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AN EVALUATION OF CADMIUM SULFIDE AS A NUCLEAR RADIATION DETECTOR

THESIS

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AN EVALUATION OF CADMIUM SULFIDE AS A NUCLEAR RADIATION DETECTOR

THESIS

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By
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Preface

This work was performed to determine if CdS would make a superior nuclear radiation detection material. Although I found CdS did have definite limitations, certain parameters, such as the energy required to create a hole-election pairs and the mobility-lifetime product of the carriers in CdS were determined.

The facilities I used for carrying out this work were those of the Aeronautical Research Lab. Also the CdS crystals which I tested were those grown by the Solid State Branch of ARL. Without their support this work could not have been carried out.

I wish to express my appreciation to Dr. William Lehmann, Dr. William Price, and Don Reynolds for their cooperation and guidance in this work. I would also like to thank my wife for typing the rough drafts and Judy Rowell for typing the final draft.

Darrel L. Mills

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Abstract

Solid state radiation detectors were constructed using crystal platelets of CdS. Both intrinsic and p-n junction detectors were made and evaluated. Although alpha particles were detected by both types of detectors, the mobility-lifetime product of the charge carriers resulted in the pulse response not being proportional to the energy of the incident particle. The best experimental value for the mobility-lifetime product in CdS was 2.6×10^{-6} cm^2/volt for the electrons and 2.1×10^{-6} cm^2/volt for the holes. Also an experimental value of 5.2 electron volts dissipated per hole-electron pair formed was determined. A resolution of 6.8% was obtained with one detector. Other phenomena such as the trapping of the charge carriers and the ionization of neutral impurity atoms also were evident in the crystal.

I. Introduction

This work was carried out in an effort to find a superior material for use in solid state radiation detectors. The silicon crystal has thus far exhibited the best characteristics for use in solid state detectors, but it has one important limitation. This limitation is that it does not, at present, effectively detect lightly ionizing radiation, such as, gamma rays. The reason for this is that the silicon crystal can not be fabricated large enough to stop gamma rays and still retain the crystals desirable characteristics.

A logical approach to solve the problem of detecting lightly ionizing radiation is to choose a material with a greater stopping power (higher Z material) than silicon, and still have all of the desirable characteristics of silicon. The study of CdS, which has twice the atomic weight of silicon, was an attempt to find such a material.

CdS has not been seriously considered before because earlier studies indicated that the holes in CdS were virtually immobile. However, these studies were incomplete. Since higher purity CdS crystals have recently been fabricated by the Aeronautical Research Lab. and therefore were readily available, it was decided to proceed with this investigation.

As a preliminary study, silicon solid state detectors were constructed and tested. These crystals did detect alpha

particles but excess leakage current was present which affected the performing of the detector.

Solid state detectors were then constructed using platelets of CdS. Both intrinsic and p-n junction were made. Although alpha particles were detected by both types of detectors, the mobility-lifetime product of the charge carriers resulted in the pulse response not being proportional to the energy of the incident particle. The best experimental value for the mobility-lifetime product in CdS was 2.6×10^{-6} cm^2/volt for the electrons and 2.1×10^{-6} cm^2/volt for the holes. Also an experimental value of 5.2 electron volts dissipated per hole-electron pair formed was determined. Other phenomena such as the trapping of the charge carriers and the ionization of neutral impurity atoms also were evident in the crystal.

The balance of the thesis will contain a chapter each on the following: (1) the theory of solid state detectors, (2) a description of the experimental techniques, (3) a summary of the results, (4) an interpretation of these results, and (5) conclusions.

II. Theory

The solid state detector is similar to the ionization chamber in its principle of operation. In an ionization chamber a voltage potential is applied across a layer of gas so that any charge created in the gas by an ionizing particle is collected at the terminals. In a solid state detector, a potential is applied across a semiconductor. When an ionizing particle enters the semiconductor, and creates hole-election pairs a pulse of charge proportional to the energy of the incident particle is collected at the terminals.

There are, however, necessary conditions for this proportionality to exist. These conditions are (1) the incident particle dissipates essentially all of its energy in the sensitive region of the semiconductor, (2) the mobility-lifetime product of the charge carriers is sufficiently large so that all of the created carriers are collected, (3) the semiconductor crystal is sufficiently pure so that large numbers of trapped ionized charges do not exist in the crystal which would disrupt the uniformity of the electric field, and (4) the crystal will not break down or the leakage current will not be excessive when the collecting bias is applied. Each of these conditions will be discussed in detail later.

The energy necessary to form a hole-electron pair in a semiconductor is much less than the energy necessary to form an ion-pair in a gas. For comparison it requires 30-35 ev./i.p. for most gases and approximately 3 ev/hole-electron pair for a semiconductor.

The band gap of a semiconductor is normally much less than the energy required to form a hole-electron pair. According to Shockley (Ref 2:14), the difference between the band gap energy and the mean energy for forming a pair, takes the form of kinetic energy which is shared by each carrier. These carriers then lose this energy by phonon emission. The mean energies per pair appear to be proportional to the band gap energy. For silicon and germanium the mean energy per pair is about three times the band gap. Czaza (Ref 2:13) has shown that this proportionality is plausible according to Shockley's theory, and that it should extend to other class IV semiconductors. With the exception of heavy fission fragments, the mean energy per pair is constant. For heavy fission fragments it only deviates about 10%

After the charge has been collected at the terminals of the semiconductor, it is fed into a charge sensitive pre-amp. The voltage pulse "V" that results is given by

$$V = \frac{Q_c}{C_a + C_d} \qquad (1)$$

where Q_c = charge collected

C_a = input capacitance of the pre-amp

C_d = the depletion layer capacitance of the detector.

The depletion layer capacitance of the detector is given by

$$C_d = \epsilon A / 4\pi D \qquad (2)$$

where A = cross sectional area of the crystal

ϵ = dielectric constant

D = thickness of the depletion region.

Intrinsic Detector

The simplest solid state detector design is the intrinsic detector. This detector is similar to a parallel plate capacitor in that the semiconductor replaces the usual insulator and the plates serve as ohmic contacts to the crystal. The conducting "plates" are necessary so that a uniform electric field exists in the crystal.

The intrinsic detector design has definite limitations because almost all semiconductors contain excessive free charge carriers which are due to the donor and acceptor impurity atoms in the crystal. These impurities result in a low resistivity material. Therefore, when a bias is applied, the free carriers are collected along with the charges produced from the ionizing particle. Obviously, only a very small number of these carriers, compared to the carriers liberated by the ionizing particle, can be tolerated in an intrinsic detector or the resolution of the pulse will be damaged considerably.

Some semiconductors have recently been made with resistivities of 10^8 ohm-cm and higher which can serve as intrinsic detectors (Ref 2:5). This high resistivity has been achieved primarily by introducing impurities that compensate for the acceptor and donor impurities. These added impurities act as trapping centers however, and greatly reduce the lifetime of the charge carriers. Unless the life-time of both carriers is significantly greater than the time for collection, the proportionality between the energy of the particle and the charge collected is lost. Also the mobility is

normally lowered by a factor of two by the impurities.

An added complication of impurities results when preferential trapping of one of the carriers occurs. With one carrier collected and the other trapped, polarization results which greatly disturbs the field distribution. This build up of a net charge can cause the charge of the opposite sign to be injected to neutralize the system. This current, called secondary current, adds to the ionization current and again proportionality between energy dissipated and charge collected is lost. The secondary current normally takes the form of a pulse of a much slower rise time and duration than the ionization pulses (Ref 8:1).

The product $\mu\tau$ where μ is the mobility, τ is the recombination lifetime, and E is the electric field is a very important parameter for determining the usefulness of a particular semiconductor. This product has the dimensions of length and is a measure of the distance traveled by a carrier. Miller and Gibson have developed a method to determine the $\mu\tau$ of the carriers by measuring the charge collected with respect to the charge liberated by the ionizing radiation (Ref 9:1-7). This method is derived as follows:

The charge collected is related to the total charge liberated by

$$Q_c = Q_L \frac{\Delta V}{V(o)} \qquad (3)$$

where Q_c = charge collected

Q_L = charge liberated

$V(o)$ = potential difference across the crystal

V = potential difference across which the carrier actually traveled.

The charge liberated Q_L depends upon the range of the radiation R and the ionization density N (y).

$$Q_L = q \int_0^R N(y)\, dy \tag{4}$$

where q = electronic charge

$N(y)dy$ = number of charges created between y and $y + dy$

R = range of nuclear particle.

N(y) and R may be determined from standard nuclear radiation detection textbooks, such as Price (Ref 11:2-12). However, for this development the range of the nuclear particle will be assumed small compared to thickness of the crystal.

An expression for the difference in potential ΔV is

$$\Delta V = \int_y^d E\, dx = \int_y^d e^{-\int_y^x \frac{dx}{\mu\tau E}} E\, dx \tag{5}$$

where y = point where the carrier was created

x = point where the carrier terminated

d = position of collecting terminal.

Since $\mu\tau E$ has the dimensions of length, the $e^{-\int_y^x \frac{dx}{\mu\tau E}}$ is a probability factor.

When equations (4) and (5) are combined with equation (3), the following equation for the charge collected results:

$$Q_c = \frac{q}{V(0)} \int_0^R N(y) \int_y^d e^{-\int_y^x \frac{dx}{\mu\tau E}} E \, dx \, dy \qquad (6)$$

If it is assumed that the range of the ionizing particle is very short compared to the thickness of the crystal, i.e., all of the charge is created at the surface, then y = 0.
Intergration yields

$$Q_c/Q_L = \frac{\mu\tau E}{W}\left[1 - e^{-\frac{W}{\mu\tau E}}\right] \qquad (7)$$

where W = thickness of the crystal.

Thus, if Q_c/Q_L is experimentally determined at different electric fields, then the product $\mu\tau$ can be calculated. Also if hole-electron recombination is the only phenomena affecting the charge collection, then the product $\mu\tau$ will remain independent of E.

Junction Detectors

The junction detector makes use of a p-n junction to solve the problem of free carriers in the conduction band. One type of junction, called the diffused junction, is made by diffusing donor atoms into one surface of high resistivity p-type material (Ref 4). The diffusion is very shallow (approximately one micron) and the result is a thin layer of material with a high concentration of acceptor atoms.

When a reverse bias is applied to a p-n junction a charge-free or "depletion" region is established on both sides of the junction. An illustration of this charge free region and example of the circuit necessary to produce this region is shown in Fig. 1.

The thickness "D" for a constant charge density on either side of the junction is approximately (Ref 7:388)

$$D = \left(\frac{\epsilon V}{2\pi q N}\right)^{1/2} \tag{8}$$

where ϵ = dielectric constant

V = potential difference across the region

q = electronic charge

N = concentration of ionized impurity atoms per unit volume.

The ratio of the thicknesses is given approximately by

$$D_p/D_n = \left(\sigma_n/\sigma_p\right)^{1/2} \tag{9}$$

where σ = conductivity, and subscripts p or n applies to the p or n region.

Therefore, the thickness of the charge-free area in the base material will be very much thicker (typically 1000 times) than the thickness in the diffused layer. This makes possible a very thin "dead layer" that the nuclear particle must penetrate before it reaches the sensitive region. If all of the hole-electron pairs created in the depletion region are collected, then the pulse is proportional to the energy dissipated. However, if hole-electron

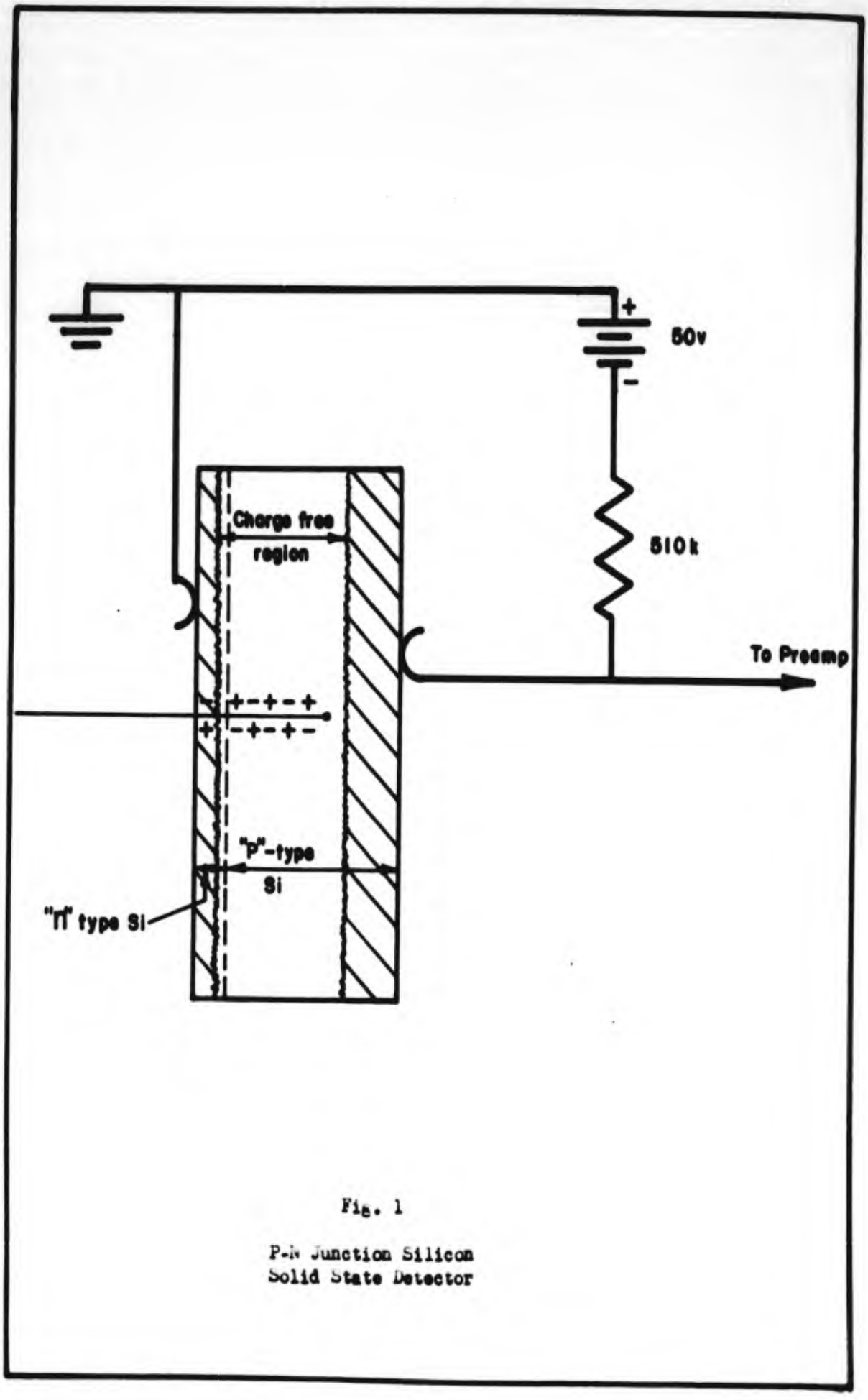

Fig. 1

P-N Junction Silicon
Solid State Detector

pairs are formed outside the depletion region they will diffuse around until they recombine or enter the depletion region. For this reason, it is essential that the depletion region be thicker than the range of the nuclear particle.

From Eq (8) it can be seen that

$$D \simeq \left(V/N\right)^{1/2} \tag{10}$$

Consequently, the higher the voltage bias and resistivity of the material, the thicker the depletion region. The resistivity is limited by fabrication techniques while the voltage bias is limited by the breakdown potential or leakage current through the material.

For a uniform distribution of ionized impurities, the electric field is linear with a maximum value at the junction. From Eq (8) it follows that the maximum electric field is given by

$$E_{max} = \left(\frac{8\pi eNV}{\epsilon}\right)^{1/2} \tag{11}$$

Another useful method to construct a p-n junction detector is to form a surface barrier junction instead of the diffused junction. The two types of junctions are similar except that the junction is formed by surface treatment of the crystal. Surface barrier junctions have the advantage of being very thin, about 1/10 as thick as the diffused junction. They have the disadvantage of being moderately sensitive to certain ambients such as hydrocarbon vapors. The equations developed above for the diffused junction detector are also applicable to the surface detector except for Eq (9).

One problem that is of concern for both junction and intrinsic detectors is surface leakage current. In many cases surface leakage can be the major contribution to the total leakage current. This leakage cam result from the collection of ionic contaminents on the surface, or from ambient conditions that produce inversion layers.

III. Experimental Techniques

The experimental work was divided into two parts. The first step was to become familiar with the present state of the art of solid state detectors. To do this, some silicon detectors were made using standard techniques. The second part was to study cadmium sulfide as a possible detector material. Operating detectors were obtained in each case.

The following section describes the equipment used to test the detectors. The next two sections describe the construction techniques for making the silicon and cadmium sulfide detectors.

Testing Equipment

A block diagram of the equipment used in testing the detectors is shown in Fig. 2. The method of operation for this equipment is as follows: An alpha source irradiates the solid state detector thereby producing hole-electron pairs in the crystal. Since the range of an alpha particle is very short in air, the detector and alpha source are placed in a vacuum chamber. A Pu^{239} alpha source (5.147 Mev) was used when the silicon detectors were tested. Later an Am^{241} alpha source (5.476 Mev) was obtained when the CdS detectors were tested. A voltage bias is applied across the detector so that the charges are collected. (See Fig. 3 for circuit diagram for applying the bias.) The current pulse is fed into a charge sensitive pre-amplifier and then into a linear amplifier. From

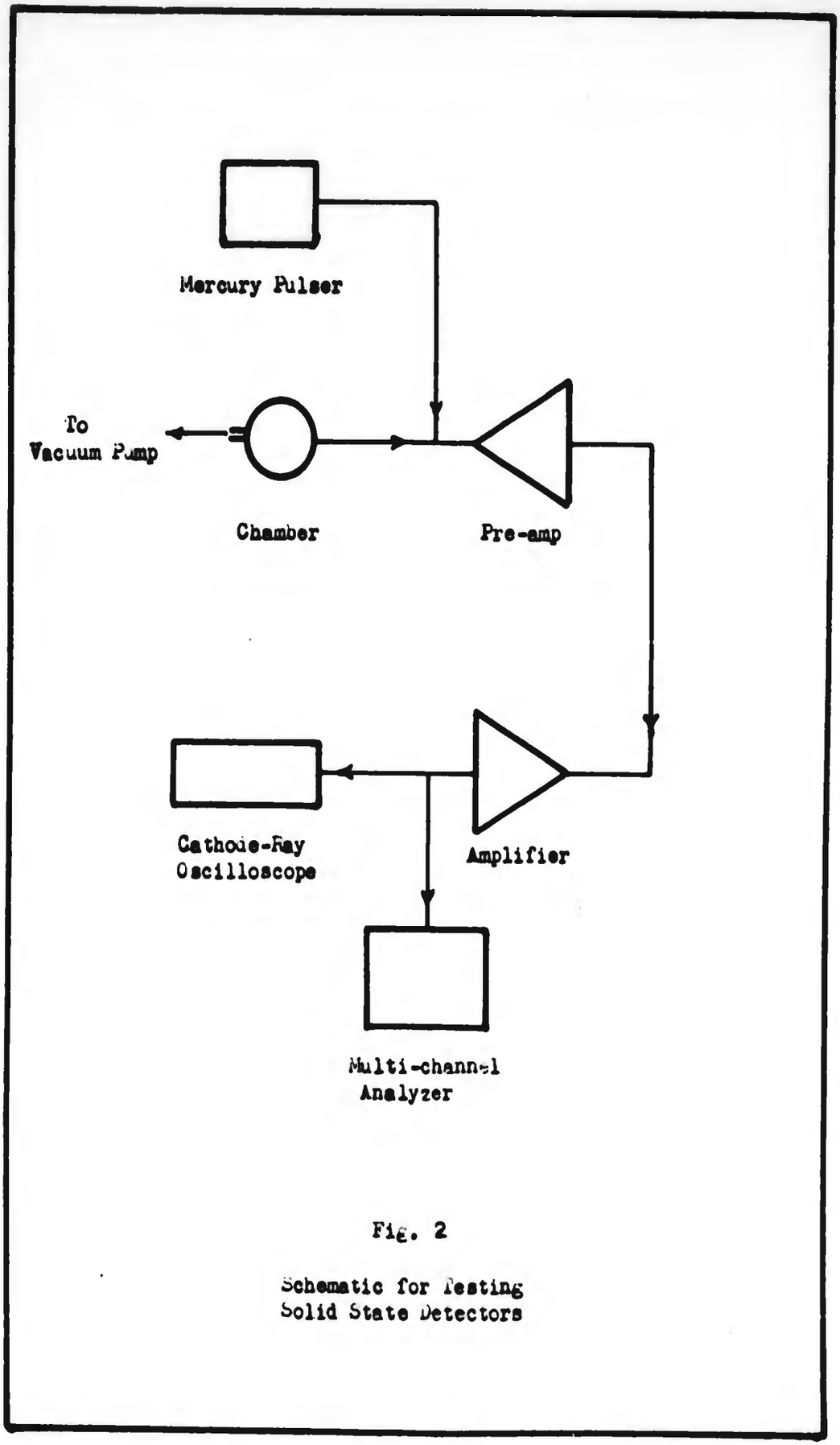

Fig. 2

Schematic for Testing
Solid State Detectors

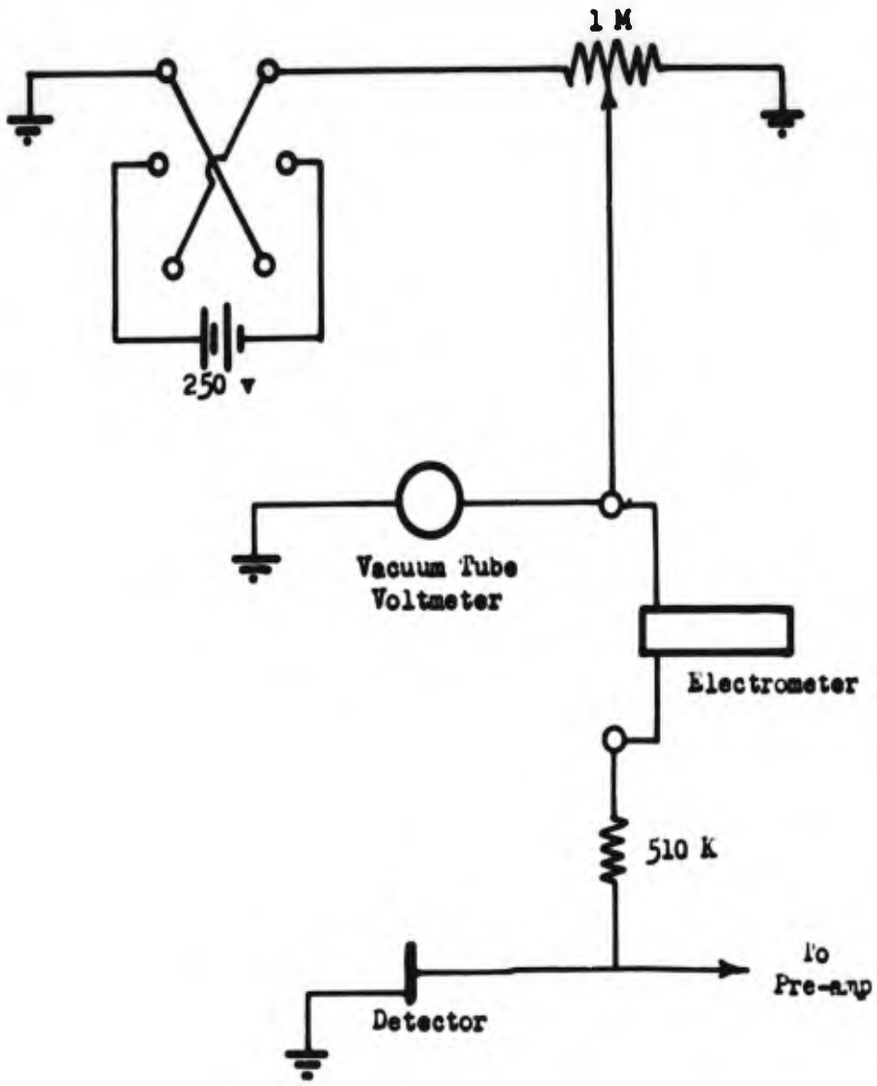

Fig. 3

Circuit Diagram for
Applying Voltage Bias to Detector

the linear amplifier the pulse can be fed into a cathode ray oscilloscope to view the pulse, or into a multichannel analyzer to determine the pulse height resolution.

All of the pulse height measurements were made by matching the pulse height from the calibrated mercury pulser with the pulse height from the detector. The voltage pulse from the pulser was fed into a 5 pf. capacitor to simulate the charge from the detector. Thus a 10 mv pulse from the pulser was equivalent to 50×10^{-15} coulomb of charge from the detector by $q = CV$. In this manner all of the pulse height readings from the detectors were measured in terms of millivolts.

The method of measuring the pulse height was checked as follows:

Pulses were obtained from an ORTEC detector using the Pu^{239} and Am^{241} sources. The pulse heights were the same height as a 48.2 and 53.0 mv pulse from the pulser. Assuming ϵ for silicon is 3.3 ev/pair (Ref 2:13), then the charge created from the 5.147 Mev alpha should be equivalent to a 49.5 mv pulse height from the pulser. Similarly, the charge from the 5.476 Mev alpha should be equivalent to a 53.1 mv pulse height from the pulser. Thus it can be seen that the above values compare favorably, and the method of measuring the pulse heights is within reasonable error.

A description of each component is as follows:

The chamber was a small brass pot with a diameter of about 5 in. and a height of about 4 in. Inside the pot were small holders for the alpha source and detector. Also a small 6 watt light was inserted

which could be turned on and off from outside the chamber. There was a cold trap installed between the chamber and the vacuum pump so that hydrocarbons from the pump would not pass into the chamber.

The pump was a Welsh Duo Seal Vacuum Pump.

The pre-amplifier was one designed by Chase, Higenbotham, and Miller of Brookhaven National Laboratory (Ref 1) and was devised especially for use with solid state radiation detectors. The pre-amp was charge sensitive and employed negative charge feedback so that the output amplitude was independent of the value of the detector capacitance. It also had a high open loop gain, a fast response, and little tendency to oscillate.

The linear amplifier was a Baird-Atomic Model No. 215. It was modified so that it would take either a positive or negative pulse input. It had an RC clip of 1.6 micro-seconds.

The cathode ray oscilloscope used as a Tektronix Type 531.

The multi-channel pulse height analyzer was a 256 channel, Radiation Instrument Development Laboratory, Model 3301, analyzer.

The mercury pulser was manufactured by Radiation Instrument Development Lab., Mod M-60.

The internal circuitry for applying the bias to the detector is shown in Fig. 5. A Keithley, model 210, electrometer was used to measure the current that passed through the detector. The voltage drop across the 510 K resistor and the electrometer was subtracted from the voltmeter reading to determine the potential on the detector.

on the detector.

The input capacitance of the pre-amp and the detector capacitance can be determined experimentally by the following method: A known voltage pulse V_p which is fed into a capacitor C_p will result in charge q by $q = C_p V_p$. If this charge is fed into a charge-sensitive pre-amp, the output of the pre-amp

$$V_o = \frac{G q}{C_i + C_x} = \frac{G V_p C_p}{C_i + C_x} \qquad (12)$$

where C_i = input capacitance of the pre-amp

C_x = external capacitance added in parallel

G = gain of the pre-amp.

The constants $GV_p C_p$ can be combined into 1/k so that

$$1/V_o = k(C_i + C_x) \qquad (13)$$

A plot of $1/V_o$ vs. $(C_i + C_x)$ can be made by varying the known capacitance C_x. The slope of the curve, k, at $C_x = 0$ is then used to determine the input capacitance C_i. With C_i and k known, the capacitance of an intrinsic detector can then be determined by inserting it in parallel with C_i.

The testing equipment was checked by testing some commercial detectors. Two RCA diffused junction detectors and three ORTEC surface barrier detectors were tested. Parameters such as pulse height vs. voltage bias, resolution, and leakage current were studied.

Construction of Silicon Detector

Diffused Junction Detectors. Three small p-type silicon crystals in which a p-n junction had been formed by diffusing phosphorus into one face, were investigated. The crystals were about 1/8 x 1/4 x 1/16 in.

The crystals were cleaned, etched, and rinsed according to the procedure recommended in the Harwell Report (Ref 3:2). However, they were etched for only 1 1/2 minutes rather than the 5-10 minutes Harwell recommended to insure that the junction was not etched off. Immediately after rinsing and before any oxidation could take place, a small gold dot about 80 $\mu g/cm^2$ thick was evaporated on the junction side of the crystal. An electrical contact was made by gluing a small copper wire to the gold dot using silver print (General Cement No. 212). The print was not allowed to run off the dot since this might harm the junction. On the back side silver print was spread over all the surface so that a uniform electric field could be formed across the crystal.

To avoid contamination, the crystals were mounted using a method suggested in the Harwell Report. The detectors were tested to determine the leakage current, diode action, and pulse height vs. bias relationships.

Surface Barrier Detectors. A surface barrier detector was made using a piece of silicon dendrite web obtained from the Electron Technology Lab. of the Aeronautical System Div., AFSC. The web was 94 ohm-cm

n-type silicon, and was about 3 in. long and ½ in. wide. The web was cut into 3/16 in. strips by either a hypersonic abrasive tool or a diamond saw. The method by which the crystals were cut appeared to have no effect on the performance of the crystal.

The Harwell Report recipe (Ref 3:2-4) was followed in the preparation of the surface barrier detector. The crystal was then mounted and tested in the same manner as the diffused junction detector.

In an attempt to reduce leakage current the crystals were potted in epoxy resin (Bonding Agent R-313, made by Carl H. Briggs Co.), so that just the face of the crystal was exposed. The mounting of this detector was patterned after the construction of the ORTEC surface barrier detector.

Construction of Cadmium Sulfide Detector

The Solid State Physics Branch of the Aeronautical Research Lab. has recently developed a method for growing high purity CdS crystals (Ref 13). This process, called the vapor phase process, is one in which crystals are grown from the vapors of CdS powder as it is distilled. The crystals take the form of thin platelets or whiskers. A few of the platelets grow relatively large, i.e., about 1/2 cm^2 area by 200 microns thick, and therefore are in a convenient shape to make detectors. A higher purity crystal can be obtained by a second distillation of the crystals.

Intrinsic CdS Detectors. The first detectors made with CdS were intrinsic detectors. These were made in the following manner: The platelet was etched by holding it in Hcl acid vapor for about ten seconds and then rinsing it in tap water. After it had dried on a clean tissue, indium (which makes an ohmic contact with CdS) was soldered on to one face of the crystal with an ultra-sonic soldering iron. The most convenient method to do this, since the crystals are very fragile and easily broken, was to lay the crystal of the blunt surface of the soldering tip (which had solder already on it), and then pull the crystal off with a pair of tweezers. Normally the solder adhered to the back surface and did not overlap on to the front. The back electrical contact was made by taking a small pre-tinned copper wire and sticking it into the molten solder as the crystal was pulled from the soldering tip. After the solder hardened it served to support the fragile crystal.

Indium, about 50 μ/cm^2 thick, was evaporated on to the front of the crystal. The border of the crystal was masked during the evaporation so that the crystal would not be short circuited. The border was normally about five times the thickness of the crystal. A copper lead was then either glued on with a small drop of silver print, or soldered on with indium solder. The soldered lead proved to be superior because it was much less likely to come off, especially through temperature cycling.

The crystals were held in the testing position by the terminal leads. Tests to determine resistivity, pulse height vs. voltage bias, and effects of temperature and light were made.

Junction CdS Detectors. The junction detector was constructed similar to the intrinsic detector except that a p-n junction was formed on the front face, rather than the ohmic indium contact. An attempt to produce a p-n junction on CdS was made using the following electroplating solution to deposit the cuprous ion on to the surface (Ref 5)

80 ml - HNO_3

320 ml - H_2O

2 g - $Cu(NO_3)_2$

5 ml - polyethylene glycol 600.

However, the resistance of the platelets was too high to pass the necessary current so the following method was used to implant the cuprous ion on to the crystal surface: The cuprous ion was electroplated on to a copper wire and the plated substance scraped off and smeared on the face of the CdS crystal. The crystal was then warmed gently until bubbling of the electroplated substance occurred. Evidently some chemical reaction was taking place, since it would start at one point and spread throughout the substance. After the bubbling had stopped, the crystal was placed on a hot plate that was pre-heated to 300° C. The crystal was allowed to heat until the substance turned black (approximately ten seconds). After cooling, the excess charred substance was wiped off gently with damp tissue paper.

If the original solution was fresh, the resulting residue on the surface was sufficiently thick so that good conduction across the crystal face occurred. The front electrical connection was made with a small copper wire glued with silver print.

This crystal was then tested in the same manner as the previous crystals.

IV. Summary of Results

A summary of the results obtained is presented in this chapter. The interpretation of these results will be presented in the next chapter.

Silicon Detectors

Three diffused and four surface barrier silicon detectors were constructed and tested. Upon irradiating the detectors with a Pu^{239} alpha source, all of them produced a signal except one diffused detector. Table I shows the results of the tests made on these detectors.

The largest pulse height possible with a silicon crystal using a 5.147 Mev, Pu^{239} alpha source, can be calculated to be 49.5 mv. The pulse heights obtained with the experimental detectors were much less, because the bias could not be increased beyond 6 volts without excessive noise. Therefore the depletion layer thickness was less than the range of the alpha. Fig. 4 shows the voltage-current relationship for surface barrier detector No. 2, which is typical of the other detectors. A high leakage current is evident at low bias.

With the surface barrier detector an effort was made to reduce the surface leakage by potting the crystal immediately after the rinsing was complete. However, during the potting some discoloration of the surface was noted. This was believed to be due to epoxy vapors. Since none of the six detectors in which potting was attempted would detect, these vapors evidently affected the surface so that the

Table I

Results Obtained with Silicon Detectors

	Max. Pulse Height (mv)	Pulse Height Resolution % *
Diffused Detector #1	19.0	18.0 %
Diffused Detector #2	15.0	37.0 %
Diffused Detector #3	No signal	
Surface Barrier Detector #1	31.0	14.0 %
Surface Barrier Detector #2	26.0	50.0 %
Surface Barrier Detector #3	20.0	18.8 %
Surface Barrier Detector #4	28.0	17.3 %

* Defined as full width at ½ max.

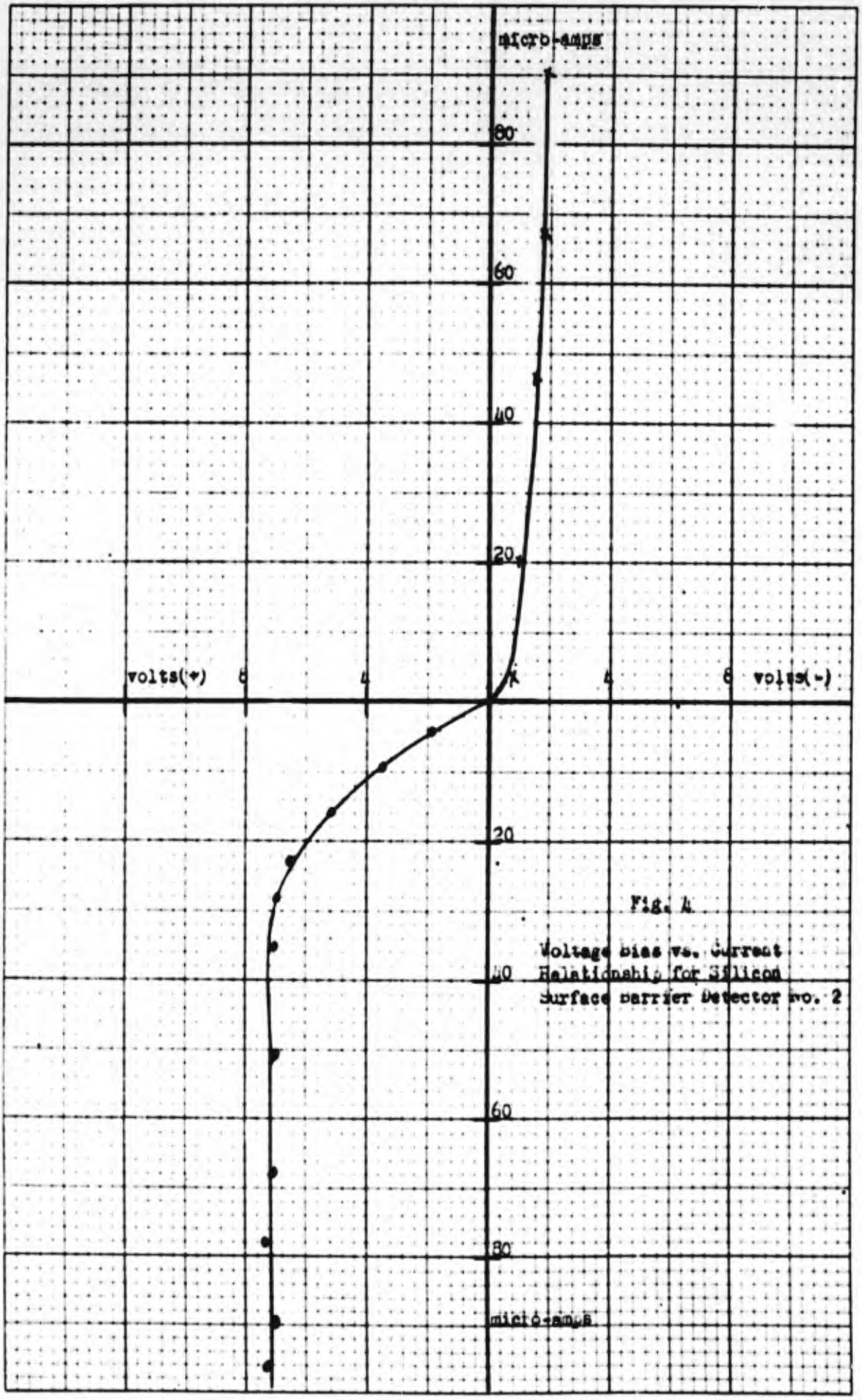

Fig. 4

Voltage bias vs. Current Relationship for Silicon Surface Barrier Detector No. 2

barrier would not form. However, when the surface of two of the detectors were reprocessed after the epoxy had hardened, some intermittent pulses were observed.

Cadmium Sulfide Detectors

After the techniques for constructing the CdS detectors were developed, the procedure was quite straight forward. The most difficult problem in the construction was to insure the platelets were not broken.

Evidently CdS does not form the surface states as readily as silicon because although the CdS crystals were rinsed with tap water, a voltage bias of 60 volts was reached before the leakage current excessed 1 μa on most of the crystals. Assuming that the leakage current is negligible, the following table gives some characteristic resistivities for some intrinsic detectors. These values were obtained using a 20 volt bias.

Crystal	Thickness (Microns)	Resistivity (ohm-cm)
C-3	100	2.1×10^{10}
RS-1	60	6.1×10^{9}
M-1	460	1.6×10^{8}

Harshaw reported 6.2×10^{8} ohm-cm resistivity for the CdS vapor phase crystal (Ref 6:14). Normally, the thicker the crystal grows, the higher the impurity concentration. Crystals C-3 and M-1 were produced by one distillation, whereas RS-1 was from a second distillation.

RS-1 was the only second distillation crystal that was tested since these crystals normally don't grow large enough to be used for detectors.

As noted earlier in the Experimental Techniques, all of the pulse height measurements were made in terms of millivolts. Therefore, with pulse heights from the CdS detector given in mv, the charge collected can be calculated in each instance by $q = CV$.

The input capacitance to the pre-amp was determined experimentally by the method described in the Experimental Techniques to be 1500 pf. Because the input capacitance was so large it was difficult to measure the small capacitance of the detectors, but they were determined to be less than 200 pf.

The data obtained with each CdS detector is presented in the following sections.

Crystal No. C-3. The first intrinsic crystal that successfully detected alpha particles was C-3. Its dimensions were 100 microns thick and almost a cm square, which is the largest, most uniform crystal that was obtained. The crystal detected when either positive or negative bias was applied. The front terminal where the radiation was incident was always grounded and the bias was applied to the rear terminal. The pulse height varied with voltage (both + and -) as did all the CdS detectors tested. A plot of the voltage bias versus pulse height for C-3 is shown in Fig. 5.

At a constant voltage bias, the pulse height decreased with time. For this reason the values of pulse height given in Fig. 5

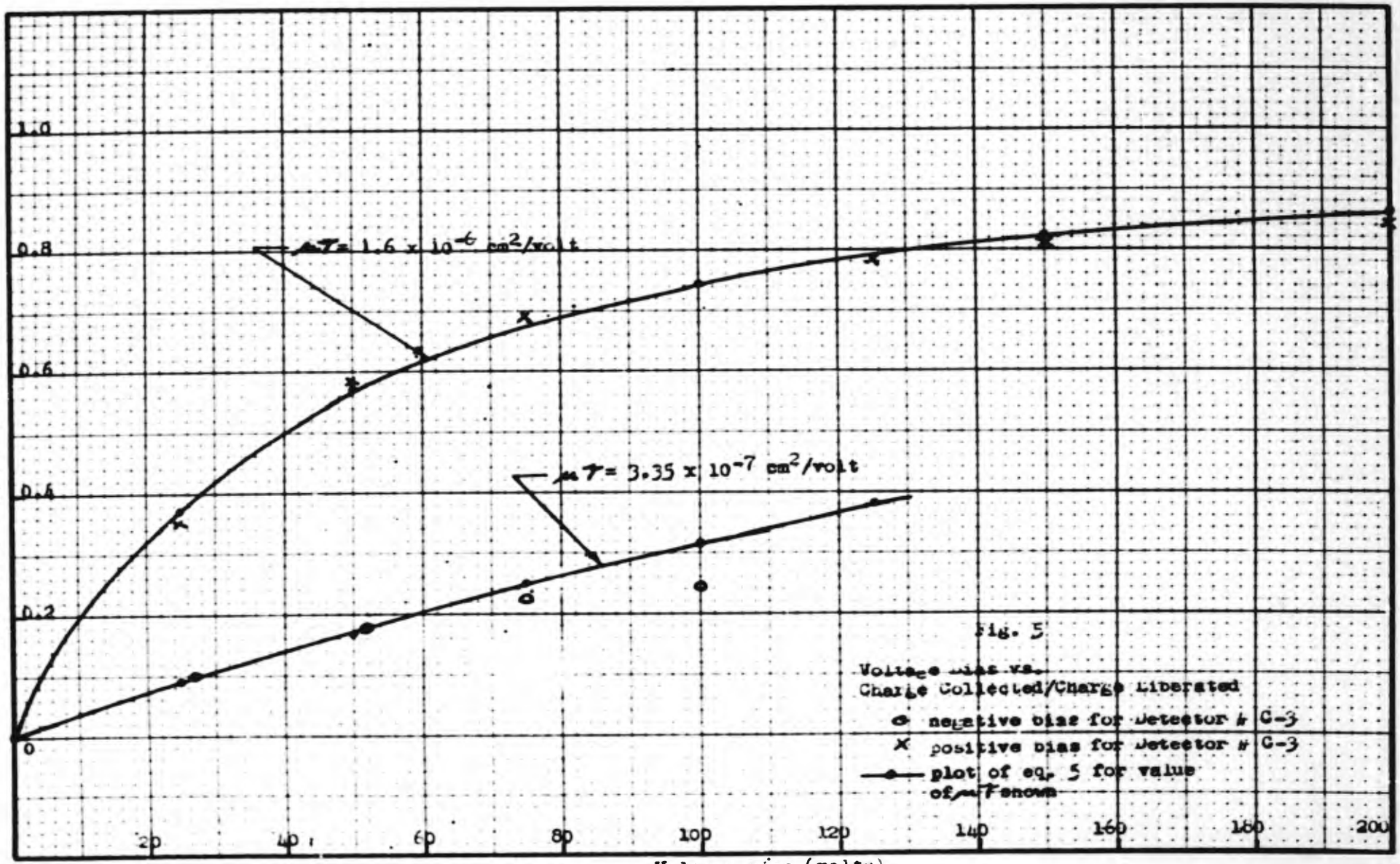

Fig. 5

Voltage bias vs.
Charge Collected/Charge Liberated

o negative bias for detector # C-3

x positive bias for detector # C-3

—●— plot of eq. 5 for value of $\mu\tau$ shown

were those initially observed. The following are representive data: (1) Positive bias. With 50 volts bias the pulse height was initially 19.3 mv but in two minutes it decreased to about 8 mv. Eventually the pulse fell below the noise level (1 mv). (2) Negative bias. A like phenomena occurred with negative bias, except the relative pulse height decrease was slower. Beginning with a pulse height of 5.6 mv at 50 volts bias, the pulse height decreased to 2 mv in ten minutes and below the noise level after about 5 more minutes. (3) Zero bias. The voltage bias was turned off at this point and the input to the amplifier switched so that it would accept a negative pulse (the pulse normally seen with a positive bias). A 15 mv pulse was seen. This pulse slowly decreased with time so that in about 2 minutes it disappeared. If the polarity was reversed after the pulse height had decayed below the noise level, a pulse height greater than the ordinary value was observed initially. It then decayed in time as did all other pulses.

If a part of the face of the crystal was masked so that only a portion of the crystal was irradiated, the pulse height would decrease with time until it was below the noise level. But, when the mask was removed, a pulse presumably from counting in the previously masked region could be seen.

The rate at which the original pulse attenuated could be increased markly by rising the temperature of the crystal. For example, at 60° C when a negative bias of 50 volts was applied, a pulse height then decreased rapidly and was at one-half its original value in 10 seconds.

A red light was placed in the chamber so that it could be turned off and on from outside. The wave length of red light is long enough so that it will not excite an electron from the valence to the conduction band, but will excite any carriers in the forbidden gap. When the red light was turned on, it caused the pulse to decrease very rapidly and completely disappear in 5 seconds. This occurred with either + or - bias. A white light would also cause the pulse to decrease, but not as rapidly as the red light.

There was one characteristic that was common regardless of how the pulse was lessened. After the pulse had been caused to disappear and to remain below the noise level for a period time using constant bias, the pulse would reappear by increasing the bias slightly.

Once the pulse height had been reduced, it would slowly recover after the bias was turned off. For example, after the pulse height had reduced to below 1 mv with a 50 volts negative bias, it took 15 minutes at zero bias (no light) before a 2.5 mv pulse could be seen again at 50 volts bias. The pulse from a positive bias appeared to recover about twice as fast as the negative bias pulse.

When light was shone on the crystal the rate of recovery was increased by about 5-10 times the previous rate. This increased rate of recovery would result from either red or white light. The recovery with white light was more rapid but this could be because of a greater light intensity since the red shield was removed.

The crystal was lowered to about -75°F using a dry ice acetone

slush around the chamber. With a positive bias, the voltage bias vs. pulse height relationships were the same as at room temperature except it was possible to go to a higher potential. At room temperature the maximum positive bias possibly before noise occurred was 125 volts, and at -75° F it was 200 volts. The pulse height did decrease with time, but at a much slower rate than at room temperature. Also after a period of 1 hr the pulse reached an equilibrium point of 80% of the original value and no longer decreased.

With negative bias at -75° F the voltage bias vs. pulse height relationships were not the same as at room temperature, but about twice the bias was required to obtain the same pulse height. However, it was possible to go to about twice the bias setting. The pulse height also decreased with time similar to that obtained with the positive bias.

The crystal was cooled to about -250° F using liquid N_2. At this temperature the pulse height was about half the value of that obtained at room temperature for both biases. The signal also became noisy at a very low bias (about 50 volts). When the temperature was raised to room temperature the original characteristics were restored.

It was not possible to measure the resolution since the pulse height decreased with time. The voltage bias and corresponding leakage current also decreased with time for both the positive and negative bias. Since the electrometer had a response time of 20 sec., the leakage current was taken to be the meter reading after 20 sec. This value is therefore somewhat low. The V-I relationship for C-3 is

shown in Fig. 6.

Crystal No. RS-1. As mentioned before, RS-1 was the only crystal tested that was a second distillation crystal. However, most of the characteristics of C-3 were found in RS-1. The pulse height decreased with time but not as rapidly as with C-3. With either bias the pulse height would decrease about 25% in 4 minutes. The relationship between pulse height and + or - bias is shown in Fig. 7. As was true with C-3, the pulse height values were obtained as soon as the bias was turned on, and before the pulse had had a chance to decay.

Light had the same effect on RS-1 as on C-3 -- both with respect to reducing the pulse when the bias was on and restoring the pulse after the bias had been reduced. Also, when the bias was turned off, and the polarity of the input to the amplifier switched, a fairly large pulse would be seen. To illustrate, at 40 volts negative bias, the pulse was originally at a height of 28 mv and decreased to 15 mv after 5 minutes. The bias was then turned off, the input to the amplifier switched, and a 25 mv pulse was seen. The more the original pulse was allowed to decrease, the larger the residual pulse.

With RS-1, in addition to the pulse that had a rise time of a fraction of a micro-second, there were some other pulses with a rise time of a micro-second or more. (The faster pulses were actually shaped by the pre-amp which means they had a rise time of a fraction of a micro-second or faster). These slow pulses were very erratic and ranged up to 100 mv pulse height and could be seen with either

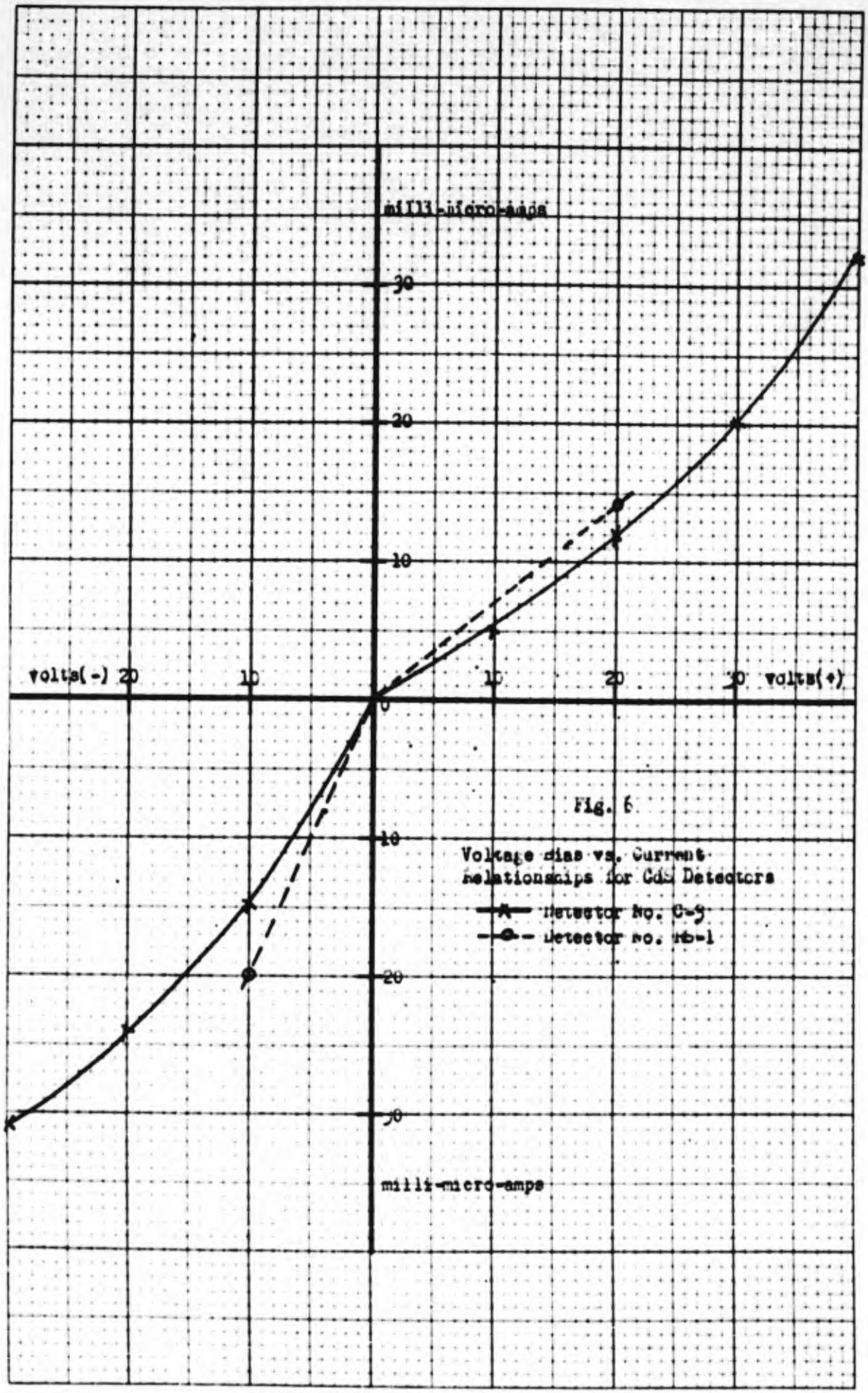

Fig. 6

Voltage bias vs. Current relationships for CdS Detectors

—x— Detector No. C-9

—o— Detector no. [illegible]-1

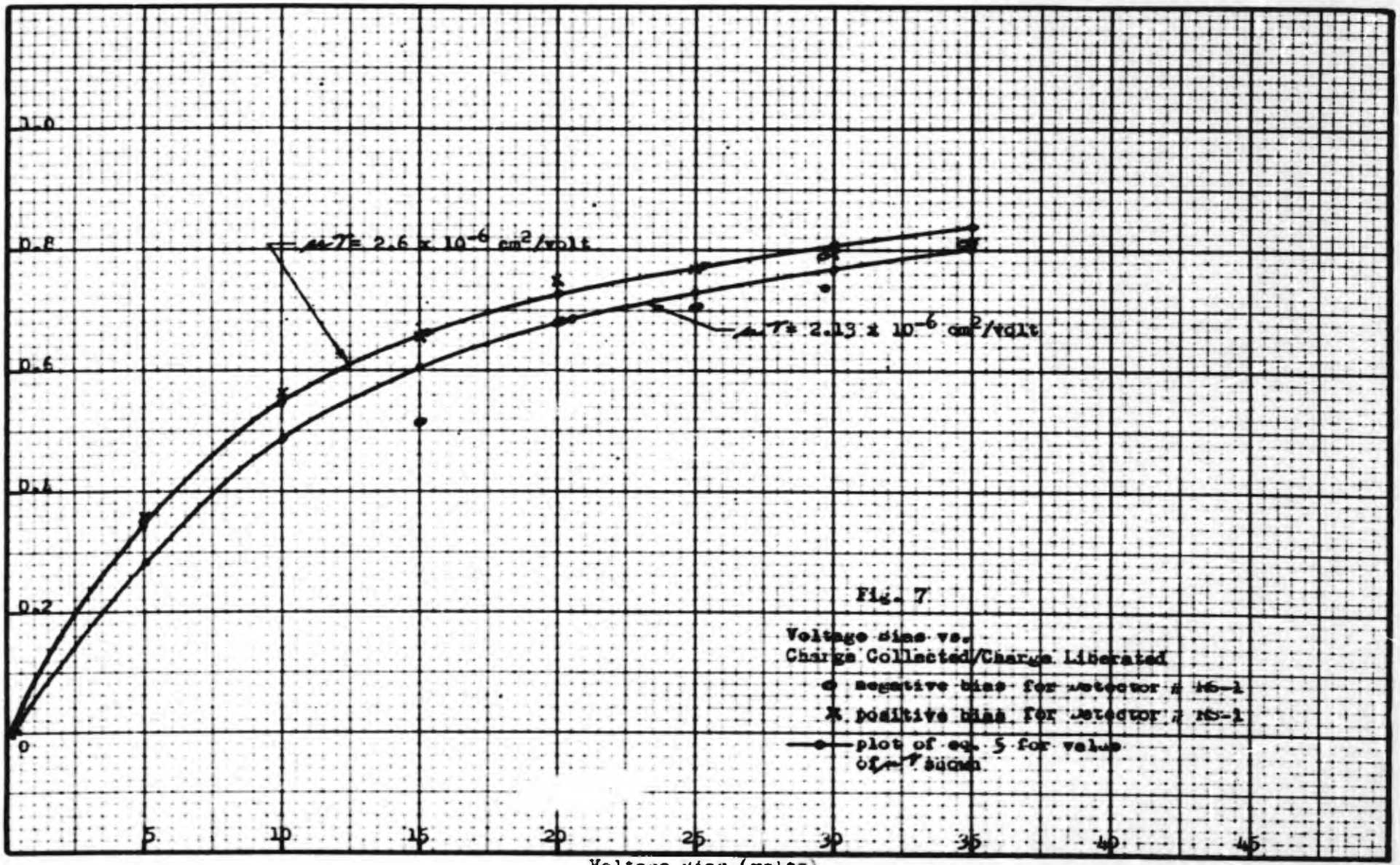

Fig. 7

Voltage bias vs.
Charge Collected/Charge Liberated

o negative bias for detector # 16-1

x positive bias for detector # 16-1

—●— plot of eq. 5 for value of $\mu\tau$ shown

positive or negative bias. However, the pulses were sufficiently infrequent that the fast rise time pulses could be distinguished.

The slow pulses appeared more prominent with the negative bias. In fact, when the negative bias was first turned on, i.e., when the detector had not previously been used for an hour or so, all that could be seen were slow erratic pulses. It took 3-5 minutes before the fast rise-time pulses could be distinguished. If the positive bias was turned on for about 5 minutes and then the bias switched, the fast rise time pulse with the negative bias could be seen immediately.

The crystal was lowered to -75° F and the pulse with a positive bias investigated. It was found that when the bias was first turned on that the pulse height obtained was about the same as that obtained at room temperature for the same bias. However, during the next couple of minutes the pulses increased about 7 or 8%. When the bias was turned off a small pulse remained. By contrast, at room temperature this build up was only very slight and was then followed by a definite decrease.

The V-I relationship for RS-1 is shown in Fig. 6.

Crystal No. R-3. The next crystal tested was R-3. The pulse height vs. positive voltage bias for this crystal is shown in Fig. 8. The pulse height did not change with time as was noted with the other two crystal. When red light was shone on the crystal, the pulse height decreased very slowly (about 25% in 10 minutes). The slow rise time pulse could be seen with this crystal similar to that seen with RS-1.

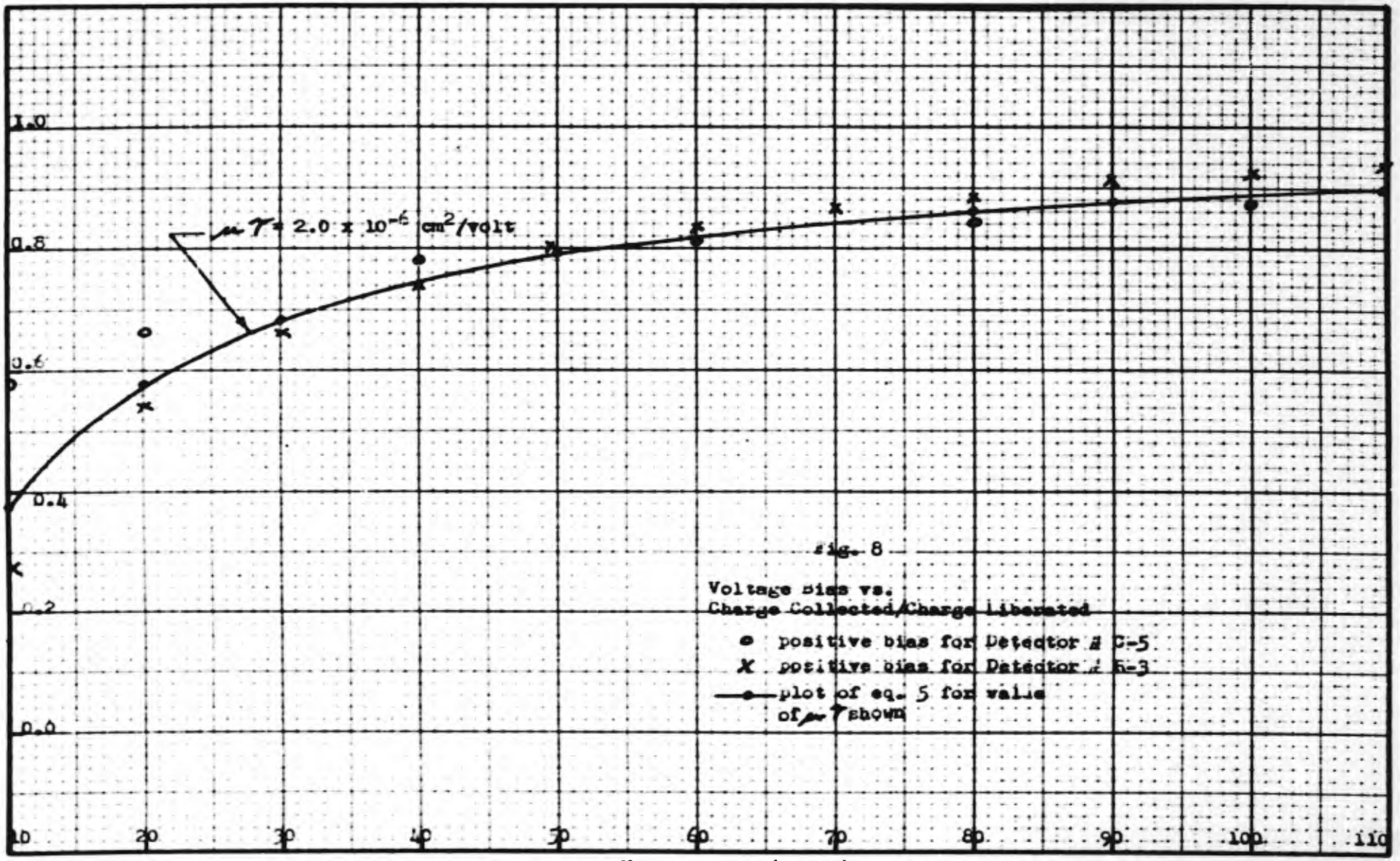

Fig. 8

Voltage Bias vs.
Charge Collected/Charge Liberated

● positive bias for Detector # C-5

X positive bias for Detector # B-3

—●— plot of eq. 5 for value of $\mu\tau$ shown

Since the pulse height did not vary with time, it was possible to obtain the pulse height resolution for this crystal. The resolution, shown in Fig. 9, was 6.8%. The sensitive detecting area was about 6 mm^2. No pulses were seen with the negative bias. The voltage-current relationships are shown in Fig. 10. Although R-3 was constructed as an intrinsic detector, it can be seen from Fig. 10 that it exhibited rectifying properties.

Crystal No. C-5. One CdS junction crystal was studied. The pulse height vs. positive bias relationships for this crystal are shown in Fig. 8. Pulses could be seen only with the positive bias. The voltage-current characteristics are shown in Fig. 10. The pulses from the positive bias did not decrease with time, but they did decrease at about the same rate as R-3 when the red light was turned on. The leakage current decreased with time as has been noted with all of the crystals discussed thus far.

This crystal was a large crystal about 250 microns thick, with a sensitive detecting area of about 25 mm^2. The pulse height resolution was very poor -- about 25%. In an attempt to improve the resolution, a portion of the crystal was masked. With 7mm^2 area exposed, the resolution was 16% and with 2mm^2 exposed, the resolution was 9.4%.

When the crystal was lowered to -75° F, the pulse height corresponding to a given bias was less than one-half that obtained at room temperature.

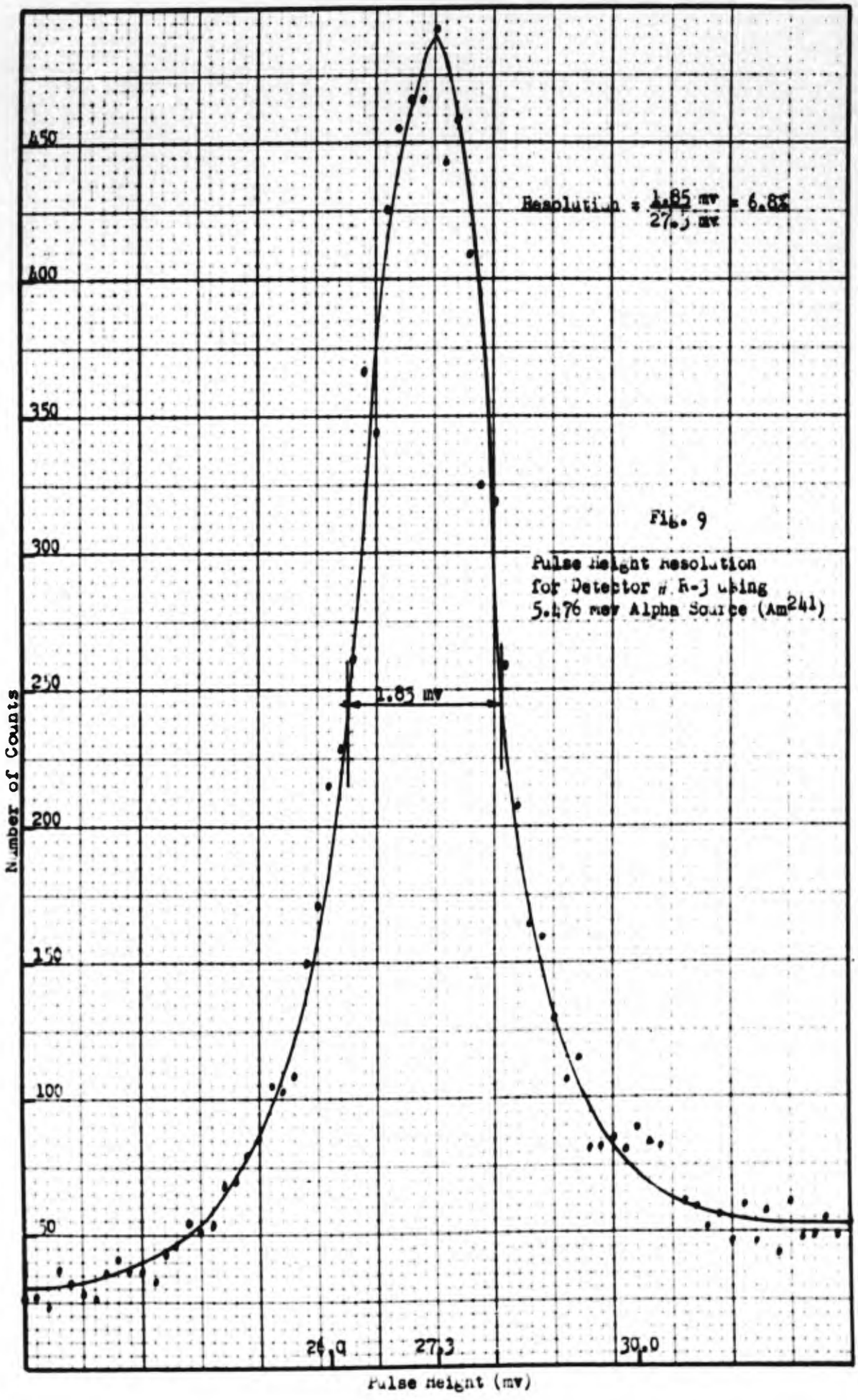

Fig. 9

Pulse Height Resolution for Detector # R-3 using 5.476 mev Alpha Source (Am^{241})

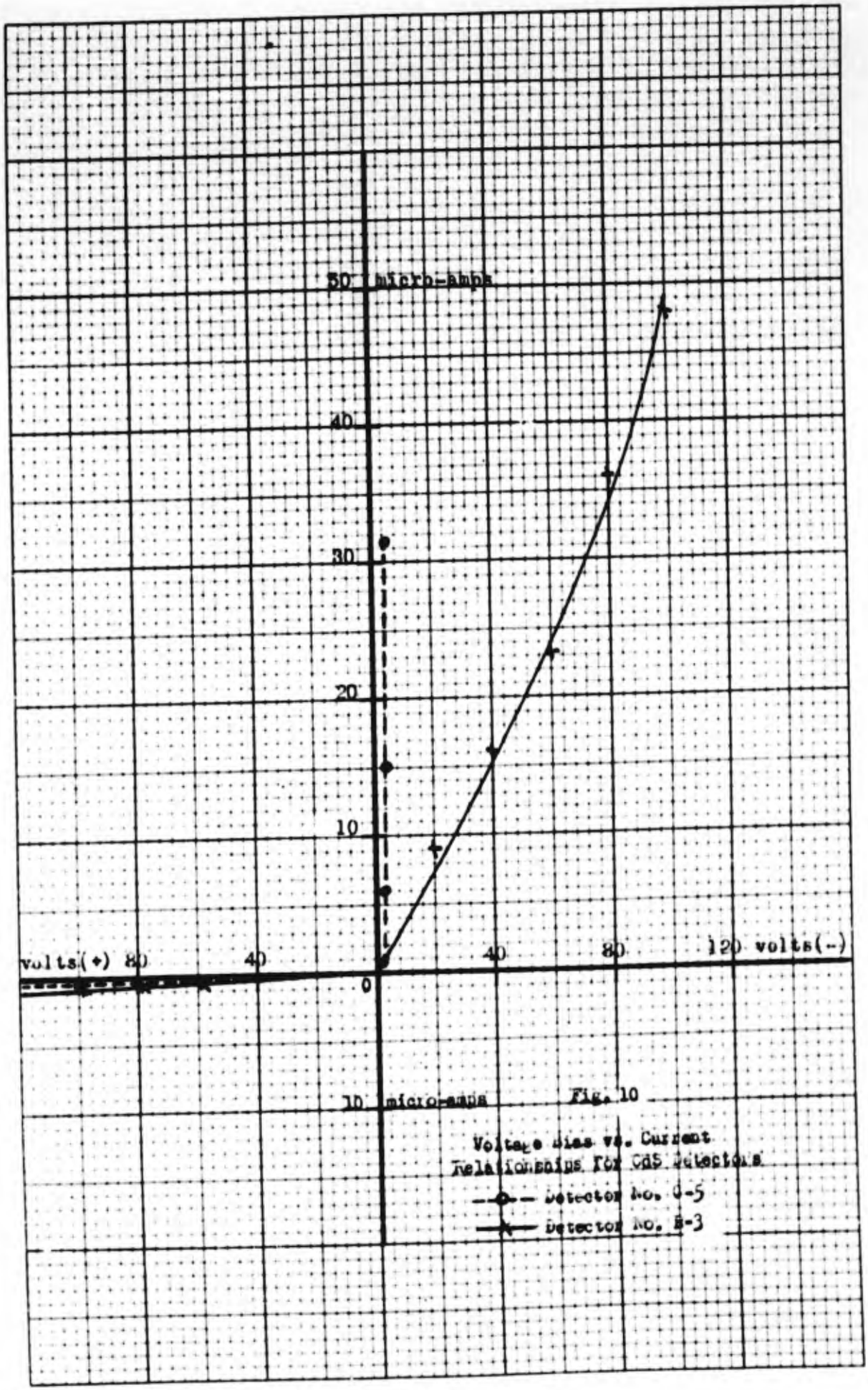

Fig. 10

Voltage bias vs. Current
Relationships for CdS detectors
—o— Detector No. C-5
—x— Detector No. E-3

Other Crystals. Five other intrinsic crystals were made: M-1, M-2, M-3, R-1, and R-2. The first four all produced a large number of slow rise time pulses with either positive or negative bias. The height and number of these slow pulses varied with the crystal. However, no fast rise time pulses could be seen on any of them.

With R-2, although the slow pulses were predominent, it was just possible to distinguish the fast rise time pulses. Due to the slow pulses, the pulse height of the faster pulses was difficult to judge, but the values obtained for both positive and negative bias are shown in Fig. 11.

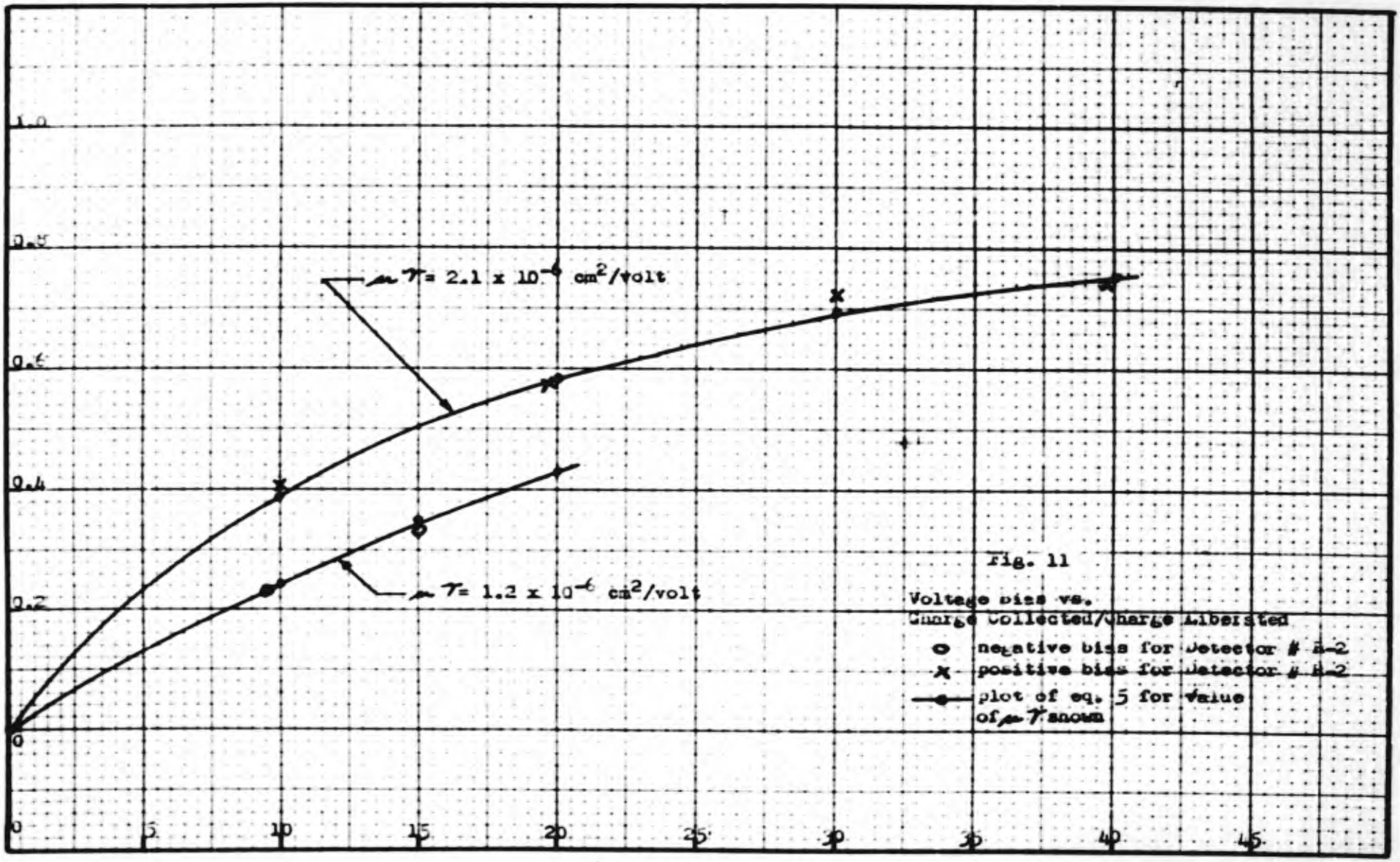

Fig. 11

Voltage bias vs.
Charge Collected/Charge Liberated

o negative bias for detector # B-2

x positive bias for detector # B-2

plot of eq. 5 for value of $\mu\tau$ shown

V. Interpretation of Results

Silicon Detectors

The intent of the work with silicon detectors was purely for the purpose of becoming familiar with solid state detectors. This was accomplished by fabricating some silicon detectors following standard techniques. Various recipes have been published for the construction of silicon detectors; in this case it was convenient to use the Harwell Report (Ref 3:2-4). The work followed successfully except for the excessive leakage current that developed in the detectors which will be discussed below.

Good commercial silicon detectors have leakage currents of less than 1 μa at 50 volts bias and resolutions of 1% or less. The silicon detectors constructed in this experiment were very excessive in leakage current, and were limited to a bias of 6 volts. The leakage current was probably due to surface effects of some kind, that is, either ionic contamination or the forming of inversion layers. This is indicated from the shape of the V-I curve (see Fig. 4) in that the p-n junction should not break down at this low bias. Also, the fact that the diffused junction detectors gave V-I curves similar to the surface barrier detectors and, of course, were constructed by a completely different process, indicates the surface leakage problem.

Although the final rinse water had a resistivity of 5 meg-ohm-cm, there is a possibility that other impurities were present which were not ionic. Therefore, in view of the precautions taken to insure

that the crystal was not contaminated with ionic impurities, it is felt that leakage was due to the formation of inversion layers on the silicon.

The pulse heights were much less than the 49.5 mv pulse height expected from a Pu^{239} source. However, a 6 volt bias produces a depletion layer only thick enough to stop a 3.4 Mev alpha. Therefore, if it is assumed that the energy lost per distance traveled is constant, the pulse height from this alpha would be 33 mv. However, since dE/dx is not constant, but increases as the particle slows down, the pulse height expected would be slightly less than 33 mv. Furthermore, for a higher energy particle, the charge created outside the depletion region will contribute little to the pulse height and actually harm the resolution. The reason is that some of the charge would diffuse into the depletion region after the other charge has been collected. As a result, in view of the high leakage current and low bias voltage, the pulse heights and resolutions obtained are reasonable.

Potting of the detectors in an epoxy resin was attempted to reduce leakage. It was later determined that some epoxy resins contain amines which impair the formation of surface barriers on n-type silicon. This was evidently the problem with these detectors, particularly since the crystals did detect somewhat after the surface was recleaned. It is felt however, that successful potting is not the answer to the leakage problem. Rather, it is probably related to the processing of the crystal before the surface barrier is formed.

At this point it was decided to drop further work on Si

detectors, since the original aim was only to develop suitable techniques for CdS detectors. Operating Si detectors were constructed, and further refinement was not attempted.

Cadmium Sulfide Detectors

The results obtained from the CdS detectors clearly demonstrate the phenomena of detection of individual alpha particles. However, the results indicate that the crystals tested in this work are limited in value for direct application as nuclear radiation detectors. This is primarily because the pulse height produced by the detectors was not proportional to the energy of the incident particle. Two factors caused this non-proportionality:

(1) The carriers, on the average, recombined before they were collected at the terminal. (This facilitated the measurment of the mobility-lifetime product for these carriers.) (2) The pulse height, obtained with the intrinsic detectors from a monoenergetic source, were not constant, but reduced with time. The pulse heights of both the intrinsic and junction detectors reduced when light was shown on the crystal.

Another detrimental phenomena which was present in most of the detectors was secondary current. This current served chiefly to harm the resolution of the primary pulse. Furthermore, it resulted in extraneous counts, which restricted the crystal from use as a counter.

Although the results indicate that CdS crystals, as presently fabricated, have limited value as radiation detectors, certain

parameters of CdS were measured which are felt to be significant. These were: (1) the measurement of the energy required to create a hole-electron pair (ϵ) in CdS, and (2) the mobility-lifetime product ($\mu\tau$) of holes and electrons in CdS by method not previously used.

The remainder of this chapter will discuss more fully the parameters measured and the other phenomena noted above in the following order: (1) determination of ϵ and $\mu\tau$, (2) secondary current, (3) decrease of pulse height with time, temperature and light, and (4) other results.

Determination of ϵ & $\mu\tau$. The mobility-lifetime product and the energy necessary to create a hole-electron pair were determined from the voltage bias vs. pulse height relationships of the CdS detectors (see Figs. 5, 7, 8, and 11.) These parameters were obtained by fitting eq (5) to these experimental relationships. In order to do this, different values of $\mu\tau$ and Q_L (the two unknowns in the equation) were inserted into eq (5), until the curve that resulted had the same shape as the experiment plot. It was possible to determine both parameters since one unknown was an exponential function and the other was a linear function.

For the six, voltage bias vs. pulse height curves obtained for intrinsic detectors, Q_L was the same, i.e., 33.4 mv. From Q_L, ϵ could be measured as follows: For a 5 pf capacitor, 33.4 mv is equivalent to 1.67×10^{-13} coulomb. Thus, since a 5.476 Mev alpha was used as a source, the resultant value for ϵ in CdS is 5.2 ev/pair.

Table II shows the values for $\mu\tau$ necessary to fit each of the experimental curves. All experimental values obtained for collecting electrons follow the curve quite well, accept for C-5 and R-3 (Fig. 8). The latter two were junction crystals and would not be expected to follow eq (5) since junction detectors have varying (linear) rather than constant electric fields. With C-5 the junction was formed on the front surface and it can be seen that the best collection and therefore the maximum electric field existed at this point. Upon examining the plot obtained with R-3, it appears that the maximum field was at the rear of the crystal, indicating that the rectifying contact was at that point. Although the field did vary within the crystal, it can be seen that $\mu\tau$ was approximately 2.0×10^{-6} cm^2/volt.

According to Reynolds (Ref 12) the mobility of electrons in CdS is approximately 300 cm^2/v-sec. The lifetime of electrons is believed to be around 10^{-6} to 10^{-7} seconds, although accurate measurements have not been made. The values in this experiment are slightly lower than that indicated above. RS-1 has the largest $\mu\tau$ product measured as would be expected in view of its higher purity.

The best experiment plot obtained with negative bias (collecting holes) was with RS-1. (It was difficult to see the primary pulse below 20 volts. Note the error in the experimental value at 15 volts.) Although only two points were obtained when collecting holes in R-2, the $\mu\tau$ obtained agreed favorably with RS-1 and C-3. The values for collecting holes in C-3 did not match the theoretical curve completely. If the curves were matched at low bias as they were in this case,

Table II

The Mobility-lifetime product for the CdS Detectors

Crystal No.	Mobility-lifetime product (cm^2/volt)
C-3	
electrons	1.6×10^{-6}
holes	3.3×10^{-7}
RS-1	
electrons	2.6×10^{-6}
holes	2.1×10^{-6}
R-2	
electrons	2.1×10^{-6}
holes	1.2×10^{-6}
C-5	
electrons	2.0×10^{-6}
R-3	
electrons	2.0×10^{-6}

then the experimental values fell below the theoretical curve at higher bias. The reason for this is believed to be that the pulse had decreased with time before the higher values of bias could be reached. The pulse had a chance to decay in this instance because it was necessary to turn up the bias rather slowly or the detector would become noisy.

It should be noted, that the crystals that exhibited low values for $\mu\tau$ when collecting holes also gave low values when collecting electrons. This follows logically, since the number of recombination centers for holes or electrons are related to the number of impurities in the crystal.

Recently, Mort and Shear (Ref. 10:314-315) determined the lifetime and mobility of holes in CdS by pulsing the crystals with 50 Kev electrons. The values for lifetime were between 1 and 3 x 10^{-7} sec, whereas the values for mobility were from 10 to 18 cm^2/v-sec with a mean value of 15 cm^2/v-sec. Therefore, an average value for $\mu\tau$ would be 3.0 x 10^{-6} cm^2/volt. This compares favorably with 2.1 x 10^{-6} cm^2/volts obtained for RS-1. Also the value of 1.2 x 10^{-6} cm^2/volt for B-2 is within the range of values obtained by Mort and Shear.

One error in the determination of the mobility-lifetime product is that the charge was not all created at the surface, but on the average of about 7 microns below the surface. Since the crystals range in thickness from 60 to 100 microns, the calculated values of $\mu\tau$ are estimated to be too large by about 10%.

Secondary Current. Of the seven intrinsic crystals constructed, six of them displayed large, slow-rise time pulses with either bias. In four of the crystals, these pulses were so prominent that the fast rise time pulses could not be seen. The explanation proposed for this is that these pulses are from charges that have been injected from the terminal to neutralize holes that were trapped. This was the situation mentioned in the Theory where preferential trapping of one of the carriers occurred before the ionized charge carrier recombined or was collected at the terminal. The one crystal, C-3, in which secondary current was not seen, probably had non-injecting terminals. This type terminal results when the recombination centers are so numerous at the terminal, that carriers can not pass through.

Secondary current has been identified before in CdS crystals (Ref 8:1). The large size and slow rise time of the pulse results when the electrons which are injected, pass through the crystal without finding the hole. The trapped hole, being immobile, can not move to meet the electron. The superposition of the primary pulse on the secondary pulses is illustrated in Fig. 9 for crystal R-3.

For RS-1 all that could be seen with negative bias for the first five minutes were secondary pulses, after which time primary pulses were observed. But with positive bias the primary pulse could be seen immediately. An explanation for this is that for the first five minutes, the holes were jumping from one trap to another across the crystal until virtually all the hole traps were filled. With the traps filled additional holes would not be trapped and move until

they either recombined or reached the terminal.

Decreasing of Pulse Height. Of the four crystals that were closely examined, the two intrinsic crystals, C-3 and RS-1, showed a decrease in pulse height with time, whereas the two junction crystals, C-5 and R-3 did not. The opposite polarity pulse could be seen with the intrinsic crystals when the bias was turned off. When light was shone on the crystals, the pulse from both the intrinsic and junction crystal decreased, but the intrinsic crystal pulse height decreased much more rapidly. The pulse from C-3 decreased extremely fast at 60° C and, both RS-1 and C-3 had a very slow pulse height decrease at -75° F.

The appearance of the opposite polarity pulse at zero bias suggests that the carriers had been trapped while moving toward their terminals, thereby creating a space charge; and this is the cause of the pulse decreasing with time. However, the effect of light and temperature indicate that neutral impurities are ionized by light or high temperature, and that they cause the polarization in the crystal. Which of the above is actually taking place is not known, possibly both are occurring. In either case one would expect light to aid in the recovery of the pulse height as was noted in this experiment.

The reason that the pulse height with a p-n junction does not decrease with time and decreases very slowly with light, is not understood at this time.

Other Results. The pulse height resolution for R-3 (Fig. 9) was 6.8% at 60 volts bias. This is especially good considering the

secondary current present and the fact that only 82% of the charge was collected at that bias. From the appearance on the CRO of the pulses from the intrinsic detectors, they probably would give similar values for resolution. However, these resolutions could not be obtained since the pulse decreased with time. The resolution of C-5 improved drastically when the area for detection was reduced. Evidently an uneven distribution of impurities, thus recombination centers, were present in the crystal.

The other results will be summarized with only suggested explanations since they are not completely understood at this time:

(1) The leakage current decreased with time with all the CdS detectors. (This indicates a build up of space charge from trapped carriers.)

(2) Often the pulse height was reduced at low temperature. (Contact problems could explain part of this.)

(3) With RS-1 at -75° F, the pulse height increased with time by about 8 per cent. (Here again the electric field distribution has evidently shifted. The cause is not known.)

(4) The decrease in pulse height with time appeared to be confined to region of the crystal that was being radiated. (This is understandable assuming the decrease in pulse height is caused by a build up of trapped carriers or the ionization of neutral impurity atoms in the region where the radiation is incident.)

VI. Conclusions

The energy required to form a hole-electron pair in CdS was 5.2 ev/pair. This quantity has not previously been measured but according to Shockley, it should be proportional to the band gap width. For Si and Ge, the energy required per pair is approximately three times the band gap width. In CdS the band gap is 2.42 ev. Thus, in this case the energy per pair is slightly over twice the band gap.

The best experimental value for the mobility-lifetime product in CdS was 2.6×10^{-6} cm^2/volt for electrons and 2.1×10^{-6} cm^2/volt for holes. Although these values were obtained by methods not previously used, they agree favorably with values obtained by other methods.

In view of the low mobility-lifetime product for both holes and electrons, the CdS crystal as presently fabricated has limited use as a detector. Other undesirable characteristics such as secondary current and the decrease of pulse height with time, high temperature, and light, further limit its usefulness.

Possible areas for further work in this field are as follows:

(1) The values of $\mu\tau$ and ϵ for CdS could be verified by studying more CdS crystals. Since only one second distillation crystal was studied, it would be especially interesting to investigate more of them.

(2) The mechanism by which the pulse height decreased with time could be studied further. The effect of high temperature on the

junction crystals would aid in explaining this phenomena.

(3) The techniques developed here could be used to determine the mobility-lifetime products and the energy required to form a hole-electron pairs for other crystals. The values for $\mu\tau$ plus other characteristics of the crystal realized during the study would determine the potentiality of that crystal as a nuclear radiation detector.

(4) The studies at low temperature (liquid N_2 or lower) were incomplete in this work and much more information about the crystals parameters could be determined by further work in this area.

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Vita

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This thesis was typed by Miss Judith A. Rowell.

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