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EFFECTS OF SAMPLE PREPARATION ON PHOTOMICROGRAPHS OF CADMIUM SULFIDE THIN-FILM SOLAR CELLS

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ABSTRACT

Cross-sectional photomicrographs of thin-film CdS solar cells revealed large, lamellar cracks in the CdS layer. Investigation of these cracks showed that they were caused, or at least aggravated, by the mounting and polishing techniques used, particularly by the absence of firm mounting of the cell sample. Use of an aluminum mechanical sandwich or of a slab epoxy mounting technique yielded cross-sectional photomicrographs of the CdS thin film solar cells showing no large, lamellar cracks in the CdS layer.

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SUMMARY

Cross-sectional photomicrographs of thin-film cadmium sulfide (CdS) solar cells revealed large, lamellar cracks in the CdS layer. Investigation of these cracks showed that they were caused, or at least aggravated, by the mounting and polishing techniques used, particularly by the absence of firm mounting of the cell sample. Use of an aluminum mechanical sandwich or of a slab epoxy mounting technique yielded cross-sectional photomicrographs of the CdS thin-film solar cells showing no large, lamellar cracks in the CdS layer.

INTRODUCTION

Thin-film cadmium sulfide (CdS) solar cells, though relatively efficient solar energy converters, are susceptible to degradation when operated in a simulated space environment. In an attempt to determine the causes of degradation, cross-sectional photomicrographs were taken at other laboratories and at the Lewis Research Center. One outstanding feature noted by these laboratories was the existence of large, long, lamellar cracks in the CdS layer of the completed solar cell.

Efforts were made to determine whether or not there was a correlation between metallographic observation of the (lamellar) cracks and the performance of the solar cell from which the metallographic sample was taken. No correlation was found. An attempt at a correlation between the nonuniform temperature distribution of an operating solar cell and lamellar cracking was inconclusive.

These negative results raised the question of whether the metallographic sample preparation could cause or at least aggravate the lamellar cracks. The cell is a very thin, flexible laminate, one layer of which is crystallite CdS, which is quite fragile in bulk form.

To isolate the possibility of sample preparation as a cause of the large, lamellar cracks, various mounting techniques were investigated. The techniques tried were mechanical sandwich, acrylic plastic sandwich, large and small epoxy potting, and epoxy slab mounting. One standard metallographic mounting technique, a thermosetting polyethylene, was undesirable because of its required high temperature and pressure.

CELL DESCRIPTION

The standard CdS thin-film solar cell is a 3- by 3-inch (7.62- by 7.62-cm) laminate, as shown in figure 1. Figure 2 shows a cross-sectional photomicrograph of a CdS solar cell. The substrate is a polyimide film on which is sprayed a polyimide enamel filled with silver flake. A flash of zinc is placed on the silvered polyimide enamel to act as an ohmic contact to the CdS. The layer of CdS is evaporated on the substrate to a thickness of approximately 1 mil (25 μm). The barrier layer of copper sulfide (Cu_2S) is then put on the cell by dipping it in a solution of cuprous chloride (CuCl). A heat treatment of the cell follows. The gold-plated copper grid is pressed onto the cell with gold-filled epoxy acting as an ohmic contact. A cover plastic of polyimide film with an uncured epoxy layer on one side is then laminated to the gridded cell. The cell is now a complete solar energy converter.

SAMPLE PREPARATION

An effort was initiated and completed to determine if the mounting technique has an effect on the integrity of a polished sample and to determine which technique yields a desirable method of mounting cells in preparation for cross sectioning. Five different mounting techniques were tried. Figure 3 illustrates the five mounts used in potting CdS solar cells. The first is a large epoxy pot about 2 inches (5.1 cm) in diameter. The CdS cell sample is positioned during potting by two cotter pins. The second mount is a small epoxy pot about $1\frac{1}{4}$ inches (3.1 cm) in diameter. The third is the mechanical sandwich mount. In this mounting technique, the CdS cell sample is placed between two half cylinders of aluminum which are then held together with screws. The fourth mount is the slab mount. For this technique, the CdS cell sample is dipped into a vial of epoxy and then lifted out letting the excess epoxy drain off. The sample thus prepared is then cured. The fifth and last mount is an acrylic sandwich. This technique is similar to the mechanical mount except that the two acrylic half cylinders are held together by a special glue. The epoxy used in the mounts was 4 parts epichlorohydrin/bisphenol A-type epoxy resin containing reactive diluent to 1 part modified fast curing liquid amine. This epoxy has a volumetric shrinkage of about 3 percent on curing.

Final preparation of the various metallographic cell samples mounted by these techniques was accomplished by first wet grinding through 600-grit paper to reach an area of interest. Then the sample was lapped and polished with 3-micrometer alumina and 1-micrometer chromic oxide.

RESULTS AND DISCUSSION

Comparison of the various mounting techniques entailed mounting parts of one production CdS solar cell which had previously been cut into a number of small pieces. This cell was a typical production CdS cell and about 4 percent efficient at air mass zero at 25° C.

A series of photomicrographs (figs. 4 and 5) depicts the damage noted in the CdS cell samples as a function of the mounting technique used. The damage in a CdS solar cell when a large epoxy pot is used is extensive, as shown in figure 4(a). When a small epoxy pot is used the damage is somewhat less extensive but still a separation between the CdS film and silvered polyimide enamel is noted (fig. 4(b)). The aluminum mechanical sandwich and the slab-mounted samples (figs. 4(c) and (d), respectively,) were the best techniques used. No damage was noted to the cells when either technique was used. The acrylic sandwich mount was the last technique investigated. Cells mounted by this technique showed damage in the form of a separation between the CdS layer and silvered polyimide enamel (fig. 4(e)).

A separate cell sample was mounted in a thermosetting polyethylene. The high pressure and temperature, 1200 psi and 350° F (827 N/cm^2 and 168° C), caused extensive damage to the sample. On this basis further investigation of the mounting technique was discontinued.

A sample cell mounted in a small epoxy pot showed more damage if it was polished in a direction perpendicular to the CdS cell sample. A parallel polish produced a minimum of damage. Cells mounted in an aluminum mechanical sandwich and cells mounted in a slab mount showed no damage irrespective of whether they were polished perpendicular or parallel to the CdS cell sample.

There are two possible reasons why the sample preparation could cause, or aggravate, large, lamellar cracks in the CdS solar cell sample. These are (1) the exothermic temperature rise of the curing epoxy, and (2) the absence of firm mounting of the cell sample. The temperature of the curing epoxy was measured as a function of time for the various mounting techniques that use epoxy. Results of these measurements are shown in figure 6. Also as the epoxy cures, it shrinks away from the sample allowing the CdS cell sample to "wobble" in the mount during polishing. In an effort to isolate these two effects, samples mounted in the aluminum mechanical mount and slab mount were subjected to a temperature of 120° C for 30 minutes. Initially, no cracks were visible in either sample, and after the heat treatment, no lamellar cracks appeared. Therefore, temperature does not appear to be the cause of lamellar cracking during mounting.

Tabulating various mounting techniques by using the criteria of relative mount separation from the cell sample, absence of firm mounting, (table I) reveals that the mounts that have the most separation have the most damage to the CdS cell sample.

An experiment was performed to pin point the absence of firm mounting as the possible cause of lamellar cracks. A cell was mounted by using the slab technique. The photomicrograph of this cell is shown in figure 4(d). There were no significant lamellar cracks visible in this sample. The mount was then placed in a small epoxy pot. After the sample was repolished, a photomicrograph was taken that revealed appreciable damage to the CdS layer of the cell, as shown in figure 5. Combining the data shown in table I and data from the preceding experiment leads to the conclusion that the absence of a firm mount in which the cell was placed causes, or at least aggravates, the lamellar cracks in the CdS layer of the thin-film solar cell.

In all samples, irrespective of mounting techniques used, etching revealed small lamellar cracks. A CdS cell sample prepared by using the slab mount is shown in figures 7 and 8 before and after etching with a 15-percent solution of hydrochloric acid (HCl). The small lamellar cracks revealed by etching are either strained regions in the CdS layer of the cell that were preferentially etched, or they are extremely small cracks enhanced by the etching process. However, these cracks are noncontinuous and appear to be fundamentally different from the large, lamellar cracks observed.

SUMMARY OF RESULTS

An investigation was made to determine the cause of large lamellar cracks that appeared in the cadmium sulfide layer of a cadmium sulfide thin-film solar cell.

1. The cracks were shown to be caused by, or at least aggravated by, the absence of firm mounting of the cell sample prior to polishing. The severity of cracking was found to increase with increasing separation of the mount away from the sample.

2. Cells prepared by mechanical mounting or by use of the epoxy slab technique showed no significant damage.

3. Lapping and polishing the sample in a direction perpendicular to the sample caused more damage to the sample than a parallel lap and polish, if any separation of the mount occurred.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 27, 1968,
120-33-01-10-22.

TABLE I. - EFFECT OF MOUNT SEPARATION

Type	Relative separation	Relative damage
Large epoxy pot	Large	Much
Small epoxy pot	Some	Some
Acrylic plastic sandwich mount	Some[a]	Some
Slab epoxy mount	Very little	None
Aluminun mechanical sandwich mount	None	None

[a]In worst case, separation can be seen with naked eye as in "white" area shown in acrylic sandwich of fig. 3.

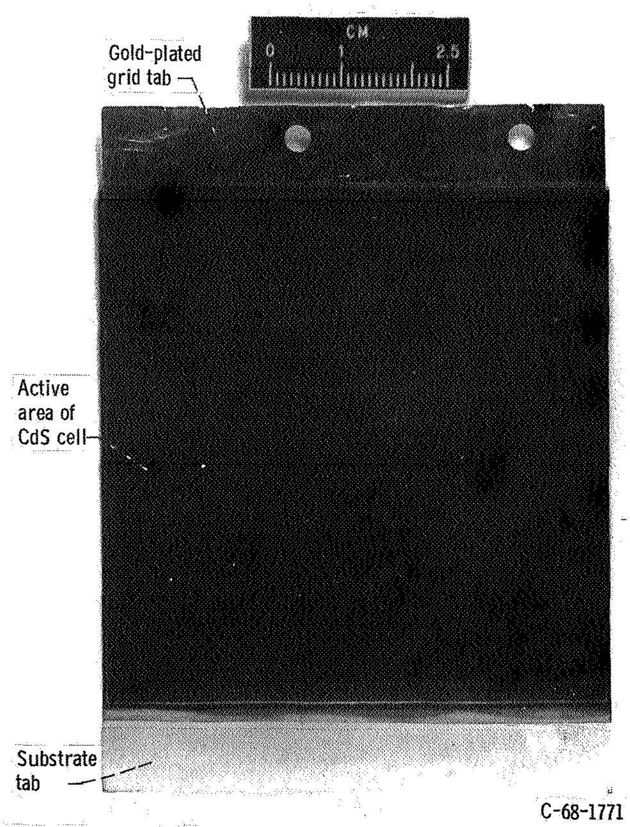


Figure 1. - Cadmium sulfide thin-film solar cell.

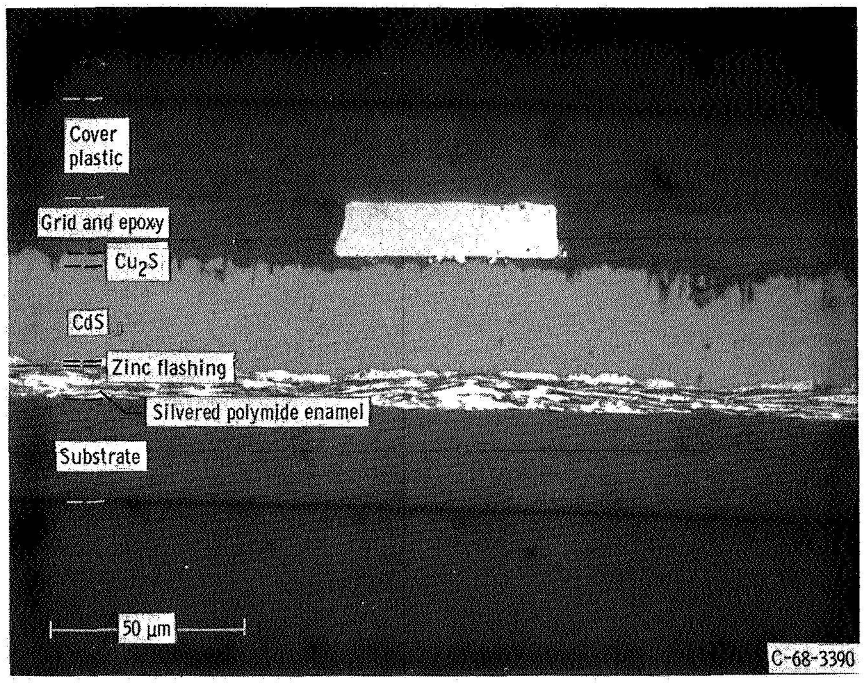
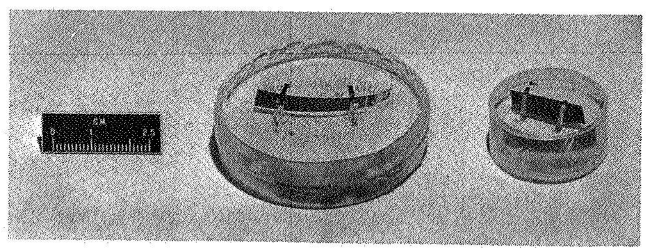
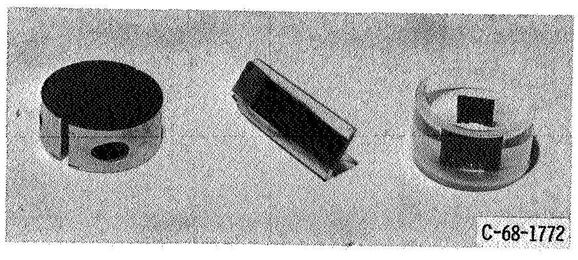


Figure 2. - Photomicrograph of standard cadmium sulfide thin-film solar cell.



Large epoxy pot (2 in., 5.1 cm)

Small epoxy pot (1.25 in., 3.1 cm)

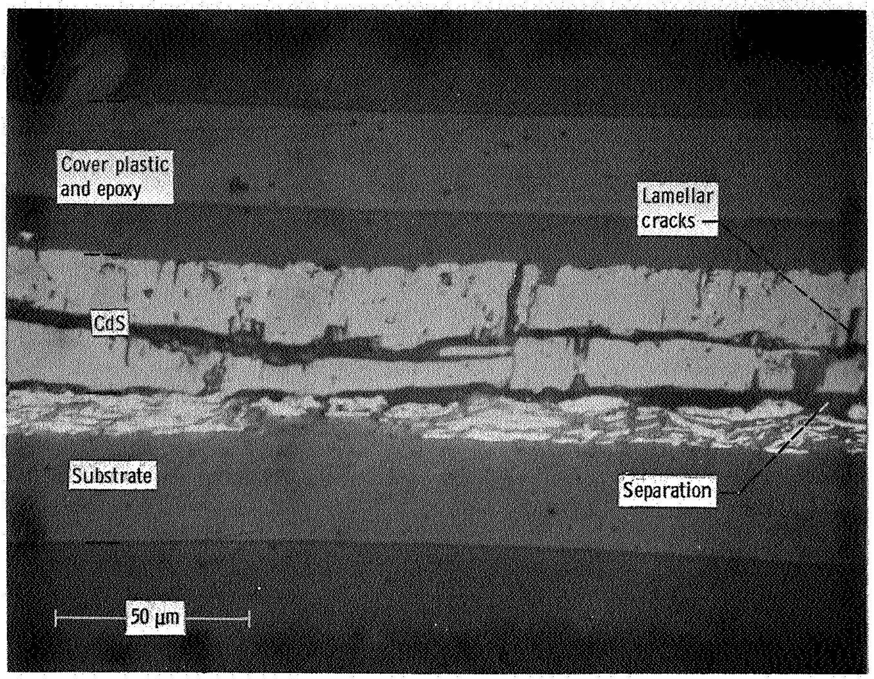


Aluminum mechanical sandwich mount (1.25 in., 3.1 cm)

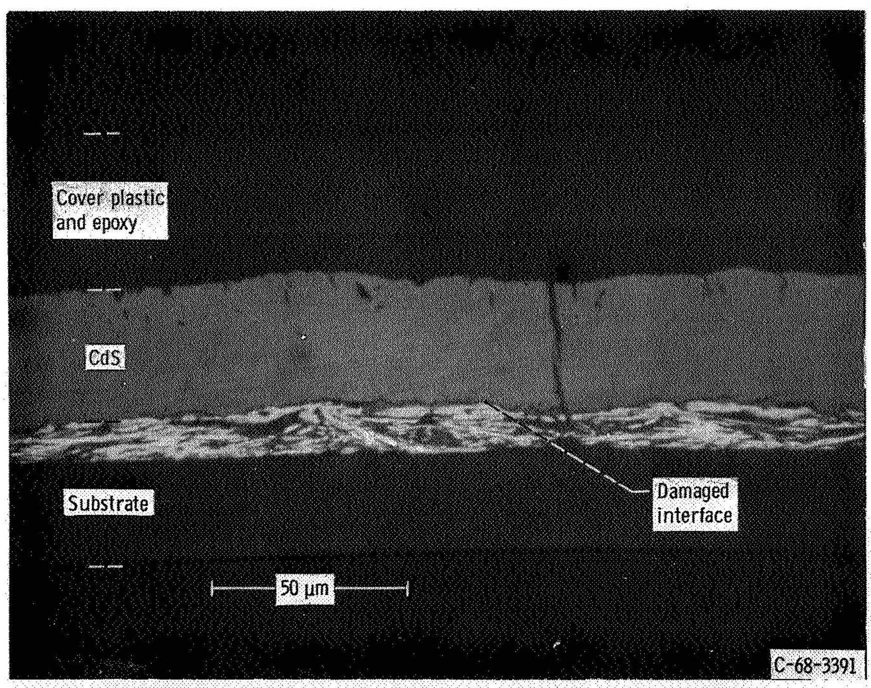
Slab epoxy mount

Acrylic plastic sandwich (1.25 in., 3.1 cm)

Figure 3. - Mounting techniques.

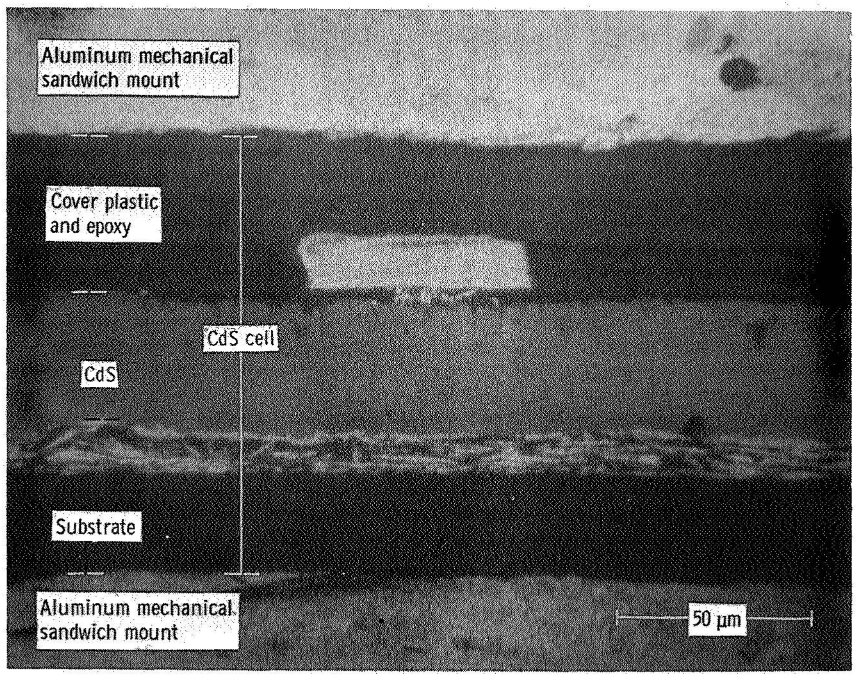


(a) Large epoxy pot.

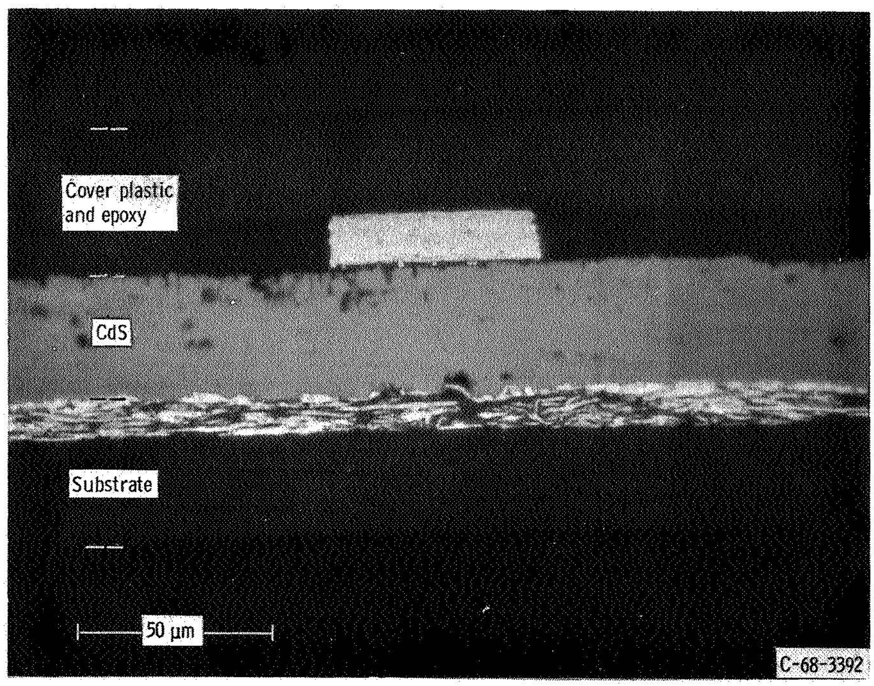


(b) Small epoxy pot.

Figure 4. - Photomicrographs of standard cell mounted and polished by different techniques. X500.

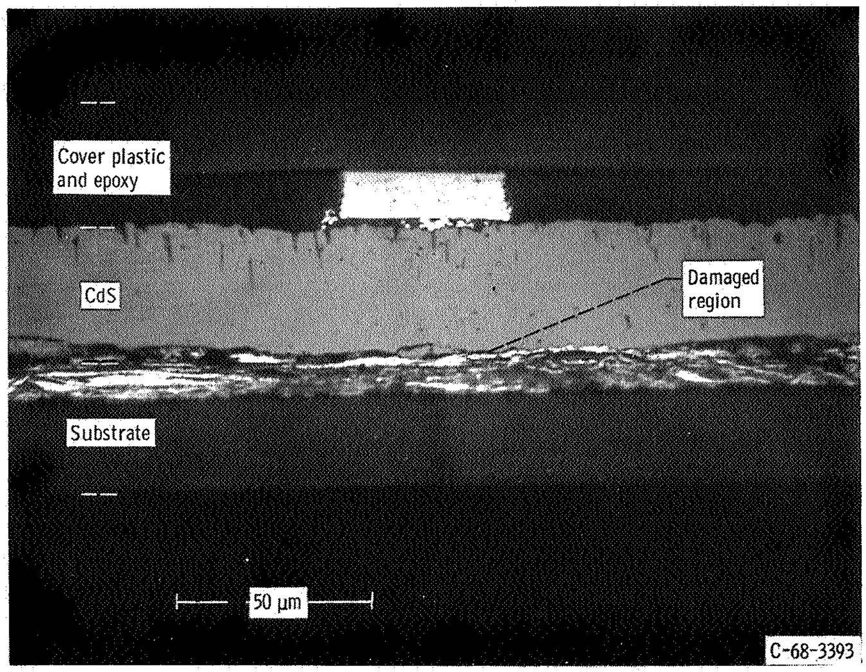


(c) Aluminum mechanical sandwich mount.



(d) Slab epoxy mount.

Figure 4. - Continued.



(e) Acrylic plastic sandwich mount.

Figure 4. - Concluded.

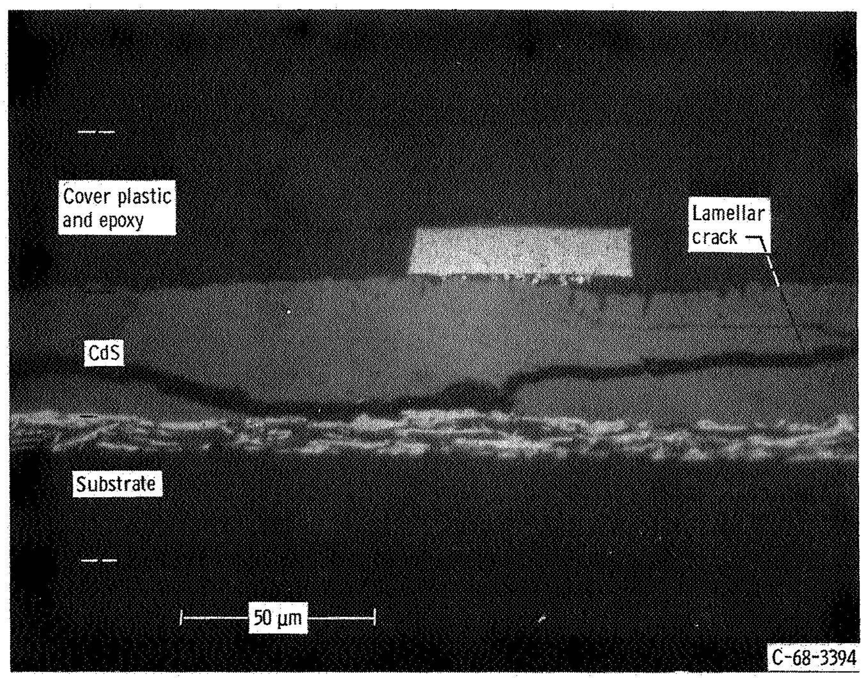


Figure 5. - Photomicrograph of same sample as shown in figure 4(d) after remounting in a small epoxy pot and repolishing.

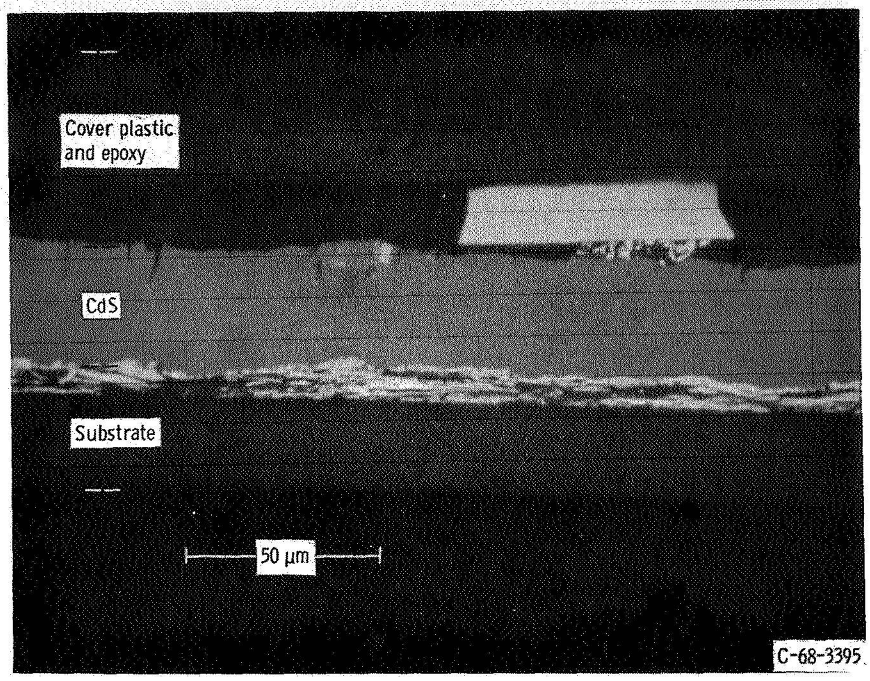


Figure 7. - Photomicrograph of cadmium sulfide cell before etching.

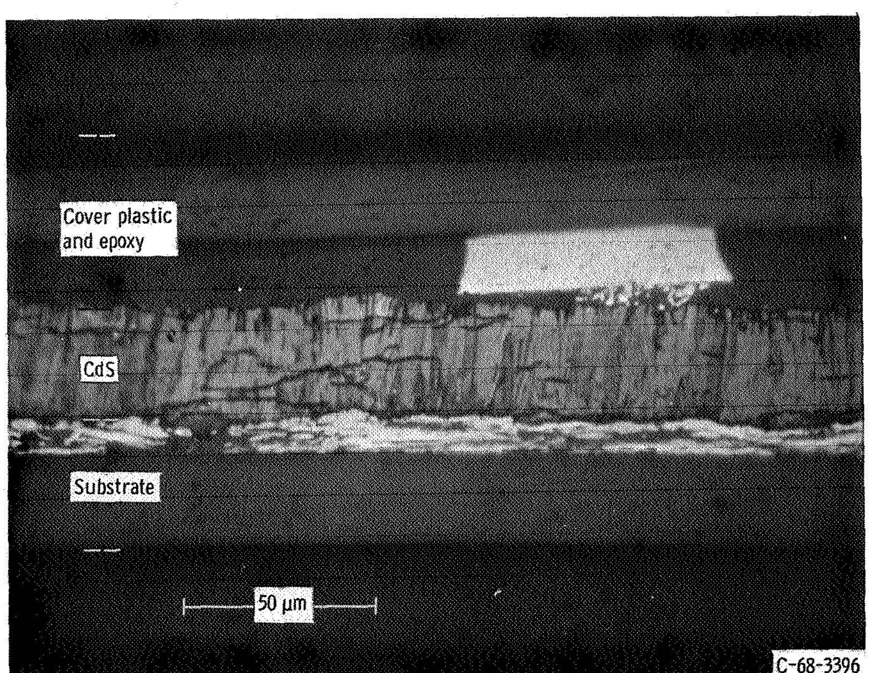


Figure 8. - Photomicrograph of cadmium sulfide cell after etching.

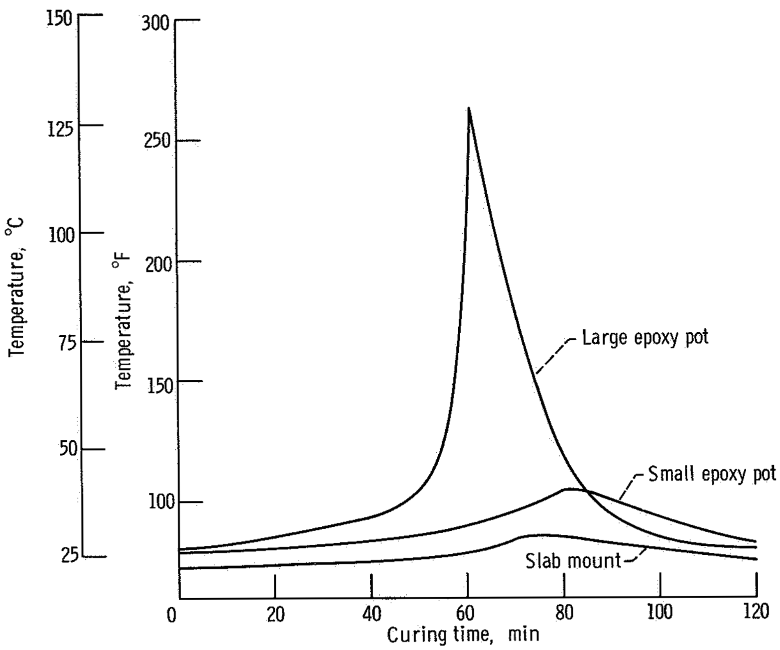


Figure 6. - Temperature-time profile of curing epoxy.

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