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STUDY OF THIN FILM LARGE AREA PHOTOVOLTAIC SOLAR ENERGY CONVERTER

F. A. SHIRLAND, J. R. HIETANEN, F. AUGUSTINE, and W K. BOWER

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ELECTRONIC RESEARCH DIVISION

CLEVITE CORPORATION

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CLEVITE CORPORATION   ELECTRONIC RESEARCH DIVISION

STUDY OF THIN FILM LARGE AREA
PHOTOVOLTAIC SOLAR ENERGY CONVERTER

Second Quarterly Report
February 25, 1966 through May 24, 1966

Contract No. NAS 3-8502

SUMMARY

In the second quarterly period efforts were continued on working out
the difficulties of fabricating the revised design cells of both the plastic and
copper substrate constructions. Further design changes were introduced,
including the substitution of a gold pigmented epoxy cement for grid attach-
ment in place of the conductive silver epoxy cement, and the substitution of
clear epoxy cement to laminate the cover plastic to the cell in place of the
nylon plastic used previously.

It is believed that with these additional modifications the large area
CdS thin film solar cell will prove to be stable on extended shelf storage, in
moderately humid ambients, and on temperature cycling. The fabrication of
enough cells for a thorough evaluation of cell stability could not be accom-
plished during the last period because of processing problems but is expected
to be accomplished and the tests well underway in the coming period. Prelimi-
nary indications are that these cells are now relatively unaffected by high
humidity atmospheres, and that the grid contact no longer loosens with time.

Because of the processing problems associated with the new cell con-
structions, average output levels and the yield of good cells were down for this
period. These lower levels are definitely due to the new designs and processing
methods. Large area cell efficiencies of 6 to 8% were readily attained in this
period when making a few of the older less stable cell construction. It is
expected that outputs and yields will steadily climb to higher levels in the
next period as personnel zero in on the new cell designs and processing
methods.

CELL FABRICATION

    a.   Standard Process Line

        During the second quarter further changes were introduced in the
design of the CdS thin film solar cell. These were chiefly the use of a gold
filled epoxy cement for grid attachment in place of conductive silver epoxy
cement, and the use of clear epoxy laminating adhesive in place of Capran
(nylon) to hold the cover plastic in place.

        These changes were for cell stability reasons. These were
discussed in more detail in the first quarterly report. [1] (In brief, the conduc-
tive silver cement against CdS cell barriers gave rise to a transient degradation
of cell output when the cells were exposed to elevated temperatures, and the

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Capran adhesive was hygroscopic. It was found that gold cement did not give rise to cell degradation on heating, and that cells laminated with epoxy were relatively impervious to moisture degradation.)

However, these changes on top of those that were made in the first quarter made the task of cell fabrication that much more difficult. As a result, the yield of good cells continued poor, and the average efficiency of cells fabricated on the standard process line was even lower than in the first quarter. The data are summarized in Table I.

For most of the first month of this quarter severe difficulties were encountered with precipitation of elemental copper during barrier dipping of the plastic substrate cell. This design cell was withdrawn from fabrication until the end of the first month when the cause was isolated and corrective action taken (see below). About midway through the second month severe difficulty was encountered with the copper substrate cell. This was cracking of the copper substrate during cell lamination and was so severe that fabrication of this cell design was discontinued for most of the rest of the period. This copper substrate cracking is associated with particular lots of copper foil. It is expected that the problem can be eliminated by closer specification of the hardness of this material.

b. Cell Processing

Most of the effort in this period was spent in studying the difficulties encountered in fabricating the revised design cells on the standard process line and in developing ways of getting around the difficulties.

It was well established that the difficulties were due entirely to the changes in cell design and fabrication methods and not due to lack of control over the film deposition or barrier formation processes. On several occasions during this period a few cells were fabricated of the old design, using the processes that were standard at the time, and cell outputs were comparable to what had been obtained earlier. When the conditions of 6 to 9 months ago were used, cell outputs averaged between 6 and 7%; when the conditions of 12 to 18 months ago were used, cell outputs averaged between 4 and 5%. Therefore, attention was focussed on trying to eliminate the "bugs" in the process for making the more stable new design cells.

A difficulty that had been experienced intermittently for many months was the precipitation of elemental copper onto the barrier surface during the barrier dipping process. This difficulty was manifested by a reddish cast to the formed barrier and resulted in low cell efficiencies. At first it was thought that it might be the result of too much zinc of the surface of the substrate. The zinc is electroplated onto the substrate to ensure an ohmic contact of the CdS. However, all attempts to reduce the amount of zinc present, and to build up the thickness of the CdS layer so that the zinc would not "show through" did not solve the problem. Finally it was discovered that the lacquer used to mask the edges of the cell during barrier dipping was not completely dry. Residual solvent was being driven out of the lacquer when the cells were immersed in the hot CuCl solution, and this solvent was precipitating copper. The problem

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TABLE I

STANDARD PROCESS LINE CELLS - SECOND QUARTER

Construction	Month	Total Cells	Scrap Cells	Efficiency Min	Max	Avg.	Power/Wt. W/lb
Copper Substrate	1st	69	5	3. 0	6. 3	4. 8	47
	2nd	24	5	3. 2	5. 4	4. 2	42
	3rd	0	--	--	--	--	--
	Total	93	10	3. 0	6. 3	4. 7	46
Plastic Substrate	1st	5	0	3. 7	4. 2	4. 0	65
	2nd	73	33	3. 0	5. 4	3. 5	58
	3rd	54	28	3. 3	5. 4	4. 0	68
	Total	132	61	3. 0	5. 4	3. 7	62

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was eliminated by using masking tape in place of lacquer for edge masking, though an oven drying step was found to be equally efficacious. However, the use of masking tape is more convenient as it is easier to remove after barrier dipping.

Another major problem has been the development of a practical process for forming the conductive coating on the Kapton plastic substrate. When the process was first evolved last year this was done by spraying multiple coatings of silver-Pyre M. L. mixture onto the plastic substrate, drying out the solvent, curing in place, scraping away dust particles and other imperfections and then burnishing after each coat. The process was slow and painstaking and the yield was low since a single large dust particle could result in a large pinhole and a scrap piece. Attempts were made to reduce the number of coats required to secure the needed conductance and at the same time to reduce or eliminate the need for burnishing, by increasing the ratio of silver pigment used in the spray mixture.

At first it appeared that this was successful, but difficulties with poor adhesion and with uneven coatings forced a return to the earlier fabrication process. Part of these difficulties were probably due to agglomerates of silver particles that were never properly dispersed in the spray mixture, and part were probably due to magnetized iron particles which were later found to be prevalent in the flake silver raw material.

Other difficulties with the plastic substrate preparation process arose during the zinc plating operation. Inhomogeneities in the distribution of silver pigment or burnishing of the surface of the conductive coating cause uneven current distribution during electroplating which results in aggravating the inhomogeneities. This in turn causes pinholes or other flaws in the structure of the CdS film deposited as the next step. It was found that if the zinc was deposited by vacuum evaporation it was easier to control and gave much more uniform surfaces for CdS deposition. Unfortunately the facilities for evaporating zinc as a separate step were limited and it was believed prudent not to risk contamination of the CdS film evaporators with zinc until such time as it was established that cell quality would not be adversely affected.

The use of zinc on the silver-Pyre M. L. coating of the plastic substrate cell is questionable in any case. The zinc does not alloy into the silver, as it does for the copper substrate, and the vapor pressure of zinc at the substrate temperatures used for present CdS film deposition is well above the residual pressure in the vacuum chamber. The zinc is being used only because it was used earlier on the very successful plastic substrate cells fabricated during the previous contract, and because there has not been sufficient time or help available to prove-in an alternate material. It is believed that other metals such as chromium or germanium would be equally satisfactory in establishing an ohmic low resistance contact between the CdS film and the substrate and would be free of any tendency to re-evaporate from the substrate during CdS film deposition. It is planned to evaluate such contact metals in the next quarter.

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Difficulties with the copper substrate cell developed with cracking of the copper substrates during the cell lamination step. The cracking occurred on certain batches of copper foil and are believed to be a result of those batches being too hard to withstand the pressures applied during normal cell processing. The cracking became very prevalent at the time when a new lot of copper foil was received. A number of sheets of copper foil from a batch that had given much difficulty in this respect was annealed in an inert atmosphere to a temperature in the range of 350° to 450°C for a short period. The cells made from these sheets did not exhibit the cracking. However, the annealing step did cause some oxidation which was difficult to remove and which in turn caused trouble with CdS film adhesion. During the last month no copper substrate cells were made on the standard process line. It is planned to go back to copper substrates however as soon as a process for annealing the copper can be developed, or when suitable hardness controls can be established for the material as received from the vendor.

The low cell outputs experienced in this period are attributable chiefly to a poor electrical contact between the grids and the cell barriers. This has been found to be due mainly to the poor quality of the gold filled epoxy cement used for grid attachment. A suitable commercial gold filled epoxy cement was not available in this period and hence it was attempted to make do with what could be made in our own laboratory. The process used was to mix a gold powder of about 1 micron particle size with a clear epoxy resin, thin with MEK solvent and apply liberally to a gold plated copper grid with a soft bristle brush. After evaporation of the solvent, enough resin with embedded gold particles would remain on the grid to enable it to be cemented to the cell barrier and give reasonable adherence. However, it developed that cell outputs from this cement were much lower than had been experienced in the first quarter with the silver-epoxy cement, or in the previous year with the pressure contacted grid.

The photograph of a cross-section view, greatly enlarged, of a typical cell of this construction is shown in Fig. 1 and helps to explain the lower cell outputs experienced. This is a copper substrate cell. Clearly visible is the thin region at the surface of the copper where the zinc has alloyed in, and the rough texture of this copper foil substrate which contributes so effectively to good CdS film adhesion. However, it is also evident that there are very few conducting paths from the surface of the cell to the grid wire because the gold particles do not bridge the gap effectively. While this is a view of just one small portion of the grid contact, it is typical of several hundred of such cross-section views taken at other places on the same cell and on similar cells. It is estimated that less than one third of the area of the grid contact for the average cell of this construction was successfully established through the gold pigment.

However, when the ratio of the gold pigment in the epoxy binder was increased, the contact did not seem to be improved. A careful analysis was then made of how the epoxy resin behaves during the various cell processing steps from grid attachment through final lamination and heat treatment. For study purposes cells were fabricated with the epoxy minus the gold powder in the grid application step of the process. These cells were fabricated under the
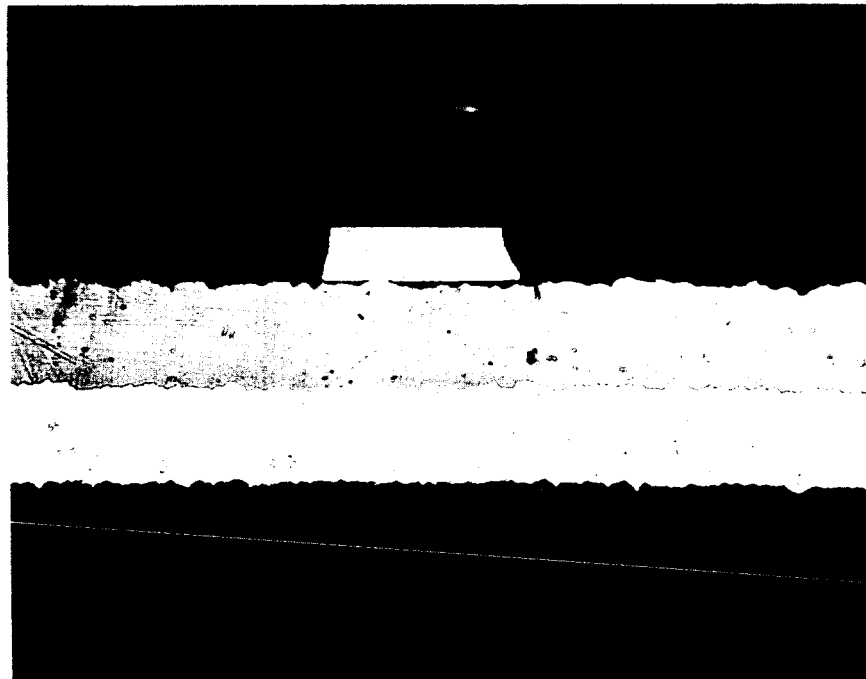
5
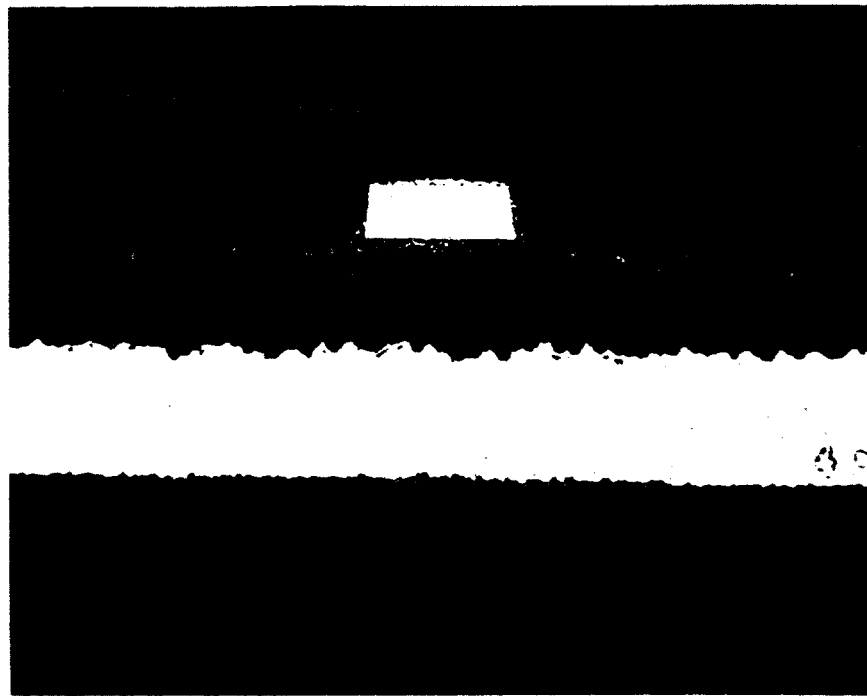
Mylar Cover
Plastic

Epoxy

CdS Film

Copper
Substrate

500X

Plate 13832

FIG. 1. Enlarged Cross-Section View of Copper Substrate Cell with Grid
Attached with Gold Filled Epoxy Cement

Mylar Cover
Plastic

Epoxy

CdS Film

Copper
Substrate

500X

Plate 13790

FIG. 2. Enlarged Cross-Section View of Copper Substrate Cell with Grid
Attached with Unpigmented Epoxy Cement.

microscope and prodded from time to time to determine what kind of a bond was effected between the grid and cell barrier. These observations revealed that the conditions normally used to cement the grids (as recommended by the manufacturer of the epoxy) are not sufficient to fully cure the epoxy. While the room temperature bond strength is good, above 70°C the bond strength is poor and the subsequent application of the cover plastic and the curing cycle for the epoxy used to laminate the cover plastic would cause a softening of the grid adhesive. As a result, the grid would be pushed away from the barrier by the new epoxy used to laminate the cover plastic.

Increasing the time of cure of the grid adhesive (from 30 minutes at 196°C to 2 hours) was not sufficient to improve the bond so that the grid contact would not be interrupted in lamination. Another grade of epoxy cement from the same supplier was tried, but did not solve the problem. It was found that a 15 hour heat treatment in a vacuum oven at 130°C was adequate to cure the grid adhesive so that it would not loosen during the subsequent lamination step.

It is interesting to note that when the gold pigment was left out of the cement used to attach the grids to the cells, reasonable cell outputs were obtained if the epoxy cement was applied to the grid in a thin enough layer. About 0. 05 mils thickness was about optimum in this respect. More adhesive would give lower outputs and less adhesive would give a poor bond. Apparently enough high spots of the barrier surface push through the very thin epoxy layer to establish good electrical contact. This is illustrated in Fig. 2. The stability of cells with this type of grid contact has not been determined, nor have very many such cells been made. Cell efficiencies in the range of 6 to 8% were - obtained in a number of instances.

At the end of this quarter a commercial fabricator of epoxy cements furnished a sample of a conductive gold epoxy especially formulated for our application. This was the same epoxy used previously in the conductive silver epoxy composition with gold substituted for the silver. Initial trials of this cement are encouraging, but microscopic examination of the bond of this cement indicates that it too apparently requires a longer heat soak to adequately cure so that subsequent lamination will not loosen the bond.

CELL DESIGN

    a.   <u>Thinner CdS Films</u>

The development of a process for depositing thinner CdS films, in the range of 0. 4 to 0. 5 mils thick, that are well structured, adherent and pin-hole-free and which are suitable for fabrication into high efficiency cells was started in this period.

There are a number of potential advantages to being able to use such thinner films for CdS solar cells. First, such thinner films will enable thinner substrates and cover plastics, on the same order of thickness, to be used. This will make possible a much thinner (approximately 0. 002" overall thickness), much more flexible cell. The specific power-to-weight ratio for arrays of

plastic substrate cells should then be on the order of 200 watts per pound of array weight with 5 to 7% efficiency cells, and of course proportionately higher for higher efficiencies. The stresses that build up in large area cells due to mismatch of CdS film and substrate thermal expansion should be appreciably less than at present. This should make for greatly improved CdS film adhesion and should eliminate the present tendency for the CdS films to develop stress cracks on the plastic substrates during processing. Also, the cost of the cells would be reduced since less material would be used in them.

CdS films and plastic substrate and cover layers on the order of 0.5 mils each seem like a practical thickness level. Films of this thickness appear to possess adequate strength so that large area cells and arrays should be practical and should be convenient for handling and processing. A few cells of this thickness level have been fabricated and have given good output levels -- hence it is known that they are possible.

Preliminary experiments have indicated that a higher level of CdS film perfection is required to secure good cells from thinner films. However, improved CdS film quality is a virtual necessity in any case if large area CdS film cells are to be used for space power supplies with their stringent quality assurance requirements.

The approach planned to secure such improved CdS film quality is to improve the cleanliness and homogeneity of the substrate by more stringent preparation procedures, and to improve the perfection of the CdS film deposited on such better surfaced substrates by the use of higher substrate temperatures and evaporation sources that are designed to eliminate CdS spattering.

In this period 0.4 to 0.5 mil thick CdS films have been deposited on 1 mil thick copper substrates using 0.001 mol% indium doped CdS and a substrate temperature of 350°C. The 1 mil copper was used because it was conveniently available. An improved copper cleaning process was developed. This consists of a hot Alconox (detergent) scrub, an HNO_3 etch, a second hot scrub and then a final etch and a thorough rinsing. Zinc is then electroplated to a thickness of about 4000 A from a fluoborate bath. The zinc alloys with the copper to form a brass surface layer when the substrate is pre-heated in the vacuum deposition chamber.

This process has yielded excellent appearance, very adherent pinhole-and-spatter-free CdS films 0.4 to 0.5 mils thick. The first films were cut into 1-1/2" squares and made into cells using various etching and barrier dipping conditions. A series of such experiments indicated that best results might be obtained using a 10 second pre-etch in 1:1 HCl solution, and a 2 to 3 second dip in CuCl solution. Longer dip times of 4 to 5 seconds gave very dark blue colored barriers which appeared to be shorted. Shorter dip times of 1 to 2 seconds gave very low output cells.

A second controlled experiment was run on separate 1-1/2" square pieces cut from the same 0.40 to 0.45 mil thick CdS film and processed using different barrier dip times. The results are summarized in Table II and verify the earlier indications.

TABLE II

Effect of Barrier Dip Time - 0.40 to 0.45 mil Thick CdS Films

Cell No.	Barrier Dip Time	OCV (V)	SCC (A)	J (mA/cm^2)	Fill (%)	Eff. (%)
758a	5 sec.	———————— shorted ————————				
758b	4 sec	———————— shorted ————————				
758c	3 sec	0.47	.255	22.2	54	5.6
758d	2 sec	0.41	.130	10.9	50	2.2

Note: 10 sec. pre-etch in 1:1 HCl.

The best results were obtained with a 3 second dip time, but even this cell had a poor characteristic curve with a 54% fill factor which was the result of a poor shunt characteristic. Further work is needed, possibly with a less active barrier dip solution and/or with a gentler etch, before a really satisfactory process is evolved.

One problem area in making the thinner CdS films (which may also be affecting the barrier formation process) is associated with the zinc coating on the substrate. Even though the zinc does alloy with the copper substrate there appears to be enough extra zinc present that much of it evaporates from the substrate at the 350°C substrate temperature during CdS film deposition. At first this zinc deposits on the cooler tooling and bell jar areas of the deposition chamber, but as CdS film evaporation progresses these areas warm up considerably and the zinc re-evaporates. Even during the final anneal period zinc may continue to be redistributed in the vacuum chamber and it has been found on the top surface of the CdS film in some cases.

As discussed previously, alternate metallic coatings are being evaluated for the plastic substrate and these should be equally desirable for copper substrates. With higher substrate temperatures during CdS film deposition, a metallic coating having a much lower vapor pressure than zinc is required for establishing an ohmic contact between the CdS film and the substrate.

b. Copper Substrates

The copper substrate has been found to possess many advantages over the molybdenum substrate that had been used before for CdS thin film solar cells. External connections can be readily established to the copper substrate by soldering, while it is difficult to make a suitable electrical connection to molybdenum. Copper, of course, is an excellent electrical conductor, it is lighter in weight and is much more economical. Its coefficient of thermal expansion however is much larger than that of CdS so there is much more tendency for the composite film to curl than in the case of molybdenum. But in practice this has not been any real difficulty as the back surface of the copper substrate can readily be work hardened by a light rubbing action to just offset the stresses due to the CdS film on the front surface.

The difficulties experienced in this period with the copper substrates cracking during cell processing, while they did interfere with cell fabrication, are not believed to be a serious obstacle to the use of copper for this application. The copper foil used is not normally controlled for hardness as this property does not seem to be of concern to the major users of the foil. Annealing the copper for a short period in an inert atmosphere, or in vacuum, to 350° to 450°C is adequate to soften the copper so that cracking does not occur.

In addition to the cracking some difficulty was experienced in cleaning the copper foil prior to CdS film deposition. This was finally traced to a chromate coating placed on the copper by the supplier to facilitate adhesion in the manufacture of printed circuit boards, which is the chief end application of this copper foil. This chromate coating however interfers with CdS film adhesion. It is difficult to remove, however, and has been a factor in the poor yields experienced with copper foil substrate cells in this period. In the coming period it is planned to secure the copper foil without the chromate coating, and anneal the copper prior to use. With these steps it is expected that satisfactory fabrication of copper substrate cells can be resumed.

c. Thinner Substrates

The cells presently being fabricated are approximately just under 0. 004" in overall thickness. The substrate and the cover plastic are both 0. 001" in nominal size, the CdS film is just under 0. 001" and the collector grid is 0. 0005" thick. There is a 0. 0005" plastic insulator strip positioned under the grid where it crosses the edge of the cell to prevent shorting to the substrate, and at this point the cell is 0. 0040" thick nominally. The adhesive used to hold these parts bonded together squeezes out to a very thin layer and does not normally affect cell thickness.

With molybdenum it was difficult to go below 0. 002" in substrate thickness as the manufacturing process for rolling thinner molybdenum appeared to affect the structure of the metal adversely for CdS solar cell purposes. Chemically milling the molybdenum to thinner gages after completion of the cell has been used[2] to secure thinner and lighter weight cells, but this is a tedious and wasteful practice.

There has been no difficulty whatever in going from 0.002" to 0.001" thicknesses for either the plastic or the copper substrates used in our cell fabrication process. A few preliminary tests have indicated that substrate thickness can be further reduced to 0.0005" for both copper and plastic substrate cells with little or no difficulty. It is planned to carry out this reduction in the last half of the contract period and to combine the thinner substrates with thinner CdS films and thinner cover plastics so that overall cell thickness will be reduced to about 0.002". This is believed to be a very practical cell thickness where excellent performance parameters can be secured in conjunction with a very thin very light weight package that will still possess more than adequate physical strength and integrity.

d. Integral Leads

The use of designs with the negative lead an integral extension of the cell substrate and the positive lead an integral extension of the collector electrode grid has proved to be very practical in fabricating CdS film cells. The increased reliability of this design, and the elimination of the extra processing steps of attaching separate leads more than compensates for the slight amount of extra care required in handling and processing cells with the protruding lead tabs.

STABILITY STUDIES

a. Shelf Storage

A few CdS thin film solar cells of 4 to 5% conversion efficiency have been quite stable on long term dry shelf storage.[1, 3] However, other cells have not been stable and the causes for this have been determined to be due to loosening of the grid contact in most cases. Extensive data on this effect have been reported previously.[3] Confirmation of the cause of this degradation was obtained by re-laminating selected degraded cells and observing that the original high output was restored. The cells that were on dry shelf storage and reported in the First Quarterly Report, are still on shelf storage, have been read again, and are showing no changes from the trends reported earlier.

There are only a few cells with epoxy cemented grids old enough to have shelf storage data available. These are all cells with the conductive silver epoxy rather than the conductive gold epoxy now being used. The scant data available does indicate that the epoxy cemented grid cells are stable on shelf storage when the epoxy is fully cured and when the cell output reaches its full value.

Cells fabricated with epoxy cement, for either grid attachment or for cover plastic attachment, may take some time to reach full output. This effect depends on the particular type of epoxy used and in some cases times as long as 1 or 2 months appear to have been needed. This effect may be associated with stress relieval due to the shrinkage of the epoxy during cure, or it may be an indication that the epoxy is not fully cured right away. In any case, it appears that the effect can be greatly speeded by annealing the cells at higher temperatures.

b.   Temperature Exposure

Cells with silver epoxy cement used to attach the grid to the cell barriers experienced a temporary degradation when exposed to elevated temperatures. This appears to have been the cause for cells with silver epoxy cemented grids failing on temperature cycling test. The effect is severe with temperatures on the order of 230°C used to laminate the cells, 10 to 15 minutes being enough to drop the output to less than 20% of the full value. Such cells recover if allowed to stand at room temperature for a few hours. At lower temperature exposures much longer times are required for the effect to occur and this explains why several hundred cycles were required in some cases, with temperatures on the order of 60 to 80°C, to degrade the cells on that test.

However, the degradation of output on higher temperature exposure does not appear to occur at all if gold is used as the contact material to the barrier. For this reason, present design cells use gold filler in the cement used to bond the grids to the cells. Also, as an extra precaution, the grids are gold plated just in case they should touch the cell barrier directly without the conductive gold cement between.

c.   Moisture

The epoxy cemented cover plastic cell construction appears to have alleviated the moisture sensitivity problem sufficiently so that no special precautions are now required to protect these cells from degradation due to moisture in the ambient atmosphere. One 55 cm^2 has been on 80% relative humidity test at room temperature for 12 weeks and has been tested for output weekly. Throughout this period the cell gave readings between 6. 0 and 6. 2% conversion efficiency. This variation appears to be due to the reproducibility of our output test. There is no trend evident in the data. Other cells have been submerged in distilled water for several weeks and shown degradations of 10% or less of the initial   4 to 6% conversion efficiencies

More complete stability testing is planned for the present design cells in the coming period. Tests during the last two periods have been minimal because of changes in cell design that were being worked out to overcome known weaknesses in cell performance or stability. In particular, extensive testing on shelf and elevated temperature storage will be carried out, and on high humidity storage. A large number of the latest design cells with good gold epoxy cemented grids will be delivered to the Contract Monitor for testing on vacuum thermal cycling.

WORK PLANNED FOR NEXT QUARTER

The major emphasis in the third quarter will be on fabricating larger quantities of the new stable cell designs and characterizing these cells fully for performance and stability. In particular, larger numbers of cells will be placed on dry shelf storage, on storage in humid atmospheres and on elevated temperature storage. Also, larger numbers of cells will be delivered to NASA for evaluation and testing on thermal cycling.

Improvements in cell design will be continued with efforts to prove-in the process for using 0.5 mil thick CdS films, and 0.5 mil thick substrate and cover plastic layers.

Cell output improvements will be sought by further improvements in the grid contact, the contact between the CdS film and the conducting portion of the substrate, and by improvements in the barrier formation process. The use of alternate ions to chlorine in the barrier dip process will be evaluated further. The spectral response of cells will be checked to see if changes in residual impurity content may not improve the output of these cells under AMO conditions.

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