345
+180
30

hr. 1250

DSE-4042-T17

COMMERCIALIZATION OF THICK FILM SOLAR CELLS

Quarterly Progress Report for September 21–December 31, 1979

By
G. D. McDonald
G. Goodman

Work Performed Under Contract No. EG-77-C-01-4042

Globe-Union Inc.
Milwaukee, Wisconsin

MASTER

U.S. Department of Energy

Solar Energy

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COMMERCIALIZATION OF THICK FILM
SOLAR CELLS

Quarterly Progress Report
Sept. 21, 1979 to Dec. 31, 1979

By

G. D. McDonald, Principal Investigator
G. Goodman, Contract Manager

Globe-Union Inc.
Milwaukee, WI 53201

Prepared For:

SERI

Subcontract No. XS-9-8104-2
Prime Contract No. EG-77-C-01-4042

ABSTRACT

Starting materials for the preparation of thick film cadmium sulfide and cadmium telluride solar cells have been comminuted. Initial trial films of cadmium sulfide showed that during the next phase of this work, the printing of films, one of the major problem areas will be to obtain sufficient reflow in the printed films to remove the screen-caused variation in film thickness. The thin areas corresponding to the screen pattern caused pinholes to form in the fired parts.

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1.0. Introduction

The objective of this program is to evaluate the use of screen printing as a technique for producing large area solar cells. The attributes of screen printing which make this approach of interest is that screen printing is a well established technique in the electronics industry with well established requirements, has a demonstrated capability for producing large areas of electronic quality films with good fidelity, provides a means for separating the conditions required for film deposition from those required for sintering, providing control, for example, over microstructure, and has a high material utilization efficiency. A possible disadvantage of screen printing may be that the starting material must be reduced to a powder form and that the ultimate lower film thickness that can be reliably formed may be greater than that obtainable by other approaches. The fact that efficient small area solar cells can be produced by screen printing is well established in the literature⁽¹⁾.

The work of this contract is an investigation of the costs involved in preparing cadmium sulfide and cadmium telluride based solar cells using screen printing. This will allow an evaluation of this technique relative to competing technologies. The first step in this contract is to develop the capability of producing high quality semiconductor films on glass. Subsequent to this, the problems associated with multi-layer cells will be investigated.

2.0. Experimental

2.1. Materials

Five-nines purity cadmium sulfide and cadmium telluride were obtained from Materials Research Corporation. The cadmium sulfide was a highly structured, low density powder (through 360 mesh screen) and emission analysis, see Table 1 and 2, compared favorably with the analysis provided by the vendor, see Table 3.

Cadmium telluride was in one-eighth inch pieces which were reduced to a coarse powder by grinding in an agate mortar and pestle. Atomic absorption showed that this material contained 0.2 weight percent selenium and emission analysis, see Tables 4 and 5, showed that it contained approximately 0.002 percent copper, 0.020 percent iron, 0.008 percent nickel, and 0.002 percent silver. Materials Research Corporation has been contacted and are rerunning their analysis given in Table 6.

Cadmium chloride was obtained from Fisher Scientific Company. Emission analysis of this material is given in Tables 7 and 8 and shows no contamination by the elements analyzed for.

Glass substrates type 7059 were obtained from Corning Glass Works. They were cleaned in Micro brand cleaning solution manufactured by International Products Corporation. They were then rinsed in distilled water.

Clear acrylic spray type 65908 for coating the inner surfaces of the jet mill was obtained from Sears.

2.2. Equipment

A four-inch diameter fluid energy mill was obtained from the Fluid Energy Processing and Equipment Co., see Figure 1. A vibratory feed was fabricated to insure a constant feed rate.

Powdered materials were dispersed in organic vehicles using a Bunell-Castor dispersing mill equipped with three two-inch diameter rollers. The fineness of grind was measured on a FOG gauge manufactured by the Precision Gage and Tool Co. and the ink rheology determined on a Haake RV Rotovisko viscometer.

Thick films were screened on glass substrates using a Presco screen printer manufactured by Precision Systems Co. The films were fired in a three zone, controlled atmosphere, four inch belt kiln manufactured by the Lindberg Company.

Film resistances were measured using four screened-on silver terminations. Commercial silver ink was used, Electroday 415SS from Acheson Colloids Co., see Figure 2. Resistances were measured on a 5900 Dana Digital Multimeter. Contact resistance was investigated using a Model 575 Tektronix curve tracer. Film thickness was measured using a model 4 Taly Surf manufactured by Rank Taylor Hobson Company.

The scanning electron microscopy used to characterize the powders was a JEOL 35-C SEM equipped with an Ortec 4 mm diameter detector capable of resolving 150 ev. The unit has a NS 880 Traco Northern data handling system and is the property of Midwest Research Microscopy. All magnifications were determined by using 1.091 micron diameter latex spheres from Ernest F. Fullam, Inc.

Emission analysis was performed on a Hilger-Engis Model 1000 monochromator equipped with a DC Baird Atomic arc and an RC-3 microphotometer automated plate reader. Atomic absorption analysis was performed on a Perkin-Elmer 403 spectrophotometer.

3.0. Technical Discussion

3.1. Comminution of Starting Materials

Work on this contract is in the initial phases. Work performed to date has been primarily to obtain and process the materials needed to prepare films of solar cell materials. The "99.999%" cadmium sulfide and cadmium telluride were received. The cadmium sulfide as received consisted of fused agglomerates of micron particles, see Figure 3. To break up these agglomerates with the minimum of contamination jet milling was used. Quartz was run through the mill first to determine the operating characteristics and the amount of contamination which might be expected from the walls of the mill during milling. Emission spectrographic analysis of the milled quartz indicated a pickup of 0.24% iron, 0.12% aluminum, and 0.10% nickel. To decrease this contamination, the internal surfaces of the mill were coated with acrylic resin. Subsequent analysis of milled materials showed that this approach was reasonably successful.

The first run on the jet mill using cadmium sulfide went smoothly. Towards the end of the run air leakage was noted between the venturi nozzle and the feed hopper assembly. No blockage was found inside the equipment and the cause of the leakage was felt to be related to too fast a feed rate causing temporary blockage of the mill.

During the second run 800 grams were fed into the mill in 4.5 hours. Leakage was again noted in the same area mentioned above and only 400 grams were recovered. The manufacturer of the mill recommended a decrease in the feed rate or a reduction of the grinding nozzle pressure relative to the feed pressure at the venturi. Further study of the mill showed that the actual source of the leakage was the result of too great a tolerance between the nozzle and its housing. By applying a small quantity of silicon rubber to this gap this problem was resolved. On the next run 1035 grams of CdS were processed in two hours and

20 minutes. Of the material milled only 10 grams were lost, less than one percent.

Microscopic examination of the milled cadmium sulfide showed that the agglomerates had been successfully broken up, see Figure 4. Analysis of the milled cadmium sulfide, see Tables 9 and 10, showed only a pickup of an average of 115 ppm of iron. The average particle size was about 1.6 micron.

The higher density and more brittle nature of the cadmium telluride compared to cadmium sulfide made jet milling considerably easier. In one hour and 53 minutes 875 grams were milled. Recovered was 855 grams, a loss of 2.3 percent.

The particle size of the cadmium telluride was measured by ultrasonically suspending some of this material in freon and subsequently placing a drop of the dilute slurry on an aluminum stub. A dilute aqueous suspension of the latex spheres was sprayed onto the stub and allowed to dry. SEM pictures were taken of the dispersed powder after carbon coating. The magnification was chosen to allow easy measurement of at least 200 particles on a total of five photographs. SEM photographs of the latex spheres were taken for each sample stub, stage position, and magnifications. A grid, whose spacing does not exceed the dimensions of the larger particle was placed over the photograph. An unbiased selection of particles to be measured was made by only using particles that touch the lines of the grid.

The statistical linear diameter^(2,3,4) was then measured for each particle used - the average subjective linear diameter through the maximum cross sectional area. The errors introduced by this subjective average are small⁽⁴⁾ (normally two percent) compared to the other measurements.

The particle dimensions were then translated into real particle dimensions using the actual magnification found from the latex spheres. The largest source of error is generally from the uncertainty in the magnification which is the sum of the uncertainties of the dimensions of the spheres and the astigmatism

of the SEM. The particle sizes were then graphed as a function of frequency of occurrence over various size intervals against diameter size on a histogram⁽⁵⁾ or fitted to a smooth number frequency distribution function. The most common choices are the log-normal^(6,7) and Rosin Rammler⁽⁸⁾ distributions and may be empirically treated for their applicability to each powder.

A typical SEM photograph of cadmium telluride is shown in Figure 5. A histogram of the particle size distribution for cadmium telluride is given in Figure 6. The data obtained could not be fitted to a Rosin-Rammler distribution. A log normal plot, see Figure 7 clearly shows a bimodal distribution indicating that the milling operation broke up the cadmium telluride into smaller pieces and into agglomerates.

Milling of cadmium chloride went smoothly with 461 grams being milled in 1.5 hours. Recovered were 397 grams, a loss of 14 percent. SEM photographs, see Figure 8, shows an approximate particle size of five micron with particle sizes ranging from one micron to approximately ten microns.

3.2. Preparation of Layers of Cadmium Sulfide

Initial trial films of cadmium sulfide were prepared. The composition of the vehicles used in the inks used to screen these films are considered company proprietary and will be referred to by a letter code.

The first ink used a vehicle consisting of 24 weight percent AB, 38 weight percent BCA and 38 weight percent AT. The ink was prepared by premixing by hand 20 weight percent of the above vehicle with 80 weight percent cadmium sulfide. This mixture was then dispersed on the three roll mill. The fineness of grind for this formulation was measured to be 41 micron and therefore suitable for printing through a 325 mesh screen. A one centimeter by two centimeter pattern was then screen printed onto a glass substrate, see Figure 2, and fired at 700°C using the kiln profile shown in Figure 9. The layer formed after firing was dark brown

and was not adherent to the substrate. The substrate was etched where the cadmium sulfide had been in contact with it. A lower firing temperature was then used. A 630°C firing profile was used, see Figure 10. The films were again dark in color but adherent to the substrate. The thickness of the films was 25 micron as determined by using the Taly Surf. Four silver electrodes were screened onto the film, see Figure 2, to give a one square pattern (one centimeter by one centimeter). The average resistivity of the films were 0.24 ohm cm. The silver contacts were non-ohmic with an average contact resistance of 15 ohms.

The dark color of the films was thought to be due to a carbon residue remaining after the firing. A new ink was prepared containing 20 weight percent AT and 80 weight percent cadmium sulfide. This ink was printed and fired at 630°C using the same profile as before. The fired film was again brown in color and had resistivity of 1.45 ohm cm.

An ink containing 20 weight percent EGBEA and 80 weight percent cadmium sulfide was printed and fired at 630°C. The resulting tan film had a resistivity of 2.8 ohm cm. A 300°C air prefiring or a 190°C vacuum firing before the 630°C firing had no effect on the color of the films formed nor their resistivity.

Additions of cadmium chloride to the formulations had no effect on the resultant appearance of the fired films. An ink containing 20 weight percent AT, five weight percent cadmium chloride, and 75 weight percent cadmium sulfide was printed and fired at 630°C. The resistivity of this film was 0.02 ohm cm.

At this point in time it became apparent that the brown coloration probably was not due to residual organic matter. Investigation of the kiln showed that during a recent move the kiln had been converted to the compressed air line rather than the nitrogen line. Rectification of this error is in progress.

All the film produced to date retained on their surfaces the imprint of the screen mesh. When fired the thinner areas of the films become pinholes. Future inks will contain lower solids loadings and more viscosity modifying components.

3.3. Safety

All cadmium containing materials were handled in an exhausted hood. All personnel working in the area were fit tested to approved high efficiency respirators by the Globe-Union Industrial Hygiene Department. Air samples were taken during the milling operation on 0.45 micron filters. Analysis showed that exposure levels of all persons monitored were less than the applicable OSHA limits for exposure without respirators. Norton earmuffs (4540) and Comfit ear plugs were used together during the milling operation.

4.0. Future Activity

Future work will involve firing of the previous formulations in nitrogen and modification of the vehicles to obtain greater post-screening flow. Layers of cadmium telluride will be prepared. Additions of indium to the silver termination ink will be made to effect ohmic contact to the films.

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6. ob cit 3 pp. 41-43.
7. ob cit 4 pp. 27-33, 36, 39-47.
8. ob cit 2 pp. 31-36.

Table 1. Emission Analysis of As Received Cadmium Sulfide

♦ ♦ ♦ G L O B E U N I O N ♦ ♦ ♦

♦♦♦ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ♦♦♦
 CORP. ADDRESS 1XE DATE 12/03/79
 PHONE 2366

PHOTO PLATE # 1623.24

MTL # 7911-0458 EXPOSURE 7

T&R SHEET # 9-11/124

ELEMENT CONCENTRATION
 IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ♦♦♦♦ COS-45-1 Cadmium Sulfide '99.999%'

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	< .002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	< .003
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	< .003
78 PLATINUM	< .033
14 SILICON	< .002
47 SILVER	< .0010
11 SODIUM	< .050
50 TIN	< .003
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

Table 2. Duplicate Emission Analysis of As Received Cadmium Sulfide

♦ ♦ ♦ G L O B E U N I O N ♦ ♦ ♦

♦♦♦ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ♦♦♦
 CORP. ADDRESS 1XE DATE 12/03/79
 PHONE 2366

PHOTO PLATE # 1623.24

MTL # 7911-0458

EXPOSURE 8

T&R SHEET # 9-11/124

ELEMENT

CONCENTRATION
 IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ♦♦♦♦ CDS-45-1 Cadmium Sulfide "99.999 %"

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	< .002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	< .003
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	< .003
78 PLATINUM	< .033
14 SILICON	< .002
47 SILVER	< .0010
11 SODIUM	< .050
50 TIN	< .003
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

materials analysis certificate

PLEASE FORWARD WITH MATERIAL

Table 3. Materials Research Analysis of Cadmium Sulfide

Customer GLOBE-UNION, INC. P/O No. 10-43404

MRC No. 31626 Date 10/3/79

Material CADMIUM SULFIDE Alloy Composition CdS

Form POWDER Grade 99.999% Lot No. 31626

Dimension P/N 48-111

Fabrication HOT PRESS

Special Cleaning & Packaging: UNDER CLEAN ROOM CONDITIONS.

Special Handling Precautions: _____

Analysis:

☐ Mass Spectrographic ☐ Leco Method (Gases) ☐ Emission Spectrographic ☐ Conductometric (Carbon) ☐ Neutron Activation

TYPICAL ANALYSIS

<u>ELEMENT</u>	<u>PPM</u>
CO	.1
FE	.5
NI	.5
PB	5.0
SO ₄	.05%
CL	.015%
AL	<10.
MG	<10.
SI	<10.
ZN	<10.

DATE: 10/4/79

MATERIALS RESEARCH CORPORATION

[Signature]

Manager, Quality Assurance

**MATERIALS RESEARCH
CORPORATION**

Orangeburg, New York 10962

Table 4. Emission Analysis of Cadmium Telluride

◆ ◆ ◆ G L O B E U N I O N ◆ ◆ ◆

◆◆◆ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ◆◆◆
CORP. ADDRESS 1XE DATE 12/27/79
PHONE 2366

PHOTO PLATE # 1650.51

MTL # 7912-0194 EXPOSURE 7

T&R SHEET # 9-12/40

ELEMENT CONCENTRATION
IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ◆◆◆◆ COTE - U3-1

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	.002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	.012
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	.008
78 PLATINUM	< .033
~~14 SILICON~~	~~< .002~~
47 SILVER	.002
11 SODIUM	< .050
~~50 TIN~~	~~.013~~
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

*cannot measure Si due to
Cd interference, also Sn.*

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

Table 5. Duplicate Emission Analysis of As Received Cadmium Telluride

◆ ◆ ◆ G L O B E U N I O N ◆ ◆ ◆

◆◆◆ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ◆◆◆
 CORP. ADDRESS 1XE DATE 12/27/79
 PHONE 2366

PHOTO PLATE # 1650.51

MTL # 7912-0194 EXPOSURE 8

T&R SHEET # 9-12/40

ELEMENT CONCENTRATION
 IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ◆◆◆◆ CODE - UJ-1

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	.002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	.027
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	.007
78 PLATINUM	< .033
~~14 SILICON~~	~~< .002~~
47 SILVER	.002
11 SODIUM	< .050
~~50 TIN~~	~~.010~~
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

*cannot measure di due to
 Cd interference, also Sn*

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

materials analysis certificate

PLEASE FORWARD WITH MATERIAL

Table 6. Materials Research Corporation Analysis of Cadmium Telluride.

Customer GLOBE-UNION P/O No. 10-43404
MRC No. 31626 Date 10/3/79

Material CADMIUM TELLURIDE Alloy Composition CdTe

Form PIECES Grade 99.999% Lot No. 31626-1

Dimension 1/8" PIECES P/N 48-114

Fabrication HOT PRESS

Special Cleaning & Packaging: UNDER CLEAN ROOM CONDITIONS

Special Handling Precautions: _____

Analysis:

☐ Mass Spectrographic ☐ Leco Method (Gases) ☐ Emission Spectrographic ☐ Conductometric (Carbon) ☐ Neutron Activation

TYPICAL ANALYSIS

ELEMENT	PPM
TI	0.001
CR	0.01
W	0.01
MN	0.1
FE	0.2
CO	0.1
NI	0.1
CU	0.1
ZN	0.5
AL	0.5
SI	1.0
SE	1.0
AS	ND

MATERIALS RESEARCH CORPORATION

DATE: 10/4/79

R. Salomaggi

Manager, Quality Assurance

**MATERIALS RESEARCH
CORPORATION**

Orangeburg, New York 10962

Table 7. Emission Analysis of Cadmium Chloride

♦ ♦ ♦ G L O B E U N I O N ♦ ♦ ♦

♦♦♦ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ♦♦♦
CORP. ADDRESS 1XE DATE 01/04/80
PHONE 2366

PHOTO PLATE # 1669.7

MTL # 7912-0489

EXPOSURE 3

T&R SHEET #

ELEMENT

CONCENTRATION

IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT: ♦♦♦♦♦

Cd Cl₂ Jm-1

---

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	< .002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	< .003
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	< .003
78 PLATINUM	< .033
14 SILICON	< .002
47 SILVER	< .0010
11 SODIUM	< .050
50 TIN	< .003
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

Table 8. Duplicate Emission Analysis of Cadmium Chloride

♦ ♦ ♦ G L O B E U N I O N ♦ ♦ ♦

♦♦♦ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ♦♦♦
CORP. ADDRESS 1XE DATE 01/04/80
PHONE 2366

PHOTO PLATE # 1669.7

MTL # 7912-0481. EXPOSURE 4 T&R SHEET #

ELEMENT CONCENTRATION
IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ♦♦♦♦ CdCl2 JM-1

---

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	< .002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	< .003
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	< .003
78 PLATINUM	< .033
14 SILICON	< .002
47 SILVER	< .0010
11 SODIUM	< .050
50 TIN	< .003
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

THIS ANALYSIS HAS BEEN CORRECTED FOR ANY CONTAMINATION INTRODUCED BY THE REAGENTS USED IN THE SAMPLE PREPARATION. THIS INFORMATION IS AVAILABLE UPON REQUEST.

Table 9. Emission Analysis of Milled Cadmium Sulfide

♦♦♦ MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ♦♦♦
 CORP. ADDRESS 1XE DATE 12/04/79
 PHONE 2366

PHOTO PLATE # 1623.24

MTL # 7911-0458A EXPOSURE 9 T&R SHEET # 9-11/124

ELEMENT CONCENTRATION
 IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ♦♦♦♦ CDS-JM-1 Cadmium Sulfide "99.999%"

13 ALUMINUM	< .003
51 ANTIMONY	< .010
33 ARSENIC	< .100
56 BARIUM	< .100
83 BISMUTH	< .010
20 CALCIUM	< .050
48 CADMIUM	> 1.000
24 CHROMIUM	< .003
29 COPPER	< .002
27 COBALT	< .005
79 GOLD	< .010
26 IRON	.014
25 MANGANESE	< .0010
12 MAGNESIUM	< .003
80 MERCURY	< .100
28 NICKEL	< .003
78 PLATINUM	< .033
14 SILICON	.111
47 SILVER	< .0010
11 SODIUM	< .050
50 TIN	< .003
22 TITANIUM	< .010
23 VANADIUM	< .003
40 ZIRCONIUM	< .010
30 ZINC	< .050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

Table 10. Duplicate Emission Analysis of Milled Cadmium Sulfide

*** MATERIALS TEST LABORATORY SPECTROGRAPHIC REPORT ***
 CORP. ADDRESS 1XE DATE 12/04/79
 PHONE 2366

PHOTO PLATE # 1623.24

MTL # 7911-0458A

EXPOSURE 10

T&R SHEET # 9-11/124

ELEMENT

CONCENTRATION
 IN % BY WT OF SAMPLE AS RECEIVED BY MTL

CUST. IDENT. ***** CDS-JM-1 Cadmium Sulfide "99.999%"

13 ALUMINUM	<	.003
51 ANTIMONY	<	.010
33 ARSENIC	<	.100
56 BARIUM	<	.100
83 BISMUTH	<	.010
20 CALCIUM	<	.050
48 CADMIUM	>	1.000
24 CHROMIUM	<	.003
29 COPPER	<	.002
27 COBALT	<	.005
79 GOLD	<	.010
26 IRON	<	.009
25 MANGANESE	<	.0010
12 MAGNESIUM	<	.003
80 MERCURY	<	.100
28 NICKEL	<	.003
78 PLATINUM	<	.033
14 SILICON	<	.134
47 SILVER	<	.0010
11 SODIUM	<	.050
50 TIN	<	.003
22 TITANIUM	<	.010
23 VANADIUM	<	.003
40 ZIRCONIUM	<	.010
30 ZINC	<	.050

THE ACCURACY OF THESE RESULTS AT THE 95% CONFIDENCE LIMIT IS APPROXIMATELY + OR - 50% OF THE AMOUNT PRESENT AT THE .01% LEVEL. THE ACCURACY DECREASES SUBSTANTIALLY AS THE ELEMENT CONCENTRATION APPROACHES THE CURRENT SPECTROGRAPHIC DETECTION LIMIT.

Figure 1. Fluid Energy Jet Mill.

Figure 2. Cadmium Sulfide Film.

10,900 $\pm$ 200x

Figure 3. As Received Cadmium Sulfide

10,900 $\pm$ 200x

Figure 4. Milled Cadmium Sulfide

10,000 X

Figure 5. Milled Cadmium Telluride

Figure 6. Histogram of Particle Size Distribution for Milled Cadmium Telluride.

Figure 7. Log Normal Plot of Milled Cadmium Telluride.

10,000 X

Figure 8. Milled Cadmium Chloride

Figure 9. Kiln Profile for 700°C Firing.

Figure 10. Kiln Profile for 630°C Firing.