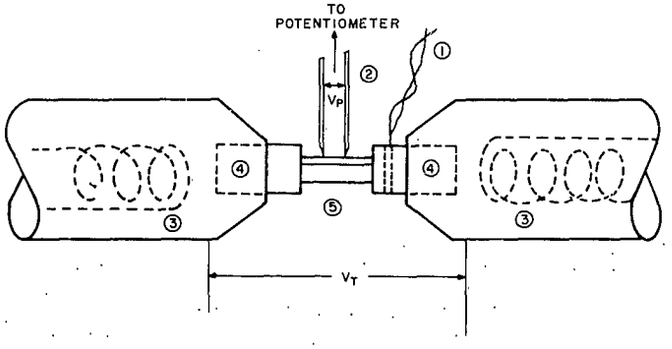

Figure 2. Assembly of Heaters and Electrical Contacts to Semiconducting Diamond

① Thermocouple Leads

② Knife-Edge Potentiometric Probes

③ Heaters

④ Graphite Contacts

⑤ Diamond Sample

in which

$$R_S = R_P \frac{L_S}{L_P} = \frac{V_P L_S}{I L_P}$$

where

R_C = combined resistance of two end contacts (ohms)

R_T = total resistance of sample plus contacts (ohms)

R_S = resistance of sample (ohms)

R_P = resistance of the section of sample located between probes (ohms)

V_P = voltage drop between two probes (volts)

I = current through sample (amperes)

L_P = spacing between probes (centimeters)

L_S = length of sample (centimeters)

3.1 Electrical Contacts to Samples

A variety of contacting techniques were used. These involved pressure contacts composed of metals and graphite (solid, powdered, and colloidal), as well as evaporated contacts. Alloyed or diffused contacts were not used, since it was important not to alter the properties of these rare samples.

3.1.1 SOFT METAL CONTACTS

One type of end contact studied consisted of a combination of soft metal and graphite (Figure 3), to which mechanical pressure was applied.

For the metallic part of these contacts, silver was selected because of its high thermal conductivity and its mechanical softness. It served as a good thermal conductor between the heat source and the diamond. Intimate thermal and electrical contact with the diamond was obtained by gently pressing the corners of the diamond into the silver, in the hollow conical sections, as shown in Figure 3. It was found that the addition of Aquadag in the hollow conical sections, lowered the contact resistance and provided relatively "stable," and reproducible contacts.

To prevent oxidation at the elevated temperatures, measurements were made within a dried inert atmosphere of helium, or argon. Temperatures were measured with a 0.005-inch diameter Chromel-Alumel thermocouple imbedded in the silver within a few thousandths of an inch of the diamond, and in good thermal contact with it. Temperature readings were taken with a Leeds and Northrup Type K-3 potentiometer.

To avoid the possibility of errors due to temperature differences between the diamond and its "surroundings," the current densities were kept small. To avoid errors due to photoelectric effects, all measurements were made with the sample in the dark.

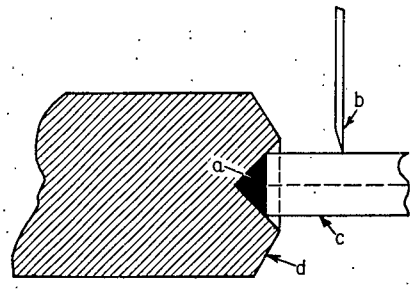

Figure 3. Detail of Silver and Aquadag Contact

a AQUADAG b PROBE c DIAMOND SAMPLE d SILVER CONTACT

Another type of contact, described below, was used in order to eliminate possibility of damage to the sample due to mechanical pressures, and to eliminate the formation of a conducting film on the sample at high temperatures when using the silver contacts.

3.1.2 GRAPHITE CONTACTS

The current-carrying contacts, shown in Figure 2, contain graphite blocks in contact with the ends of the sample. The graphite blocks, in turn, are supported by cylindrical iron heaters. Temperatures were measured with 0.005-inch diameter thermocouples imbedded in the graphite close to the diamond.

It appeared that fairly large area current-carrying contacts could be made between the soft graphite and the diamond by mechanically "working" the two materials together under light pressure. Contacts made in this manner appear to be fairly reproducible in performance. The resistance of the graphite to metal contacts, in series with the sample, was checked and found to be negligible.

The potentiometric contacts, or "probes," were made by using two honed knife edge blades of stainless steel, placed on an edge of the rectangular parallel piped sample, as shown in Figure 2. In order to determine precisely the spacing between these two probes, which were independently movable and spring loaded, a microscope and scale were used. The probe positions were checked under the microscope before and after each temperature cycle. A photograph of the experimental equipment is shown in Figure 4.

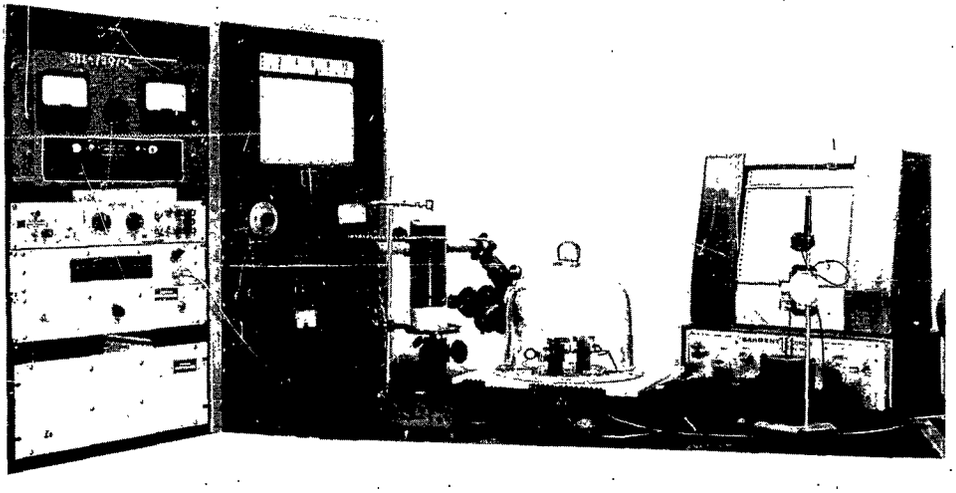

Figure 4. Photograph of Experimental Equipment

4. RESISTIVITY VERSUS TEMPERATURE

A curve of electrical resistivity as a function of temperature, assuming a homogeneous sample, is plotted in Figure 5.

Over the lower temperature ranges, the electrical resistivity is a sensitive function of temperature. At room temperature, for example, the resistivity changes about 3 percent per °C. At higher temperatures the resistivity becomes much less sensitive to changes in temperature. In the vicinities of 300°C and 450°C, for example, the resistivity changes approximately 0.2 percent per °C. A minimum in electrical resistivity occurs between 350 and 400°C.

In the lower temperature ranges, activation energies indicated by the slope of the curve are around 0.3 to 0.35 ev. If the curve were extended below room temperature, it should be more nearly linear and yield slightly larger activation energies.

As the temperature of the sample is raised through temperatures below the exhaustion region, the increasing current carrier density results in a decrease in electrical resistivity. As the temperature continues up through the exhaustion region, the increasing lattice scattering of the carriers causes an increase in resistivity.

There appeared to be no permanent change in the resistivity of the sample nor in its physical appearance, as a result of the controlled heat cycles in an inert atmosphere involved in these measurements.

The resistance of the diamond and the resistance of its current carrying contacts were measured as a function of temperature for current densities which were varied by two orders of magnitude, using the contacts shown in Figure 2. The extrapolated resistance of the sample (assuming homogeneous material), as well as the contact resistance, was a function of the current density. Changes in the current-carrying contact resistance may have been due to changes in the actual areas of conduction as a function of current density and applied electric field. Changes in bulk resistance in turn may have been due to distortion of electric fields within the sample because of small internal cracks and inhomogeneities.

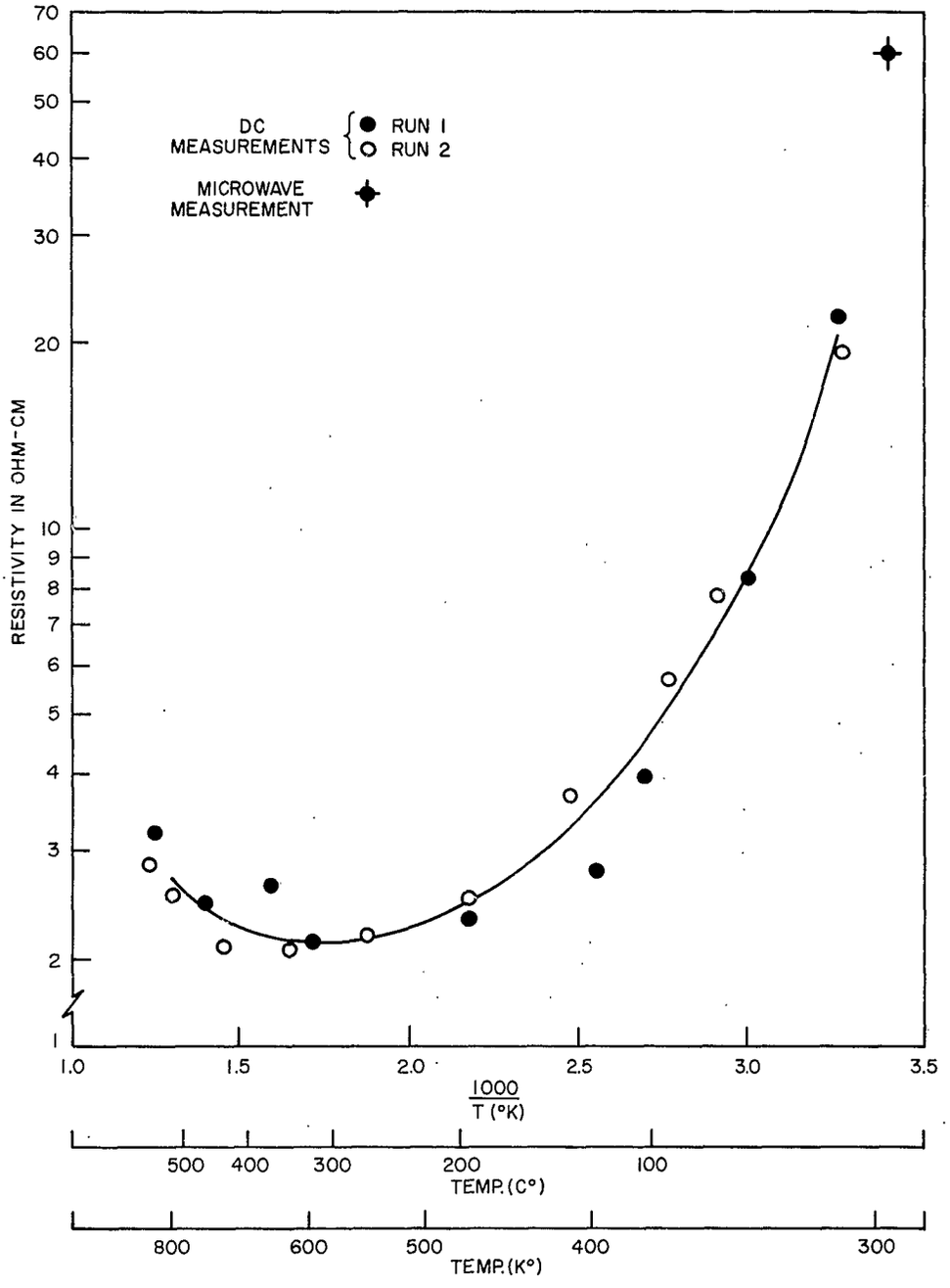

Figure 5. Resistivity vs. Temperature of Semiconducting Diamond, March 1962 (Sample 62-005) Microwave Measurement after Sirvetz and Oldfield, AFCRL-62-159

5. COMPARISON OF D-C AND MICROWAVE MEASUREMENTS OF RESISTIVITY

Techniques for applying electrical contacts become increasingly difficult as sample dimensions become very small or irregular in shape. When one considers future studies of properties of synthetic diamonds, he is confronted with problems involving samples which are very small and of irregular shapes. Measurement techniques which do not involve making electrical contacts to the samples would appear to have considerable merit for such applications. One such approach has been employed by Sirvetz and Oldfield[25] to measure electrical resistivity, by using microwave techniques with an x-band resonant cavity. Their microwave electrical resistivity measurement at room temperature is shown in Figure 4 in comparison with d-c measurements made at AFCRL from room temperature to higher ranges. The results thus far indicate close agreement between the two techniques.

6. SOME FACTORS INFLUENCING MEASUREMENTS

6.1 Surface Contamination

The presence of conducting layers of foreign material on the surface can introduce errors in the readings. Prior to measurement, the samples were carefully cleaned by chemical etching and then rinsed in acetone, alcohol, and distilled water. At elevated temperatures, however, with some contact materials a thin layer of foreign material coated the surface of the diamond. This occurred when silver and gold-plated copper contacts were used, and again with solid silver contacts. However when graphite contacts, backed by iron heaters were employed there was no evidence of deposited film at elevated temperatures on the diamond.

6.2 Inhomogeneity of Sample

There are some as yet undetermined factors which influence electrical resistivity. One of these is the inhomogeneity in impurity distribution and crystal structure of the sample. Some inhomogeneities are visible to the naked eye. Others can be detected by using a microscope, polarized light, X-ray diffraction, or electron diffraction.

Mutch and Raal[26] reported on a diamond sample that was extremely inhomogeneous. Their sample was clear at one end and blue at the other. The resistivity varied from 20,500 ohm-cm in the clear section down to 98 ohm-cm in the blue region.

Some diamonds contain "naats" or "knots," and a disordered kind of structure which is very difficult to saw or grind. These samples also present difficult problems involving shattering when cleaved. From microscopic observations, there appears to be evidence of this kind of structure in the sample studied. Schwuttke's[27] electron diffraction examination of several samples, including the sample measured here, revealed the presence of a large density of edge dislocation lines. Furthermore, these dislocations were distributed throughout the samples with a nonuniform density.

6.3 Space Charges

Space charges built up in the diamond may also be a factor to contend with in this type measurement. The effect of a strong incandescent light on resistivity and on the density of trapped charges was examined briefly. At room temperature the illumination caused a decrease in resistivity. After the sample was again isolated from the light source, the resistivity slowly drifted back to the original dark readings. However, at higher temperatures, the light had no measurable effect on resistivity.

Apparently excess current carriers were freed optically from traps at the lower temperatures, and thermally at the elevated temperatures. The question of nonhomogeneous distribution of space charges in this material, as well as correlation with the distribution of defects, warrants further investigation.

7. SUGGESTIONS FOR FURTHER STUDY

In order to examine inhomogeneity of diamond crystals, it is suggested that "profile" resistivity measurements be made. Such measurements may use either a single scanning probe, or a pair of closely spaced scanning probes.

Another area which might bear further investigation is that of resistance versus current density. In general, the resistance values of the sample and of the contacts were higher for smaller current densities. It may be possible to correlate this for several samples with gross defects within the crystals.

The resistivity values for increasing temperatures in general were lower than for decreasing temperatures. It seems doubtful that this was attributed to

dimensional changes with temperature, since probe spacings were checked with a microscope before and after each run. More work appears necessary, in order to determine whether this is a contact or a bulk hysteresis phenomenon. Supplemental studies of space charges in semiconducting diamond using controlled illumination may help clarify this question.

8. CONCLUSIONS

At temperatures below approximately 200 to 250°C, the electrical resistivity of a sample of semiconducting diamond is a very sensitive function of temperature. At room temperature, the electrical resistivity changes by approximately 3 percent per degree centigrade. At elevated temperatures, the resistivity is more nearly constant, with a minimum occuring between 350 and 400°C. There appears to be no permanent change in the electrical resistivity of the semiconducting diamond sample as a result of the temperature cycles in an inert atmosphere.

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Unclassified report

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2. Semiconductor material
3. Resistivity measurement

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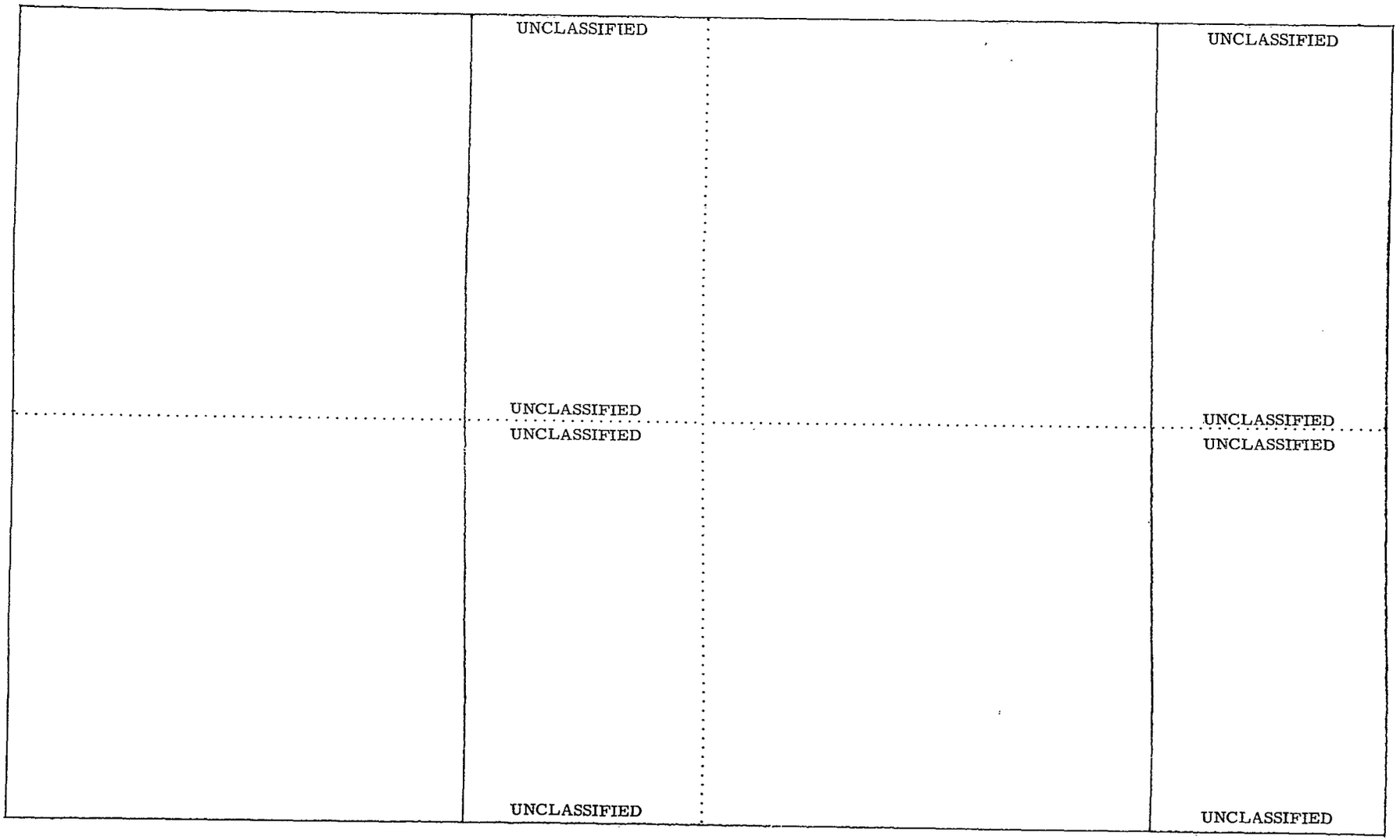
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