NASA CR 72077

THIRD QUARTERLY REPORT

STUDY OF THIN FILM LARGE AREA PHOTOVOLTAIC SOLAR ENERGY CONVERTER

F. A. SHIRLAND, J. R. HIETANEN, AND W. K. BOWER

PREPARED FOR

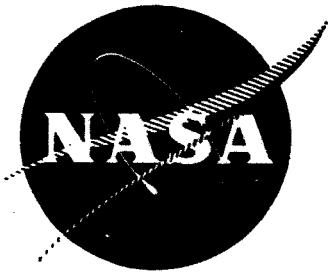
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-8502

AUGUST 24, 1966

ELECTRONIC RESEARCH DIVISION

CLEVITE CORPORATION



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STUDY OF THIN FILM LARGE AREA PHOTOVOLTAIC SOLAR ENERGY CONVERTER

Third Quarterly Report
April 25, 1966 through July 24, 1966

Contract No. NAS 3-8502

SUMMARY

The major emphasis during this reporting period has been placed on the fabrication of standard process plastic substrate cells and the determination of their stability under various conditions of temperature and humidity. Average cell efficiency for the period was recorded at approximately 5.1% but there is some question as to the accuracy of this figure because of difficulties with calibration of the thermopile used to measure light intensity. Difficulties with the conductive coating of the plastic substrate, with adhesion of the CdS film, and with attachment of the cover plastic have been encountered and have in general been satisfactorily dealt with. A process has been developed to cover the negative electrode tab with gold plating over a copper undercoating. This has improved cell appearance and the solderability of the tab.

The efforts to produce thinner CdS films which yield high efficiency cells have still not been successful. Difficulties in this area have centered around the tooling used in the vacuum evaporator to secure thinner films at higher substrate temperatures. The use of chromium as a substrate coating in place of the zinc, while yielding good low resistance electrical contacts, has not given high efficiency cells and will therefore not be evaluated further. Instead attempts will be made to develop a copper-zinc alloy on the metallized plastic substrate.

STANDARD PROCESS LINE

Cell Fabrication

Table I summarizes the cells fabricated on the Standard Process Line during this period. A total of 161 cells were fabricated, yielding an average efficiency of 5.1%. These cells were all of the plastic substrate construction. A number of problems associated with the fabrication of this cell design were defined and solved during the quarter. Chief among these were (1) poor adherence of the metallized coating on the plastic substrate (2) poor adherence of CdS film to the metallized coating (3) incomplete attachment of the cover plastic to the finished cell. These are discussed in more detail in following sections of this report.

Toward the end of the period it was found that the thermopile being used to measure the intensity of the light for testing the solar cells (as well as a secondary standard silicon solar cell used for the same purpose) was off-calibration with the result that cell efficiency figures were approximately 6 - 7% high. A careful review of the cells tested throughout this period indicates that this off-calibration probably occurred during the first month of the quarter. The data summarized above are uncorrected for this error.

TABLE I

STANDARD PROCESS LINE CELLS - THIRD QUARTER

Construction	Month	Total Cells	Scrap Cells	Efficiency* Min.	Efficiency* Max.	Efficiency* Avg.	Power/Wt. W/lb
Plastic Substrate (Mylar-epoxy cover plastic)	1st	62	4	4.0	6.4	5.2	98
	2nd	59	3	4.3	6.5	5.0	95
	3rd	40	7	4.0	6.2	5.2	103
	Total	161	14	4.0	6.5	5.1	98

* before recalibration. See section on Cell Testing.

Processing Problems

Most of the effort in this period has been spent uncovering and defining faults in the component parts and processing procedures of the plastic substrate cell and then developing means of eliminating these faults. Poor adhesion of the CdS film to the substrate has been intermittently present from the beginning of this developmental program. In this period this problem has been broken down into several individual factors, each has in turn been dealt with and reasonable solutions developed which appear to have taken care of the major part of this problem. First, adhesion of the silver Pyre-ML coating to the Kapton substrate has been improved by the use of a simplified Kapton cleaning process and an improved drying and curing operation. Most of the difficulty previously experienced appears to have been due to incomplete removal of the solvent from the silver Pyre-ML layer prior to the curing of the Pyre-ML. When the curing step is started prior to complete removal of the solvents a skin forms which traps residual solvents and leads to incipient blisters and areas of poor adhesion. This problem was partially solved by extending the drying period of the uncured Pyre-ML coating to 15 minutes at 150°C before the final cure of 30 minutes at 250°C. However, a few poorly adherent metallized layers are still being produced even with this process. A visual inspection initiated at this stage of the process has eliminated nearly all these poor substrates from further processing. This inspection consists of observing the layer from the underside of the Kapton. Poorly adherent films are characterized by a mottled appearance.

Further difficulties with the substrate coating operation have centered around the obtaining of a uniform coating thickness and the development of a proper Kapton substrate cleaning process. A simplified cleaning procedure of the substrate, consisting of an N, N-Dimethyl Formamide scrubbing using a soft paper towel followed by a drying step in which the substrate is scrubbed with another paper towel and finally a 5 minute air drying step at room temperature, has been satisfactory in eliminating the latter difficulty. The coating thickness uniformity has been and still is primarily a function of operator skill in spraying the silver Pyre-ML mixture onto the substrate and in controlling the spray mixture viscosity. This item has yielded to better operator training and supervision as well as to improved inspection techniques.

Several separate causes of poor adherence between the metallized layer and the CdS film have been considered. The first of these is poor zinc plating on the metallized layer. Careful attention to the plating bath composition, purity of materials, and plating conditions has resulted in vastly improved control over this process. Systematic analysis of plating bath composition and periodic bath filtering and electrode cleaning have also helped in securing improved control over the process.

A glow discharge step has been introduced in the vacuum evaporation process immediately prior to evaporation. This appears to have made an improvement in the adhesion of the CdS layer to the substrate. Whether this results in a removal of contaminated zinc from the surface of the zinc coating or merely a removal of absorbed gas atoms from the surface is not known; however, a number of controlled experiments using the glow discharge has shown a marked improvement in CdS film adhesion.

Difficulties with the cover plastic attachment process have been associated with voids in the adhesive layer. These are areas at the front surface of the cell where there has either been an insufficient volume of adhesive to fill the space between the cover

plastic and the cell barrier or where gases have been trapped inside the package during the cover plastic attachment process. These voids result in interfaces which impair the optical coupling between the cover plastic and the barrier and thus result in very high reflection losses. They probably also represent localized areas that would be more subject to moisture penetration, oxidation or other chemical reaction. This effect is noticeably more severe when Kapton is used for the cover plastic in place of the Mylar.

A solution to this problem appears to result from careful control of the thickness of the epoxy adhesive used to attach the cover plastic, the grid thickness and the thickness of the gold filled epoxy used to attach the grids to the cell, and the use of the proper lamination procedure. To date this adhesive has been applied by spraying on the underside of the cover plastic by diluting with solvent and using an air brush. The amount of adhesive required depends on the grid $\neq$ Au epoxy thickness. If not enough adhesive is applied a void filled package results; if too much is applied the epoxy spreads out during the attachment process and covers the lead tabs and other portions of the cell. Work has been started to see if commercial suppliers cannot apply the epoxy adhesive to the cover plastic with better control over the thickness. So far the commercial suppliers utilizing roll coating techniques have had just as much difficulty in meeting the thickness specifications as we have had with the spray process. In the meantime, the difficulty is being dealt with by the employment of a rigid inspection of the pre-coated plastic prior to the lamination step and the careful application of the grid to the cell before the cover plastic attachment step. Work is just underway on the investigation of the lamination procedure to insure that no residual gases are trapped inside the package before the cover plastic attachment is made.

CELL DESIGN

Copper Substrate

The fabrication of the copper substrate cell on the laboratory fabrication line was discontinued several months ago because of poor CdS adherence to the copper and cracking of the copper substrate during subsequent cell processing. As reported earlier, cell cracking was found to be associated with the hardness of the copper and a suitable annealing step was found to eliminate this cracking. The poor adherence was found to be due to the presence of a stabilizing agent on the surface of the copper foil used (which was fabricated primarily for printed circuit board application). During this period sources of copper supply have been evaluated in order to secure a raw material free from the stabilizing agent and in a satisfactory range of hardness. A number of 3" x 3" cells were produced in this period using a new supply of copper foil for the substrates. These cells reached efficiencies of 5.8 to 7.2% with an average of 6.4% (before recalibration). This is appreciably higher than experienced with plastic substrate cells produced at the same time.

Elongation measurements of this material as well as on other lots of material that were not satisfactory have resulted in a specification requiring copper foil with elongations of greater than 5%.

Chromium Underplate

Chromium was investigated as a substitute for the electroplated zinc on the silver Pyre-ML layer. It was hoped that chromium would give an improved low resistant ohmic contact between the CdS layer and the substrate. The major disadvantages of zinc (high vapor pressure, low melting point, etc.) were thus hoped to be avoided. The Cds films using a chromium underlayer displayed very excellent adhesion and gave cells with current-voltage characteristic curves displaying high fill factors. Approximately 30 cells were produced using a high purity chromium. A cross section of the results of this production is given in Table II. These cells are characterized by low currents and their resulting efficiencies are in the 3 to 4% range. On the assumption that a well structured CdS film had been grown onto the chromium (an assumption made on the basis of film adherence and surface texture observed under a miscroscope) a number of efforts to modify the barrier formation conditions on the films were made. These modifications were unsuccessful in increasing the current and efficiency levels. Another suspected possibility for the low observed output may be that the chromium acts as a "poison" forming trapping centers at unfavorable levels. In any case, it appears that the use of chromium is unsatisfactory and further efforts with this material are not planned.

Thinner CdS Films

Thinner CdS films are desirable in order to reduce the overall thickness and weight of the CdS thin film solar cell and to reduce cracking developed in the CdS films due to thermal mis-match between film and substrate. Present CdS film thicknesses average approximately 0.8 mils. The experience has been that when the thickness drops to 0.6 mils or below the shunt characteristics of the cells deteriorate and occasionally cells will actually be shorted.

The approach followed here has been to deposit CdS films in the range of 0.4 to 0.5 mils using a higher substrate temperature during film growth and a slower evaporation rate in order to secure a higher degree of film perfection. Some promising results were obtained with this approach using molybdenum or copper foil substrates; however, in the present period most of the work has been on the plastic substrate and two major problem areas have been encountered. First, the zinc underplating on the plastic substrates tends to re-evaporate from the substrate at the 350°C substrate temperature before the CdS film can deposit on the substrate. This resulted in very poor structured films which were completely unsatisfactory for high efficiency cells. Second, the utilization of CdS under these conditions was much poorer than had been experienced under the standard evaporation conditions and hence the films were much thinner than the desired 0.4 to 0.5 mil range even when the evaporation sources were completely filled with CdS.

In an effort to get around the latter difficulty, a semiclosed hot-wall system was constructed to operate at the 350°C substrate temperature. Most of the work in this period was directed toward trying to work out the process parameters of evaporating CdS films onto 350°C substrates utilizing techniques associated with the use of the semiclosed hot-wall chamber. These results have not been entirely satisfactory. Much of the reason for this lies in the deleterious effect of the zinc re-evaporating

TABLE II

KAPTON - SILVER PYRE-ML - CHROMIUM SUBSTRATE

Cell No.	OCV volts	SCC ma	Fill Factor	Eff %
A815C	0.435	580	63.0	2.9
A815D	.435	565	64.5	2.9
A817B	.480	670	66.5	3.9
A818C	.455	640	62.0	3.3
A819A	.455	700	70.5	4.1
A819B	.445	680	67.0	3.7
A819C	.450	670	69.0	3.8
A819D	.455	670	68.5	3.8
A820A	.470	670	69.5	4.0
A820C	.470	675	69.0	4.0
A821D	.460	605	67.0	3.4
A822A	.450	540	65.5	2.9
A822D	.455	580	68.5	3.3
Avg.	.455	634	67.0	3.5

from the substrate and from problems associated with shielding the sources to prevent overheating of the substrates during evaporation. It is suspected that the chances for the success of this technique are small until an improved evaporating source and associated shielding are developed and until the high vapor pressure zinc underplating on the substrate is eliminated. Therefore, further efforts to secure thinner CdS films will be postponed until these problems are solved or sidestepped.

Revised Grid Design

Some difficulties were encountered with the design of the etched copper grid developed earlier. These difficulties have led to a re-design of the grid. The changes consist of eliminating all but 2 of the 9 locating holes that were centered along the positive electrode tab and a strengthening of the grid wires where they join the electrode tab. The 9 locating holes previously used resulted in a handling problem with occasional tearing out of the holes. It has been found that 2 holes are adequate for locating purposes during cell lay-up. The extra holes have, therefore, been eliminated. The strengthening of the grid wires where they join the tab was accomplished by increasing the radius of curvature between the grid and the tab and increasing the width of the grid slightly at the point where the grid wire joins the tab. This appears to have strengthened the grids sufficiently to eliminate most of the difficulty previously experienced with pulling apart of the grid wires from the electrode tab during final lamination.

Gold Plated Electrode Tabs

The positive electrode tab, which is an extension of the grid, has been gold plated in order to get around the difficulty of corroded contacts. However, the negative electrode tab was not plated. It was found that it was difficult to make a solderable contact to the negative electrode tab. A copper-gold overplate of the negative contact tab was added to the process in this period to improve solderability. The copper underplating has been found necessary in order to secure a well adhering plating. The gold overplate was needed to prevent tarnishing of the copper. A copper sulfate plating bath is used for the copper; a standard gold fluoroborate bath for the gold. When proper precautions are taken to see that the tab is clean prior to electroplating a good adherent plating results. This has given a tab which can readily be soldered. It also greatly improves the appearance of the cells.

STABILITY STUDIES

Moisture Test

Cells in the standard package (i. e., with the grid cemented down with the gold-filled epoxy and covered with an epoxy-coated Mylar) have been tested for their moisture resistance. This test consists of placing the cells into an 80% relative humidity chamber maintained at room temperature and testing for output periodically. Table III summarizes the results of 9 standard 3" x 3" cells processed and tested during this quarter. The efficiency measuring equipment was modified between the initial and present reading of the first five cells listed to include a quartz-iodine light source and a Cu_2SO_4 filter so the readings here are not quite comparable. The last three cells were made under the same testing conditions. The data of Table III is still insufficient to characterize

TABLE III

CELL MOISTURE RESISTANCE TESTS

(80% Relative Humidity - Room Temperature)

Cell #	Initial Efficiency* %	Intermediate Efficiency* % @ days		Present Efficiency* % @ days	
D291A	5.1	5.0	52	5.1	101
D294A	4.3	3.8	47	3.5	96
D297C	5.1	4.9	45	4.9	94
D301A	5.1	4.9	52	4.8	101
D313C	4.9	4.6	38	4.6	87
D327A	3.1	3.1	42	3.3	91
D348C	5.4	5.3	23	5.3	72
D350F	5.4	5.3	21	5.3	70

*efficiency before recalibration of thermopile

the long-term stability of this package design in moisture ambients, but it does suggest that the present package cell is relatively unaffected by moisture. An early test cell produced in the present package has been stable in high humidities at 6.1% for over 182 days.

Temperature Exposure

Twelve standard 3" x 3" plastic substrate cells that were between 4.1 and 6.2% efficient were placed in a vacuum oven and held at 150°C for 53 days. An additional 6 cells have been in the oven for 33 days. The vacuum was maintained at approximately 10^{-4} Torr. There is a wide variation in the results with one cell showing a slight improvement, four cells showing a slight degradation and the rest showing considerable degradation. From examinations of the currents and voltages of these cells it becomes evident that the major reason for the drop-off observed in most cases is mostly due to loss in the fill factors. Comparison cells D321A and D349F on dry shelf storage have changed in the same period from 5.9% to 6.1% for the former cell and from 5.5% to 5.7% for the latter cell. The drop-off is, therefore, associated with the heat treatment.

The great variation in results makes interpretation difficult. The results on one hand look similar to those observed due to grid loosening of the old Capran-Mylar package. It may be possible that a similar effect is occurring with the epoxy cement at this high temperature. Attempts to see if this is so have been inconclusive.

Shelf Storage

Table IV gives the shelf stability data for a number of standard production line cells using the gold plated copper grid cemented in place with a conductive gold epoxy and with the Mylar cover plastic held in place with a clear epoxy adhesive. The data indicate good stability for this package.

CELL TESTING

Calibration of Light Intensity

Recent sunlight calibrations have been made at this laboratory using (1) an Eppley Thermopile (2) an Eppley Radiometer (3) a standard silicon cell and (4) a CdS cell which has been stable for the past two years. In addition, sunlight intensity data as measured on July 21, 1966 by Dr. A. J. Drummond, Director of Research for the Eppley Laboratory, Inc. at NASA-Lewis Laboratory at the same time of day as the Clevite measurements has been received. This data is tabulated in Table V. The Radiometer used by Clevite and that possessed by Dr. Drummond were further cross-calibrated at NASA-Lewis on July 22, 1966.

The Radiometer used by Dr. Drummond for this calibration is presently the standard sunlight measuring instrument accepted by the Bureau of Standards. The readings of the two Radiometers are within the limits of error of the instruments when different collimating angles of the two instruments and the difference in location of measurements are taken into consideration. The 2 year old CdS cell is in

TABLE IV

DRY SHELF STORAGE

Cell #	Initial Efficiency*	Present Efficiency *	Age (days)
D289D	5.4	5.4	99
D292D	5.2	5.4	98
D296B	5.0	4.9	96
D304A	5.3	5.0	91
D306D	5.4	5.5	90
D315A	5.8	5.8	89
D324D	5.8	6.0	83
D336D	4.6	4.6	76
D344C	5.8	5.5	70
Avg.	5.3	5.3	88

*efficiencies before recalibration

NOTE: The cells are stored in plastic boxes in a desiccator storage box containing a Silica Gel desiccant. The cells are maintained at room temperature.

TABLE V

SUNLIGHT INTENSITY MEASUREMENTS

July 21, 1966

Intensity Measuring Device	Sunlight Intensity (mw/cm^2)
Eppley Thermopile	89.6
Eppley Radiometer (Clevite)	95.8
Eppley Radiometer (Dr. Drummond at NASA-Lewis)	93.4
Silicon Cell (Secondary Standard)	84.6
CdS Cell (2 years old)	94.7

reasonable agreement with the two radiometer readings. Therefore, it has been decided to use the Radiometer as the primary source of sunlight intensity measurements.

Most previous measurements made at Clevite have used the Eppley Thermopile as the primary sunlight intensity instrument. The data of Table V indicate that this instrument yields higher cell efficiency readings than the Radiometer. The CdS cell used in these measurements gave stable readings up until March 26, 1966, the last time the cell had been checked. This reading was made using the thermopile as the primary intensity source. Thus, it appears that the thermopile has suffered some degradation in the last 4 months. It has been concluded that the efficiency readings in the last few months have been high, and that the data presented in this report can be considered to be approximately 6 to 7% high.

Effect of Cover Plastic

Figure 1 shows the plots of the percent light transmission of the Mylar cover plastic both with and without an epoxy coating after it has been processed through a typical lamination cycle in a vacuum in one case and in air in a second case. For the vacuum heated case, the Mylar by itself is approximately 85% transparent over the wavelengths measured and closer to 80% transparent when one surface is covered with epoxy. This difference is probably explainable on the basis of extra reflecting surfaces. For the air heated case, the percent transmission is about the same but the epoxy coated Mylar reveals a higher transmission than does the uncoated Mylar. This difference cannot be explained. The slight differences would apparently be quite minor in their effect on the performance of the cell.

Figures 2 and 3 show the spectral response curves of a cell before and after cover plastic application. The cell efficiency before cover plastic application was 4.5% and it was 4.7% after cover application; the increase being due exclusively to increased fill factor. Figure 2 gives the monochromatic and white light bias curves for the cell without the cover plastic. Figure 3 gives the same for the cell after cover plastic application. The chief difference between the two curves is the magnitude of the 0.51μ peak. This is probably due to the reduction in transmission of light through the cover plastic as shown in Figure 1. The lack of a similar reduction of cell output may be due to the change of the optical coupling to the cell which negates any observable effect of the relatively minor percentage reduction due to the 0.51μ light of the total light that activates the cell.

WORK PLANNED FOR NEXT PERIOD

In the Fourth Quarter standard process cell fabrication will continue at a high level and efforts will be concentrated on trying to restore the average level of cell output to the 6% range. Cell design improvements will be considered mainly with the use of thinner CdS film substrates and cover plastic and with the use of copper substrates and a copper-zinc (brass) layer on the metallized plastic substrate. Stability studies will continue with increased numbers of cells placed on shelf, moisture and high temperature exposure tests. A number of cells will be fabricated with electroplated gold grids utilizing the Harshaw process.

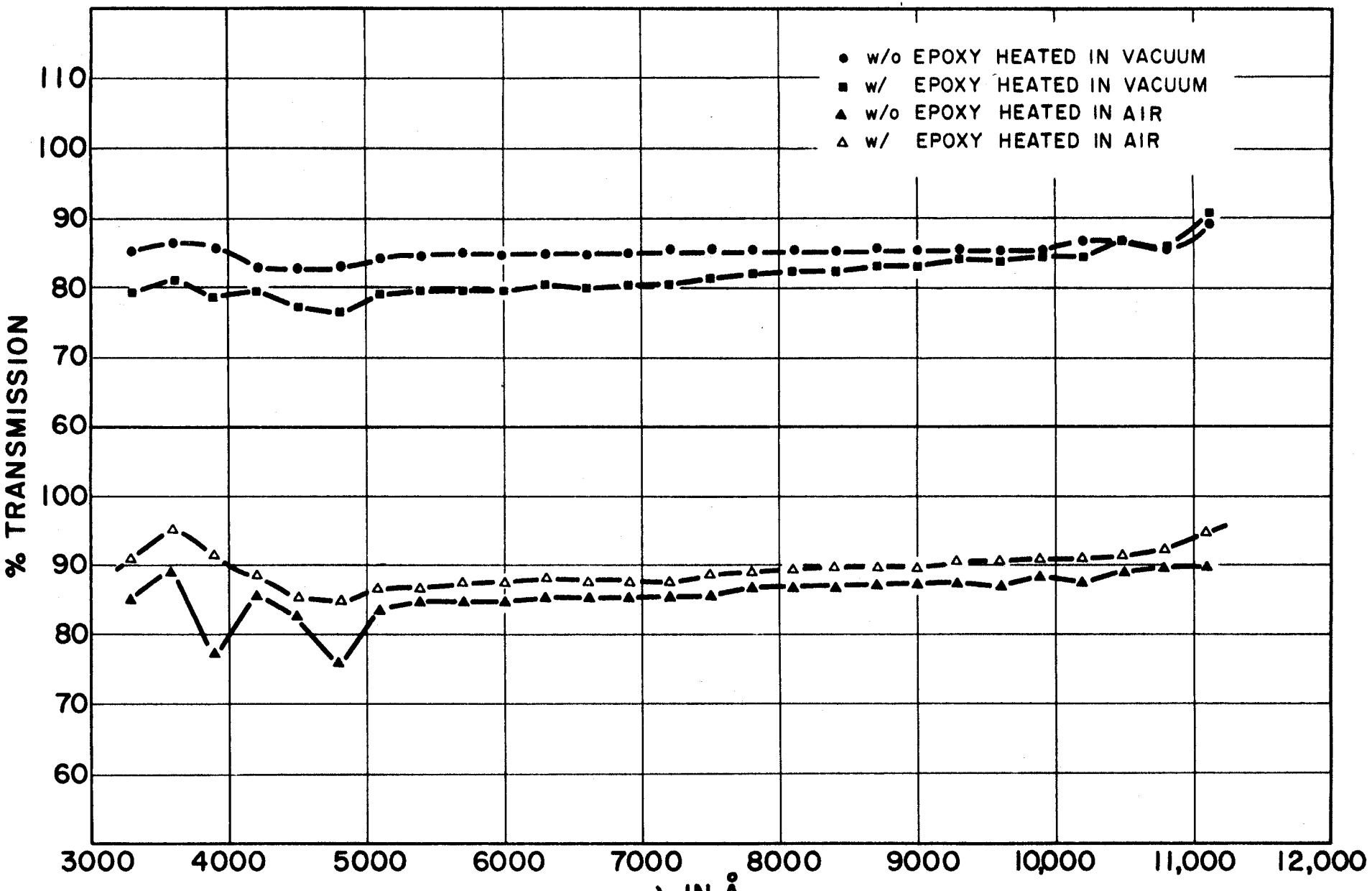
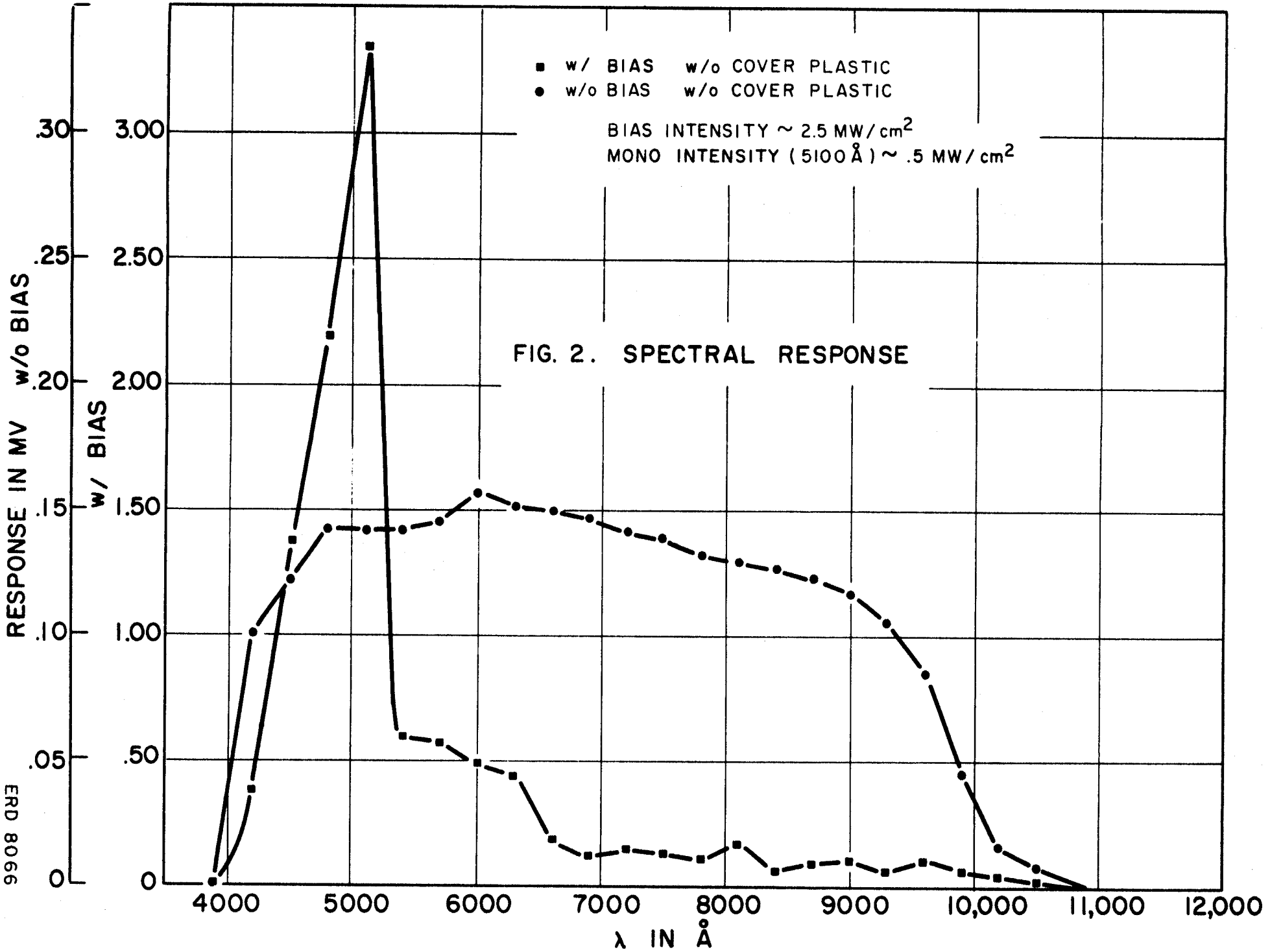


FIG. 1. PERCENT TRANSMISSION OF MYLAR COVER.

ERD 8065


■ w/ BIAS w/o COVER PLASTIC
● w/o BIAS w/o COVER PLASTIC
BIAS INTENSITY ~ 2.5 MW/cm²
MONO INTENSITY (5100 Å) ~ .5 MW/cm²
FIG. 2. SPECTRAL RESPONSE
RESPONSE IN MV
w/o BIAS
w/ BIAS
.30
.25
.20
.15
.10
.05
0
3.00
2.50
2.00
1.50
1.00
.50
0
4000
5000
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λ IN Å
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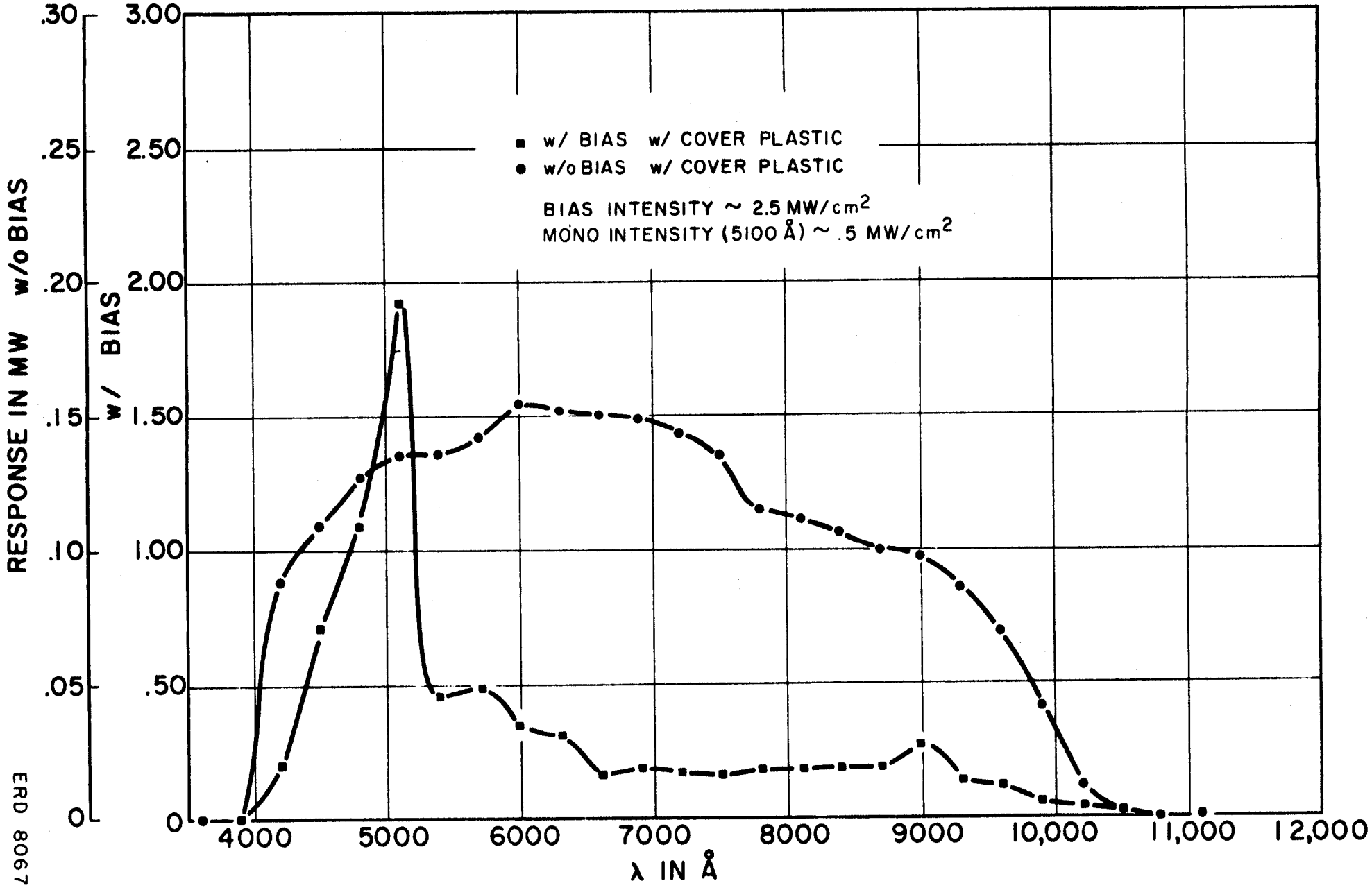

FIG. 3. SPECTRAL RESPONSE

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Radio Corporation of America
Semiconductor & Materials Division
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Sandia Corporation
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Electron Tube Division
Emporium, Pennsylvania
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Librarian

Union Carbide Corporation
Parma Research Center
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Solid-State Electronics Laboratory
Stanford Electronics Laboratories
Stanford University
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Westinghouse Electric Corporation
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Westinghouse Electric Corporation
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Massachusetts Institute of Technology
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Hughes Aircraft Corporation
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