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RESEARCH STUDY FOR
INCREASING THE SENSITIVITY OF PHOTOEMITTERS
(UNCLASSIFIED TITLE)

CONTRACT DA44-009-ENG-4913

1 JULY 1964 - 30 SEPTEMBER 1964

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RESEARCH STUDY FOR INCREASING THE SENSITIVITY OF PHOTOMITTERS

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ENGINEER RESEARCH AND DEVELOPMENT LABORATORIES

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October 22, 1964

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PURPOSE

To investigate the physical and structural properties of photoemitting materials, to study and develop processing procedures for producing improved photocathodes, and to investigate the possibilities of field induced photocatheters.

ABSTRACT

Sb-films evaporated from PtSb-beads have higher conductivity and lower light transmission than films evaporated from pure Sb-beads. They also can be oxidized. S-20 cathodes formed on oxidized Sb-films and subjected to a Na-Sb alternation at 220°C have shown higher infrared response than any earlier cathodes.

Attempts to produce S-20 cathodes by simultaneous deposition from molecular beam sources yielded a multi-alkali cathode of low sensitivity. Preliminary experiments on improved molecular beam sources and a control circuit have been made.

The modified reflection electron diffraction tube has been successfully tested using gold as a sample material.

Preliminary data concerning the structure of S-I cathodes has been obtained by electron diffraction studies. The results indicate that the finished cathode contains Cs$_2$O or a phase closely related to it. The presence of other phases is also possible.

Apparatus for vapor etching and diffusion of Ge p-n junctions for field induced photocathode has been completed.
I. PROCESSING OF S-20 PHOTOCATHODES

A. Introduction

In the previous Quarterly Report, initial experiments were described concerned with the new RCA-Lancaster process of activating S-20 cathodes on an oxidized antimony substrate. This study was continued and the earlier finding was confirmed that the new process actually involves two changes from standard procedure in that the antimony film, even before oxidation, differs from conventional antimony deposits because it is evaporated from a PtSb as distinct from an Sb-bead. In addition to these two new processing parameters, experiments were also made with a new method of introducing sodium by alternation with antimony at high temperature.

By adding the three above-mentioned processing variables to the many existing ones, the number of possible combinations has increased to such an extent that it has become practically impossible to adhere to the basic principle of experimentation, viz., that not more than one variable should be changed in any one experiment. As a result, the conclusions that can be drawn from the twenty-one experimental tubes made during the past quarter are still ambiguous in many respects; however, definite progress has been made in extending the long wavelength response of S-20 cathodes. The experiments to be described below show that under certain conditions the long wavelength threshold can be extended beyond any previously observed values.

Superimposed on this effect of threshold extension is the previously investigated effect of improving long wavelength response at the expense of blue response by using thicker cathode films which absorb more long wavelength radiation. Whereas the first effect seems to represent a genuine extension of the threshold wavelength, the thickness increase, as one would expect, only tends to increase the quantum efficiency at a given wavelength. Thus, for
applications where the response above, say, 8000 Å is the main consideration, improved performance can be obtained by greater thickness of the cathode, even though the blue response and the microampere-per-lumen value may be reduced. To sum up, in the experiments to be reported below in detail, the following four parameters were examined:

1. Effect of Sb-substrate.
2. Effect of oxidation of Sb.
3. Alternating evaporation of Na and Sb at 220°C.
4. Thickness of cathode film.

As explained above, it was often unavoidable to vary more than one parameter in one particular tube; hence, a description of individual tubes would be confusing. The following report will discuss the four stated parameters separately, although the same tube may have been used to check more than one of these parameters.

B. Processing Experiments

1. Antimony Substrate

It was suggested in the last report that Sb may deposit in the form of single atoms when it is evaporated from PtSb beads, in contrast to the Sb₄ molecules known to be produced when elementary antimony is evaporated. The main differences in the deposits were the low resistance of the deposit produced from PtSb and the ease with which this deposit could be oxidized in a glow discharge. Every tube made during the present quarter confirmed these two features. The following experiments were made to gain more information about the difference between the two types of Sb deposit.

(a) Attempts were made to oxidize Sb deposits by heating them in the presence of oxygen. In tube S.1527, Sb was evaporated from PtSb to 85% light transmission. The deposit was baked to 160°C. The resistance between two
contact strips was $10^6$ ohms at this temperature. On introducing oxygen to a pressure of approximately 0.1 mm Hg, the resistance immediately rose to a value beyond the measuring range (> $10^3$ ohms) and the light transmission increased to practically 100%. Both effects clearly indicate that the Sb film was completely oxidized.

The same experiment was performed with an Sb deposit produced from an Sb-bead. In this case, the resistance, even at 160°C, was too high to be measurable, the usual experience with Sb-bead films of this thickness. On introducing oxygen, the light transmission did not change. These results indicate that the effect of oxygen at high temperature is the same as that of the oxygen discharge, i.e., Sb evaporated from Sb cannot be oxidized whereas Sb from SbPt can be oxidized.

(b) Sb-films of identical light transmission were deposited from Sb and SbPt beads on carbon films for electron microscope and electron diffraction studies. These studies (carried out by Messrs. M. D. Coutts and W. C. Roth of the Laboratories Materials Analysis Group) are not yet complete, but the preliminary results are so clear-cut that they can be reported at this time. The electron microscope pictures show that the film produced from SbPt consists of extremely small particles (linear dimensions of the order of 35 Å) which are so uniformly distributed that hardly any structure can be detected. The films from Sb-beads consist of much larger particles (linear dimension of the order of 350 Å) which are clearly separated from each other. Initially, the electron diffraction pattern of films deposited from SbPt-beads is that of the so-called amorphous form, but crystallization sets in rapidly under continual electron bombardment.

These results suggest the following interpretation of the resistance and oxidation effects: The films from Sb-beads have immeasurably high resistance.
5. because they consist of separate particles. On the other hand, the films from SbPt-beads are sufficiently continuous to be conducting. It is also plausible that the films consisting of smaller particles are more readily oxidized. Moreover, the smaller particle size, though no positive proof, agrees well with the assumption that Sb evaporates in the atomic form from SbPt-beads.

(c) Since the particles evaporated from Sb-beads are separated by distinct gaps, one would expect that for equal amounts of Sb per cm² the light transmission should be greater than for the continuous films from SbPt. Conversely, for the same light transmission, films evaporated from SbPt should contain less Sb per cm² than films evaporated from Sb. To test this conclusion, a cylindrical tube (8.1550) with two parallel windows was made as shown in Fig. 1. A moveable semicircular nickel mask could be placed by means of a magnet so as to cover either one or the other half of the window. Sb was evaporated from an Sb- and an SbPt-bead, respectively, on the two halves to exactly the same light transmission of 70%. (As the photograph shows, the two areas actually overlap over a small sector.) The Sb-films were then simultaneously activated with Cs to form a conventional Cs₃Sb cathode. As had been expected, the cathode area formed with the SbPt-bead looked considerably thinner than the other half because it contained a smaller amount of Sb per cm². This conclusion was confirmed by the fact that the area deposited from SbPt had considerably higher white and blue, but equal red, response in agreement with the often-discussed fact that for semitransparent cathodes thinner layers are more efficient in the short wavelength range where the absorption of Cs₃Sb is very high. A more quantitative determination of the thickness ratio of the two cathode halves as well as the detailed findings of the electron microscope and diffraction studies will be reported in the next quarter.
Electron diffraction studies were made in this laboratory using the transmission electron diffraction apparatus designed for observing photocathode formation. Since the films could be evaporated and examined without breaking vacuum, this eliminated any effect of superficial oxidation which might have been present in the samples given to the Materials and Analysis Group. However, the results of our diffraction study are in essential agreement with their conclusions. We found that in no case was it possible to crystallize by electron bombardment a layer of Sb whose light transmission was greater than 60% which had been evaporated from PtSb. In contrast, layers of Sb evaporated from pure Sb were crystalline or became crystalline under beam annealing with light transmittances as high as 85%.

Our observations are then compatible with the statements made above concerning the relative thickness of the two types of films having the same light transmittance since it has been observed\(^1\) that a threshold thickness must be reached before crystallization takes place. Under normal conditions, this threshold value is about 180 Å. However, it appears that under the special conditions of electron bombardment, this value can be considerably smaller but the threshold effect is still present.

(d) One experiment (S.1535) was made to determine whether an Sb-deposit evaporated from an Sb-bead would change at high temperature to the more continuous modification. The film was therefore heated to 270°C, the highest temperature compatible with the vapor pressure of antimony, but there was neither a visual change nor an increase in conductivity, indicating that the structure of the film had not changed. It is worth noting that subsequent S-20 activation did not show any effect, beneficial or detrimental, of the high temperature treatment.

---

As far as the correlation of the Sb-film studies with actual cathode activation is concerned, there is no evidence so far that the structure and/or sensitivity of the completed cathode is affected by the particle size of the original Sb-film. Structure studies with activated cathode films are more difficult to make because the vacuum in the electron microscope is not good enough for cathode activation; attempts will be made to distinguish cathodes formed on the two types of Sb-substrate in the high vacuum electron diffraction tube, but these experiments - as indicated - suffer from the risk of changing the film structure by electron bombardment before the first diffraction pattern is obtained. The construction of an emission microscope could possibly be of some value here, although the resolution might limit its usefulness. In one respect, however, the effect of the Sb-source on the final cathode is obvious: The standard method of determining film thickness by measuring light transmission is not valid since deposits of different thickness but identical light transmission are produced depending on whether the source of Sb is an SbPt- or an Sb-bead.

2. S-20 Cathodes on Oxidized Antimony

As has been pointed out before, Sb-films deposited from Sb-beads cannot be oxidized by either glow discharge or heating in oxygen. Therefore, all experiments with oxidized Sb-substrates had to be performed with films evaporated from SbPt. This is unfortunate because it makes it impossible to determine whether any effect of oxidation on cathode activation is only obtained in combination with an Sb-film deposited from SbPt.

Many experiments were made to establish the optimum conditions for oxidation. The possibilities are almost infinite in number: One can vary the thickness of the original Sb-deposit and the degree of oxidation; additional Sb may be deposited on the oxidized film and the thickness of this top
layer can again be varied; even after completion of Sb evaporation and oxidation, the subsequent stages of the process may have to be changed from the standard procedure to obtain optimum sensitivity and red response. Since each result should really be verified in more than one tube, it is not surprising that the experiments to be described lead only to preliminary conclusions.

(a) Oxidation without Subsequent Sb-Deposition

In tube S.1522, a relatively thick (63% light transmission) Sb-film was oxidized to 73% light transmission. Subsequent standard S-20 activation produced unusual readings; in particular, the addition of Na never produced the increase in sensitivity commonly observed and the cathode showed n-type conduction throughout the process. The final sensitivity was only 100 μA/l. Thus, the omission of the additional antimony evaporation after partial oxidation of a relatively thick Sb-substrate does not look promising, although it must again be emphasized that in this type of experiment a single result cannot be considered conclusive.

In tube S.1527, a thinner Sb-film (85% light transmission) was completely oxidized by heating in oxygen. Again, no further Sb was added. The standard S-20 process in this tube produced a sensitivity of 150 μA/l and normal color response. The most noteworthy result of this experiment was that the characteristics of the final cathode were indistinguishable from those of a standard cathode containing no oxygen.

In tube S.1530, an attempt was made to form a K₂CsSb cathode on an Sb-substrate oxidized at 160°C like S.1527 and to convert this cathode into an S-20 cathode. This process, which works with unoxidized Sb, was a complete failure.

As mentioned earlier, attempts to oxidize Sb-substrates deposited from Sb-beads by glow discharge in oxygen (S.1526) and by heating in oxygen
were unsuccessful. In both tubes, subsequent S-20 activation led to sensitivities in the 100 µa/l range.

(b) Oxidation with Subsequent Sb-Deposition

In tube S.1542, a very thin (97% light transmission) Sb-substrate was oxidized and a relatively thick (70% light transmission) Sb-film was deposited on the oxide. The final sensitivity of the S-20 cathode was 180 µa/l, but the response beyond 7500 A was poor. A repeat of the process in S.1543 produced lower sensitivity, but in both experiments - though more markedly in S.1543 - an effect was observed that seems to be tied to the oxidation process: Whereas in the standard S-20 process introduction of Na at 220°C causes the sensitivity first to rise and then to drop to a value which remains constant on discontinuing the Na supply, the sensitivity appears to recover in all cases where the Sb has been oxidized. There also seems to be a correlation between this recovery of sensitivity after Na addition and final sensitivity in that the final S-20 sensitivity is lower if the recovery is more complete, possibly because the Na is not retained. This observation led to the process to be described in Section (3).

Two tubes (S.1534 and S.1538) were made to check the possibility that there may be a similarity between the effect of the oxidized antimony substrate and the improved performance of CsSb cathodes deposited on a thin manganese oxide substrate. In both tubes, a normal S-20 process was carried out after the initial Mn film had been oxidized. The results were the same in both experiments: Na₂KSb cathodes of average sensitivity were obtained but release of Cs produced an immediate drop in sensitivity, and the peak sensitivity was only in the 100 µa/l range. Thus, there does not appear to be any obvious advantage in the manganese oxide substrate, but more experiments will have to be made to establish whether the unusual drop on admission of Cs was coincidental or related to this particular process.
3. Na-Sb Alternation at 220°C

As mentioned in the previous section, tubes with oxidized antimony are characterized by the difficulty of incorporating the required amount of Na by the conventional method of exposing the K₃Sb cathode to Na vapor at 220°C. In tube S.1544, an attempt was therefore made to ensure the retention of Na through chemical binding by alternating the exposure to Na vapor with the evaporation of small amounts of Sb. This process led to a final sensitivity of 260 μA/l (the tube had cylindrical, as distinct from the usual spherical, shape and the sensitivity was probably enhanced by reflection from the anode; however, this effect is not likely to exceed 10%). More significantly, the cathode had very high response above 8000 Å, as can be seen from Fig. 2 where the curve of a high μA/l tube (S.1367) with "normal" long wavelength response is shown for comparison. An earlier tube (S.1516) with oxidized Sb-substrate but without Na-Sb alternation is also represented in Fig. 2.

The combination of oxidized Sb-substrate and Na-Sb alternation was repeated in S.1546. This cathode had lower overall sensitivity (160 μA/l) but exceptionally high infrared response, both probably the result of greater film thickness (see section (4)). The response curve of S.1546 is also shown in Fig. 2. The long wavelength threshold has the highest value measured for an S-20 cathode in this Laboratory. A significant feature of this activation process was the unusually high red response in the Na₂KSb stage.

If the good performance of S.1544 and S.1546 was not fortuitous, it could still be due either to the Na-Sb alternation alone or to the combination of Na-Sb alternation and oxidized Sb-substrate. It was therefore logical to try the Na-Sb alternation in combination with unoxidized Sb-substrate.

The Na-Sb alternation at 220°C had actually been tried earlier with another purpose in mind. In tube S.1523, an attempt had been made to convert
\[ \text{MA/WATT} \]

<table>
<thead>
<tr>
<th>( \mu \text{A/l} )</th>
<th>FILTER</th>
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<tbody>
<tr>
<td>S.1516</td>
<td>190</td>
</tr>
<tr>
<td>S.1544</td>
<td>260</td>
</tr>
<tr>
<td>S.1546</td>
<td>160</td>
</tr>
<tr>
<td>S.1367</td>
<td>200</td>
</tr>
</tbody>
</table>

\[ A \]

7000 7500 8000 8500 9000 9500
a \(K_2\text{CsSb}\) cathode into an S-20 cathode by this process. Good final sensitivity had been obtained (175 \(\mu\text{a}/\text{l}\)) but the long wavelength response was rather low so that the experiment had not been repeated. A second earlier experiment (S.1525) with the standard sequence of alkali metals and Na-Sb alternation at 220\(^\circ\)C produced an excessively thick cathode and seemed at the time not worth repeating.

In tube S.1547, an Sb-film was deposited from an SbPt-bead, but the film was not oxidized. Na-Sb alternation at 220\(^\circ\)C produced again a \(Na_2\text{KSb}\) cathode of unusually high red response, but after the Cs process, the sensitivity was only 150 \(\mu\text{a}/\text{l}\) and the infrared response was not above average.

The process was repeated in S.1548 with an Sb-film deposited from an Sb-bead. The cathode had again very high red response in the \(Na_2\text{KSb}\) stage but only average white and infrared sensitivity after the Cs process. If one dares to draw conclusions from the limited experimental evidence, the results of S.1544 and S.1546 on the one hand, and of S.1547 and S.1548 on the other, would indicate that high red response in the \(Na_2\text{KSb}\) stage is associated with the Na-Sb alternation and that the combination of oxidized Sb-substrate with Na-Sb alternation is required for high infrared response in the final S-20 cathode.

4. Effect of Cathode Thickness

It has been pointed out in previous reports that the light absorption of S-20 cathodes decreases rapidly between 4000 and 9000 \(\AA\) and that therefore the optimum compromise between light absorption and escape depth of photoelectrons lies at much smaller film thicknesses for blue light than for red and infrared light. It has also been discussed previously that, because of this variation in light absorption, the red response of S-20 cathodes can be improved by making the cathode thicker up to the point where
the escape depth again sets the limit. There are indications that the response in the range above 8000 Å can be increased by factors of two to three if the thickness is optimized for this range. However, this improvement is always obtained at the expense of response at shorter wavelengths.

In the tubes described in the previous section, the whole activation process was in many cases repeated one or more times to produce a thicker cathode and higher red response. In all the cathodes shown in Fig. 2, the red response has been optimized in this manner. The effect of increased thickness, as distinct from a change in cathode composition, is readily recognized by two effects: First, the sensitivity to blue light remains constant with the light incident from the vacuum interface but decreases with the light incident from the glass interface. Second, the sensitivity increase near the threshold is a quantitative rather than a qualitative one. In other words, with increasing thickness the threshold is not significantly shifted to longer wavelengths but the sensitivity at a particular wavelength is increased. Referring to Fig. 2, this means that a curve such as that of S.1367 cannot be changed into that of S.1516 simply by making the cathode thicker.

An experiment was made in tube S.1549 to produce a relatively thick cathode for high infrared response by evaporating a greater amount of Sb onto the oxidized Sb-substrate rather than by repeating the whole activation process as in earlier tubes. The Na-Sb alternation produced again a Na₂K₅Sb cathode of relatively high red response, but the final S-20 cathode had only 140 μA/l sensitivity and low infrared response, although it was very thick as shown by low blue response with light incident from the glass interface. This experiment has to be repeated to evaluate its significance, but it could mean that repeated processing is superior to processing a thicker initial Sb-layer.
C. Conclusions

The results described in Section (B) are so incomplete that most conclusions at this stage are necessarily of a tentative character. However, the following statements can be made with some confidence:

1. Sb-films deposited from an SbPt-bead differ from those deposited from an Sb-bead in that:
   (a) They consist of smaller particles which form a continuous film of relatively low resistivity.
   (b) They can be oxidized by either glow discharge in oxygen or by exposure to oxygen at 160°C.
   (c) Being continuous, they transmit less light for equal amounts of Sb deposit per square centimeter.

2. S-20 cathodes formed on oxidized Sb-substrates with additional Sb over-layer have higher over-all sensitivity, but not necessarily longer threshold wavelength than cathodes on unoxidized Sb.

3. Alternation of Na and Sb at 220°C produces Na₂KSb cathodes with relatively high red response.

4. Na-Sb alternation at 220°C in combination with oxidized Sb-substrate appears to be beneficial for high infrared response. As long as the physical and chemical effects of these processing features are not understood, it is very likely that they are only one way, but not the only way, to achieve the extension of threshold.

5. The infrared response of an S-20 cathode can be increased at the expense of shorter wavelength response by increasing the film thickness. This may be of considerable practical interest where sensitivity beyond 8000 Å is essential. However, it must be remembered that an improvement above 8000 Å, i.e., in a range of very low absolute quantum efficiency, is
bought at the expense of a loss of sensitivity in a range of very high quantum efficiency. Hence, for use above 8000 Å, it would probably be advantageous to evaluate cathodes by comparing the sensitivity through an infrared transmitting filter (such as the 2540 glass filter) rather than by comparing μa/l values.
II. MOLECULAR BEAM FABRICATION OF S-20 CATHODES

A. Cathode Fabrication

During this quarter, two attempts have been made to fabricate S-20 cathodes by the molecular beam method. The results of these experiments, which will be reported below, have served as a stimulus for the development of new techniques for producing calibrated molecular beams.

The apparatus for the simultaneous deposition of Na, K, Cs and Sb has been described in detail in previous reports. It consists of glass molecular beam guns for the alkali metals which are heated by external ovens and a metal gun for antimony which is heated by an internal heater. The guns are all directed towards a quartz crystal oscillator which is used to measure the mass of material deposited from each molecular gun. Cathodes are produced on substrates which are moved in front of the quartz crystal oscillator. A schematic diagram of this apparatus, which has been previously presented is shown in Fig. 3.

As a result of improvements made primarily in the quartz crystal mount as described in the previous report and the incorporation of a temperature controlled enclosure for the reference quartz crystal oscillator, it has been possible to obtain accurate measurements of the deposition rates. The improved performance has been observed (1) in the stability of the crystal frequency with no molecular beam incident and (2) in the sharp change in slope of the frequency vs time plot when the molecular beam apertures are opened.

The procedure in the two attempts to produce S-20 cathodes was to adjust the rates of deposition of Sb, Na and K as measured by the quartz crystal oscillator so that they are in a ratio of 1:3.77:3.19 corresponding to an atomic ratio of 1 Sb to 2 Na to 1 K. The major problem in these
experiments is the setting of these rates. This has been done by measuring the rate of deposition of each constituent in sequence for six minutes and then adjusting the rate as required. This procedure was continued until rates reasonably close to those required were achieved.

In the first experiment the following rates were achieved:

\[ \text{cps in 6 min.} \]

<table>
<thead>
<tr>
<th>Element</th>
<th>Rate</th>
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<tbody>
<tr>
<td>Sb</td>
<td>34</td>
</tr>
<tr>
<td>Na</td>
<td>11</td>
</tr>
<tr>
<td>K</td>
<td>8</td>
</tr>
</tbody>
</table>

The Sb rate was slightly too high, but because there was the possibility of running out of materials, the Sb rate was reduced slightly by an unknown amount and a simultaneous deposition was made on a clean substrate.

The white light photoemission rose continuously with time for 7.5 minutes and then went through a peak. The constituents were then each deposited sequentially. It was found that each component caused the photoemission to go through a peak. When all beams were stopped, the photoemission rose slightly and reached a stationary value. The sequential depositions were continued until no further increase was obtained. The material deposited sequentially made up less than 10% of the total amount of material deposited. An increase in the sensitivity was observed of only a factor of 2 as a result of the sequential deposition. The sensitivity of this cathode was 10 \( \mu \text{amps/lumen} \). Upon deposition of cesium and additional Sb sequentially, the sensitivity was increased to 30 \( \mu \text{amps/lumen} \). Measurements of the response with a red filter showed that the cathode which had been formed was clearly a multialkali cathode although of low sensitivity.

In the second experiment, the molecular beam rates were adjusted to the following values:
As before, the photoemission rose steadily but when the sensitivity reached a value of about 1 μamp/lumen the Sb gun temperature controller started to oscillate. This was seen as an oscillation in the cathode sensitivity with time. It was unfortunate that this experiment had to be terminated because the beam rates had been very close to the values required to produce Na₂K₃Sb.

In each of these experiments more than three hours were required to adjust the molecular beam sources to give the desired deposition rates. There appear to be two causes for this: (1) the long time constant of the alkali guns, and (2) the rates of deposition from the sodium and potassium molecular beam sources were considerably below those expected from calculation from the apparatus geometry and vapor pressure data. The first problem is inherent in the fact that the beam sources are made of glass and are heated externally. The low thermal conductivity of the glass limits the rate at which the molecular beam apparatus comes to equilibrium. The response to the small adjustments in the oven chamber temperature are especially slow.

The origin of the second difficulty is not completely understood. The deposition rates as measured by the quartz crystal are equivalent to those expected if the oven temperature were 25° too low, that is, they are four times lower than expected. Furthermore, the deposition rate is less dependent on temperature than expected from the calculations. A large error in temperature measurement seems unlikely. However, since the temperature of the external glass wall rather than that of the alkali metal is measured, a small error cannot be ruled out. There is evidence
of a chemical reaction between the glass and the alkali metal as seen in the discoloration of the glass. The products of this reaction (alkali oxides, Si) are expected to have low vapor pressures. Since free alkali metal has been visible in each of the guns during the course of these experiments, the chemical reaction should have little effect on the deposition rate. Another possible, though unlikely, cause for the low deposition rate is related to the desorption of alkali metal. If it is assumed that in equilibrium only one in four of the incident atoms are permanently retained, the low rates can be explained. Previous results indicate that all incident alkali atoms are retained especially when deposition is on an antimony-rich substrate.

In these experiments, alkali metal deposition immediately following an Sb deposition did not show any variation in rate as a function of time. The possibility of this effect will be studied further during the next quarter since if it does occur, the results measured with the quartz crystal oscillator may not be applicable to the cathode formation.

B. Improved Apparatus

The results of the previous experiments indicated that two improvements in the molecular beam forming apparatus would be useful. First, an oven with a shorter time constant would be advantageous; second, the excellent stability of the quartz crystal oscillator suggests that the oscillator output be used to control the power to the oven heater directly, rather than to control the oven temperature assuming, of course, that all incident atoms are adsorbed. Preliminary experiments in both of these areas have been made during this quarter.

Fig. 4 shows the experimental tube containing the new molecular beam source. The molecular beam gun consisted of a 1/4'' diameter copper
FIG. 4
tube across which a .001" thick nickel foil had been brazed. Alkali metal had been distilled into the tube and the tube had been pinched off. After the tube was baked out, a hole was punched in the copper tube and the tube was heated with a tungsten coil. The rate of deposition from this source was measured by means of the quartz crystal oscillator mounted on the upper end of the tube.

The results of this experiment showed that the time constant of this source, although shorter than those of the glass guns, was still several minutes. This is the result of the high thermal capacity of the copper tube, and the poor coupling of the tube to an external heat sink. The coupling to the heat sink cannot, however, be improved because the inefficient coupling to the heater coil would make the required input power in the present design too high. Molecular beam sources are presently being constructed to circumvent these difficulties.

The preliminary experiment on automatic control of the deposition rates was carried out in a bell jar using the circuit shown in block diagram form in Fig. 5. A simple resistance wire evaporator was used in this experiment to simulate a molecular beam source with a fast response time. The circuit was designed so that the evaporator received power at a rate which maintained the deposition rate as measured by a quartz crystal oscillator at a preprogrammed value. The output of the oscillator was impressed on a General Radio Type 1142A frequency meter which produces a dc output proportional to the frequency. At constant deposition rate, this dc voltage should increase with time at a constant rate. The output of the frequency meter was amplified and fed into one input of a differential amplifier. This was compared to a steadily increasing dc voltage which was generated by a rotating potentiometer and a battery. The difference be-
CIRCUIT FOR AUTOMATIC CONTROL OF EVAPORATION RATE

FIG. 5
between the differential amplifier inputs was used to trigger a silicon controlled rectifier control circuit which controlled the power to the evaporator so that the inputs of the differential amplifier were maintained at approximately equal values. The frequency meter is thereby made to follow the potentiometer. The deposition rate was controlled by varying the voltage across the motor-driven potentiometer.

Preliminary experiments with this circuit showed that it worked well over a large range of evaporation rates. It is limited primarily by the stability of the quartz crystal oscillator. Further experiments will be performed with this circuit when a faster molecular gun is developed since it is believed that with our present guns the temperature will lag too far behind the control signals.
III. ELECTRON AND X-RAY DIFFRACTION STUDIES

A. Molecular Beam Electron Diffraction Tube

Results given in the 8th and 9th Quarterly Reports\(^2,3\) of this project have shown that the formation of S-20 photocathodes employing sequential deposition from molecular beams is kinetically inhibited at room temperature and that it would therefore be desirable to heat the substrates. In the case of the usual processing procedures, provisions for heating the substrate would not present any serious problem but, in combination with the present apparatus, the means of heating the substrate are limited.

The failure of a method using a resistively heated substrate was reported previously.\(^4\) During the past quarter, an attempt was made to heat the substrate using a projector lamp containing a parabolic mirror for enhancing the focus of the emitted light. However, the difficulty in reproducing the lamp to substrate distance coupled with the fact that the substrate is a composite of glass and metal made the control and measurement of the temperature virtually impossible.

As a result, no attempt was made to draw any conclusions from the data gathered in this experiment although, in general, the effect of heating was to lower the sensitivity of what were already extremely poor cathodes. Perhaps the greatest drawback in this scheme is that the heat transfer characteristics of the glass and metal portions of the substrate are so different that it would not be correct to attempt to make a correlation between the photoemission measured on glass and the transmission electron diffraction


pattern obtained through the carbon-coated copper mesh.

As has been mentioned in previous reports, the dual substrate problem is eliminated by the use of reflection electron diffraction. Continued progress in the design and construction of such an apparatus is discussed below.

B. Reflection Electron Diffraction

In the previous report of this project, several reasons were advanced to explain the failure to observe an electron diffraction pattern in a tube employing the reflection technique. Several modifications were made in the tube in order to overcome these and other objections. These modifications were as follows:

1) Incorporation of a 3/16"x.015" aperture between the anode of the electron gun and the substrate. The aperture was placed about 1" from the substrate and was mounted on a 1" long x 3/4" dia. cylinder. The resultant loss in total beam current caused by this limiting aperture could, in fact, be compensated for by reducing the bias on the first grid. The aperture serves not only to reduce the size of the beam which strikes the crystal but also to lessen the background radiation by preventing secondary electrons from the walls of the tube from striking the phosphor screen.

2) Because it was virtually impossible to align the rest of the gun with the new limiting aperture, a magnetic alignment coil was used for this purpose. Satisfactory control was achieved by placing this coil about an inch behind the end of the anode of the electron gun.

3) A moveable beam stop was placed about 3/4" from the phosphor screen. This consisted of a 2-mm strip of nichrome attached to an L-shaped arm extending out of the main pump manifold. The end of the arm in the manifold had a soft iron core which allows the position of the beam stop
to be controlled by an external magnet. The use of the beam stop enhances
the contrast considerably since the haloation due to the internal reflection of light produced in the phosphor screen by the primary electron beam
in the face plate is a prime contributor to the background.

(4) An optically polished quartz substrate with essentially the same geometry
as before was used. However, great care was taken to ensure that all
portions of the substrate which would not be covered by the test material
were covered by a conducting layer of silver.

(5) An evaporator was incorporated so that the test material could be deposited
in situ. Since our test material in this case was gold, such precautions
against oxidation were not necessary.

The electron diffraction unit was placed in operation without baking
the tube but only after an ambient pressure of less than $10^{-6}$ Torr was obtained.
Bake-out was not considered to be necessary since ultra-high vacuums were not
required for these tests. Preliminary tests of the electron optics showed
that the 15-mil aperture reduced the background considerably and, in addition,
there appeared to be only a minimal amount of diffuse scattering from the
substrate when it was positioned in the beam. In addition, no charging effects
were noticed. These latter two effects had been severe in the previous experiment
where the limiting aperture was absent. A weak image of this aperture
was observed on the phosphor screen, but this was not objectionable.

A layer of gold was evaporated onto the substrate whose thickness
was later estimated to be in excess of 200 Å on the basis of the light trans-
mittance of the gold evaporated onto the walls of the tube.

By adjusting the angular setting of the substrate with respect to
the beam, it was possible to observe a diffuse diffraction pattern at an
accelerating voltage of 20 kV. The pattern was sharpened considerably by
raising the accelerating potential to 35 KV and optimized by transver-
scanning of the substrate in order to find the best diffraction region. At
this point, a final angular adjustment was also necessary. This could be
accomplished by moving the substrate with the micrometer stage or by adjust-
ment of the electron beam with the magnetic coil.

A reflection diffraction pattern of the gold layer is shown in
Fig. 6a. Pains were taken to optimize the sharpness of the pattern. For
comparison purposes, a transmission electron diffraction pattern of a 200 Å
layer of gold is shown in Fig. 6b. Both patterns were taken at an accelerating
potential of 35 KV. The transmission pattern was obtained using a standard
copper mesh substrate which temporarily replaced the reflection substrate.
Clearly, the diffraction lines are broader and the background higher in the
reflection diffraction pattern. However, these are drawbacks inherent in
the method. One reason for the diffuseness of the lines is the width of the
region over which diffraction takes place. In this case, it can be as great
as 2 mm. It should be possible, however, to reduce this width by a factor
of 2 or more. In addition, it might be pointed out that the transmission
diffraction pattern of gold shown here is not nearly as sharp as some that have
been obtained for antimony or the alkali antimonide photocathodes. (See
Fig. 1e.) Thus, one might expect that the reflection patterns of these mate-
rials might be sharper than that of gold also.

This experiment has demonstrated the basic soundness of the appara-
tus. However, the quality of the diffraction patterns at present is such
that they probably would not be very useful except for identifications.
Unquestionably it will be necessary to obtain sharper lines if useful informa-
tion is to be obtained. This, in fact, can be accomplished by operating at
higher potentials. The next step in this experiment will be to examine actu
FIG. 6
photocathodes and compare the reflection photographs with those obtained by transmission techniques. In addition, the possibility of reducing the diffuse-ness of the diffraction lines by redesign of the substrate will be examined.

C. Electron Diffraction Investigation of Ag-Cs-O Cathode

As a result of studies in this laboratory, a great deal has been learned about the structure of the binary and ternary alkali antimonide photoemitters. In contrast little or nothing is known about the composition or structure of the Ag-Cs-O (S-1) cathode. This cathode, which also happens to be the oldest of the practical photoemitters is of interest because of its relatively high infrared response. In the past, many workers have assumed that the cathode is a composite of silver and Cs₂O but there is no direct evidence for this and no good explanation as to why this rather peculiar combination displays its observed photoelectric behavior.

This examination of the Ag-Cs-O cathode was delayed several months because of difficulties encountered in the repair and construction of the apparatus and because of higher priority commitments to other aspects of this project. The conventional electron diffraction apparatus described in the Fifth Quarterly Report of this project was used for this experiment and normal processing procedures for S-1 cathodes were followed as far as possible. Diffraction patterns were always taken after: (1) the evaporation of the silver layer, (2) oxidation of the silver, and (3) addition of cesium to a maximum value of photoemission. If circumstances warranted, the processing could be stopped at some other point and the diffraction pattern examined.

A total of four cathodes were prepared. All of them had low photosensitivities, with the highest being four microamps/lumen measured relative to a standard tungsten lamp and the other three being in the 1 to 1.5 microamp/lumen region. In all instances however, the response using a red 2408 Corning filter was
between 60 and 70 percent that of the unfiltered value; a response which is
typical of an S-1 cathode. The reasons for the low sensitivities are not
known although it is usually necessary for one to alter the processing
procedure somewhat when the tube geometry is changed. In addition, it was
found that the semi-permanent source of oxygen (a side arm containing HgO)
on the vacuum system was very nearly exhausted making it difficult to gen-
erate a sufficient pressure of oxygen for normal glow discharges after the
processing of the second cathode.

Because of the poor sensitivity of the cathodes, it is difficult
to assess the relationship of the observed diffraction patterns to those
that might be observed at normal photosensitivities. However, because the
relative response of these cathodes to red and white light is what is
expected for S-1 cathodes, it would seem likely that what is seen in these
diffraction patterns would in part be seen in the patterns of normal cathodes.
Since some variation in the diffraction patterns of the different cathodes
was seen at various steps in the processing, it seems logical to discuss
them as a function of the processing steps.

The first step in all cases was to evaporate a layer of silver to
a light transmittance of 50 percent (about 100 Å thick). In three cases
the diffraction pattern was what might be expected from a thin evaporated
layer of silver. There were some differences in the sharpness of the lines
indicating perhaps that the crystallite size varied somewhat from sample
to sample. In one case, however, a very weak and poorly defined pattern
was observed. This was later shown to be the result of a poor substrate
(substrate 3, a 1 microamp/lumen sample) and had nothing to do with the
crystallinity of the layer. Because of this, diffraction data from this
substrate was not considered in this discussion although the behavior of
the cathode itself is. Thus it appears that the final sensitivity was not radically affected by any initial crystallinity of evaporated silver layer. Oxidation of the silver was carried out in a glow discharge in oxygen. The process of oxidation was followed by measuring the light transmittance of the glass portion of the substrate, the assumption being that the oxidation is complete when nearly 100 percent light transmittance is achieved. Only in the case of substrate 1 was 100 percent light transmittance observed while in the case of the other three, final values of 70 to 80 percent light transmittance were observed only after several minutes in the discharge. In these cases, continued discharge either had no effect or resulted in a slight decrease in the transmittance. In contrast, oxidation of substrate 1 was complete in a few seconds. Table A gives the interplanar spacings obtained from the electron diffusion patterns of substrates 1, 2, and 4 after the glow discharge step. All of them show the presence of silver oxide, Ag₂O. It is interesting to note that the presence of silver is noted only in the pattern of substrate 1 and yet this was the only substrate in which the light transmission measurement indicated that the oxidation was complete. In contrast, no diffraction lines of silver are seen in the other three samples where, by the same reasoning, one might expect silver to be present. The possibility exists then that not all of the silver has been oxidized but rather has been converted into an amorphous-like or extremely small crystallite size phase. This process might be brought about by continued ion bombardment heating the substrate so that initially formed oxide is decomposed leaving a deposit of silver in the above condition. This would also explain the decrease in light transmittance which was observed in some cases on continued action of the glow discharge. It is also interesting to note that the substrate which behaved normally in the glow
TABLE A

ELECTRON DIFFRACTION PATTERN OF EVAPORATED SILVER FILMS WHICH HAVE BEEN OXIDIZED BY GLOW DISCHARGE

<table>
<thead>
<tr>
<th>Substrate 1</th>
<th>Substrate 2</th>
<th>Substrate 4</th>
<th>Identification*</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>d/n</td>
<td>I</td>
<td>d/n</td>
</tr>
<tr>
<td>w</td>
<td>2.678</td>
<td>s</td>
<td>2.646</td>
</tr>
<tr>
<td>vw</td>
<td>2.337</td>
<td>w</td>
<td>2.380</td>
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<tr>
<td>vw</td>
<td>2.00</td>
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</tr>
<tr>
<td>vvw</td>
<td>1.438</td>
<td>vwh(b)</td>
<td>1.423</td>
</tr>
<tr>
<td>vvw</td>
<td>1.238</td>
<td>vw(b)</td>
<td>1.061</td>
</tr>
</tbody>
</table>

All patterns recorded at 35.0 KV

* Data taken from ASTM X-ray Powder Data File
discharge was the one which yielded the highest photosensitivity.

The processing side arm was heated to 160°C and cesium was added until a maximum value of photoemission was obtained. The diffraction patterns of the cathodes thus obtained are given in Table B. The patterns are not all the same, with the first three being weak and diffuse while the fourth contained many more lines and was considerably sharper. Crystalline silver was observed only in the first substrate (where it was known to be present) and perhaps in the fourth substrate. If the latter case is so, this would lend credence to the proposal that the S-1 cathode is formed by the reaction

\[ \text{Ag}_2\text{O} + 2\text{Cs} = \text{Cs}_2\text{O} + \text{Ag} \]

An alternative possibility in which no separate phase of silver would be observed is the formation of a discrete ternary oxide or a solid solution of the binary silver and cesium oxides. In this respect, a line corresponding to one of the two equally strongest lines of Cs₂O is observed in all four diffraction patterns. In the case of substrate four, a reasonably good fit of its diffraction pattern with that of Cs₂O is observed. Lines corresponding to unknown phases, one of which might be silver, are also present.

The conclusions to date would be that the S-1 cathode most likely contains Cs₂O or a phase closely related to it. Conflicting evidence exists as to whether silver is present in the final cathode as an elemental phase. However, the data obtained for the silver oxidation step indicates that silver could be present as an amorphous or poorly crystalline phase and thus not detectable by diffraction techniques. Since all of the data was taken from rather poor cathodes, it must be realized that the above data may not
<table>
<thead>
<tr>
<th>Substrate 1</th>
<th>Substrate 2</th>
<th>Substrate 4</th>
<th>Identification</th>
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<tbody>
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<td>I</td>
<td>d/n</td>
<td>I</td>
<td>d/n</td>
</tr>
<tr>
<td></td>
<td>vvw(b) 2.973</td>
<td>vvw 2.369</td>
<td></td>
</tr>
<tr>
<td></td>
<td>w 2.940</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>m 3.345</td>
<td>Cs₂O 3.433 (100)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>w(b) 2.940</td>
<td>Cs₂O 2.911 (100)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.078</td>
<td>Ag 2.360 (100)</td>
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<tr>
<td></td>
<td></td>
<td>1.789</td>
<td>Cs₂O 2.124 (25)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.410</td>
<td>Cs₂O 1.945 (20)</td>
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<td></td>
<td></td>
<td>1.344</td>
<td>Ag 2.040 (38)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.344</td>
<td>Cs₂O 1.688 (10)</td>
</tr>
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<td></td>
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<td>1.200</td>
<td>Ag 1.231 (26)</td>
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<tr>
<td></td>
<td></td>
<td>1.197</td>
<td>Cs₂O 1.201 (10)</td>
</tr>
</tbody>
</table>

All patterns recorded at 35.0 KV

* Data taken from ASTM X-ray Powder Data File
represent the conditions of a normal cathode. These experiments will be repeated with a view to preparing cathodes which are more representative of standard cathodes in their photoelectric behavior.

D. Crystallite Size Studies

During the past quarter a considerable amount of experimental data was taken in order to determine the crystallite size of Na2KSb, (Cs)Na2KSb, K2CsSb and Cs3Sb cathodes in different degrees of activation. This data is now in the process of being analyzed and will be presented in the next report of this project.
IV. FIELD INDUCED PHOTOEMISSION

Development of a new technique of cleaning and diffusing doping impurities into p-type germanium to produce n-p junctions for field induced photoemission was begun during this quarter. Past experience has shown that the surface, through which doping impurities are introduced into a crystal of silicon or germanium, plays an important role in determining what impurities are introduced and their distributions. If there are oxides on the surface, they may react chemically with the crystalline material. In some cases, the reaction at the surface is important in that it provides a convenient method of introducing impurities, i.e., the diffusion of phosphorus into silicon. In this case, the phosphorus pentoxide which forms on the surface acts as a source. There may also be oxides of various undesirable elements present which may react with the surface freeing the elements for subsequent diffusion.

If the surface of the crystal is covered with its own oxide, any doping impurity source introduced will have to diffuse through or interact with the oxide before reaching the crystalline material. In the case of very shallow junctions, this oxide layer, which is almost always present, undoubtedly plays an important role in determining the depth of the junction, the impurity profile, and the impurity surface concentration.

Since it is difficult to ascertain how much and what kinds of materials are on the surface, it is then desirable to remove them as completely as possible before diffusing.

The apparatus which is shown in Fig. 7 was designed with the above considerations in mind. Firstly, the surface is etched using the conventional HCl vapor etching technique exposing a surface free of foreign substances. Since the ambient is free of oxygen at all times, there is no danger of oxide

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formation. Secondly, impurities are introduced for diffusion.

The crystal sample rests on a block of similar material which is heated by an RF generator. Heating the sample in such a way prevents the introduction of foreign materials which might be emitted from the walls of the quartz reaction tube which, in the conventional diffusion apparatus, is also heated to a high temperature. It has long been suspected that the hot quartz walls have been the principle source of impurities in the past. This method of heating the sample will allow for a test of this hypothesis.

In the past, our impurity source, for n-type diffusion in germanium, has been red phosphorus which was sublimed in one region of the furnace and carried by hydrogen over the sample which was placed in another region of the furnace. The concentration of phosphorus above the sample was very difficult to control. As a result, the germanium surface became pitted due to microalloying in regions on the surface exposed to excessive amounts of phosphorus. These microalloyed regions are inevitably regions of high concentration. If the p-n junction is close to the surface, they may extend to the junction giving rise to soft i-v characteristics. If the junction is shallow but the alloyed regions do not extend to the junction plane, then soft i-v characteristics will arise due to abnormally steep impurity gradients at the junction. For deep junctions, the effects of the alloyed region are averaged out; hence, they have little effect on the i-v characteristics.

We propose to overcome this difficulty by using instead PH$_3$ as a diffusion source. Being a gas, PH$_3$ may be introduced into the reaction chamber in controlled amounts where it will decompose at the hot germanium surface freeing phosphorus for diffusion. This procedure should eliminate agglomerations of phosphorus on the surface. This leads one to believe
that the microalloying problem might be alleviated.

The operation of the apparatus which is now completely built is obvious from the diagram. Very preliminary experiments have been made to test the performance of the hardware involved. Experiments on etching and diffusion will commence shortly.
PROGRAM FOR NEXT QUARTER

(1) Study Sb-films as a function of source material.
(2) Attempt to define and understand the effects of oxidized Sb-substrate and of Na-Sb alternation at high temperatures.
(3) Continue the experiments on the formation of S-20 cathodes by the simultaneous deposition from molecular beams using the tube already built.
(4) Examine the sticking and desorption of alkali metals during cathode formation.
(5) Further develop molecular beam sources with fast response times.
(6) Further improve the reflection electron diffraction apparatus.
(7) Further electron diffraction studies of S-1 cathodes.
(8) Complete study of crystallite size in photocathodes.
(9) Initiate study of zeolite alkali ion sources for photocathode formation.
(10) Resume low energy electron diffraction studies.
(11) Investigate vapor etching and diffusion of p-n junctions for field induced photoemitters.
Man-hours expended for the period 1 July 1964 to 30 September 1964:

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<tr>
<td>G. A. Morton</td>
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<td><strong>Members of Technical Staff</strong></td>
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<tr>
<td>C. R. Fuselier</td>
<td>409-3/4</td>
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<td>W. H. McCarroll</td>
<td>418-1/4</td>
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<td>J. O. Schroeder</td>
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<td>R. E. Simon</td>
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<td>A. H. Sommer</td>
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<td>G. O. Fowler</td>
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<td>R. L. Rodgers</td>
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**TOTAL MAN-HOURS** 2088
UNCLASSIFIED

1. Processing of S-20 photocathodes
2. Molecular beam fabrication of S-20 photocathodes
3. Electron and x-ray diffraction studies
4. Field induced photoemission

Title: Research Study for Increasing the Sensitivity of Photomultipliers

Research by W. E. Atwood, W. E. McFarland, C. R. Fosler
Quarterly Technical Report No. 11 for
1 July 1966 - 30 September 1966.

AD

RCA Laboratories, Princeton, N.J.

RESEARCH STUDIES FOR INCREASING THE SENSITIVITY
OF PHOTOMULTIPLIERS, Research by W. E. Atwood,
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