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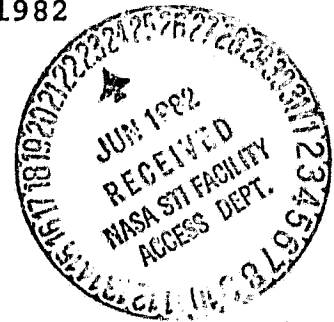
PROCESS RESEARCH ON SEMIX
SILICON MATERIAL (PROSSM)

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December 1, 1981 - February 28, 1982

Contract No. 955902



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PROCESS RESEARCH ON SEMIX
SILICON MATERIAL (PROSSM)

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December 1, 1981 - February 28, 1982

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The JPL Flat-Plate Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initial a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.

BY

John H. Wohlgemuth
Donald B. Warfield

SOLAREX CORPORATION
1335 Piccard Drive
Rockville, MD 20850
(301) 948-0202

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QUARTERLY REPORT
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ABSTRACT

During this quarter the emphasis of this contract was shifted from the development of a cost-effective process sequence to research designed to understand the mechanisms of photovoltaic conversion in semicrystalline silicon. With this change has gone a change of title from Module Experimental Process System Development Unit (MEPSDU) to Process Research of Semix Silicon Material (PROSSM). Efforts are now underway to prepare a revised program plan with emphasis on determining the mechanisms limiting voltage and current collection in the semicrystalline silicon.

The efforts reported in this quarterly report concern work done before the change in emphasis and so it reports on the continued development of the cost-effective process sequence.

While the automated process sequence effort has now been terminated a summary report of this phase will be prepared. The following is a summary of the most important developments in this phase:

1. A cost-effective process sequence was identified, equipment was designed to implement a 6.6 MW per year automated production line, and a cost analysis projected a \$0.56 per watt cell add-on cost for this line.

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2. Four process steps were developed for this program:

- Glass bead back clean-up.
- Hot spray antireflective coating.
- Wave-soldering of fronts.
- Ion milling for edging.

3. While spray dopants were advertised as an off the shelf developed product, they proved to be unreliable with shorter than advertised shelf life.

4. Equipment for handling and processing solar cells is available for all of the cell processing steps identified in this program.

During this quarter efforts included work on spray dopant, edging, AR coating, wave soldering and fluxing, ion milling and cost analysis.

1.0 INTRODUCTION

During this quarter the emphasis of this contract was shifted from the development of a cost-effective process sequence to research designed to understand the mechanisms of photovoltaic conversion in semicrystalline silicon. With this change has come a change of title from Module Experimental Process System Development Unit (MEPSDU) to Process Research of Semix Silicon Material (PROSSM). Efforts are now underway to prepare a revised program plan with emphasis on determining the mechanisms limiting voltage and current collection in the semicrystalline silicon.

The efforts reported for this Quarter concern the MEPSDU program to verify cell process steps and the adaptability of these steps to the use of automated equipment within the overall DOE flat-plate photovoltaic price goal of less than \$0.70 per peak watt in 1986. The efforts underway in this area were:

- Design and development of a detailed cost effective cell process sequence.
- Identification of automated equipment capable of performing this process sequence.

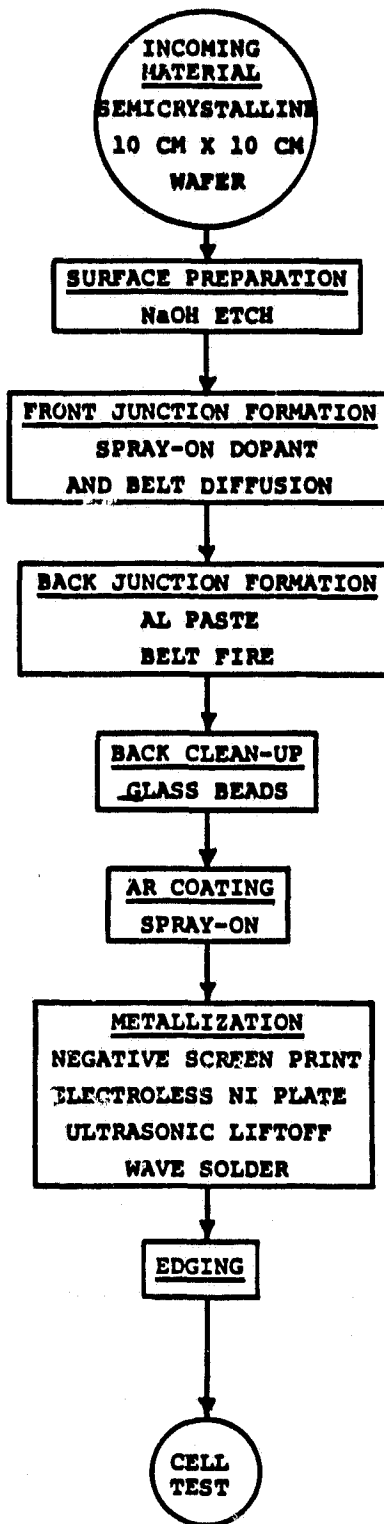
- Verification of the process sequence and determination of the expected performances.
- Performance of a cost analysis of the process sequence, including a study of the cost impact and changes required in the MEPSDU to allow the use of different types of input material.

A process sequence has been identified that we believe can meet all of the goals. This process sequence is shown in Figure 1. This process sequence was described in detail in the First Quarterly Report (1) and the modifications described in the subsequent Quarterly Reports (2,3,4). The baseline process sequence includes the following features:

- Semicrystalline silicon (10cm x 10cm) as the silicon input material;
- Spray-on dopant diffusion source;
- Belt diffusion;
- Al paste BSF formation;
- Glass bead back clean-up;

FIGURE 1

GENERAL PROCESS
DESCRIPTION



- Spray-on AR coating;
- Electroless Ni plate-solder metallization; and
- Edging - Ion Milling.

To date the program has been successful in developing four new processes that can reduce cell cost and improve efficiency.

The three are:

1. AR Coating - A new technique involving the spraying of pure titanium isopropoxide onto heated wafers resulted in a significant improvement in AR coating uniformity especially for less smooth surfaces. This technique should be less expensive than previous spray methods because the use of solvents has been eliminated.
2. Glass Bead Back Clean-Up - The use of glass bead back clean-up not only eliminated the need for costly chemicals, but also allows the Al to serve as the prime current carrier on the back of the cell, saving the cost of having to apply additional metal (such as solder) to provide for current collection.
3. Wave-Soldering - Using wave-soldering to apply the front pattern solder allows more control of the process,

reduces the amount of solder utilized and makes the solder step more adaptable to automation.

4. Ion Milling - Stacking several hundred wafers and ion milling the edges promises to be a very useful edge isolation technique. Materials usage and breakage should be negligible and the need for elaborate diffusion masking or edge following is eliminated entirely.

Disappointing results have been obtained using the spray-on dopant for diffusion. While some laboratory experiments have been very successful the short inconsistent shelf life and variation in properties from batch to batch have led us to the conclusion that the Emulsitone Phosphorofilm for Solar Cells is not ready for production at this time. Further work is required in this area. The details on the experiments performed in this quarter are given in Section 2.0.

A cost estimate has been completed for a 6.6 MW per year automated production line. The result was a projected cell add-on cost of \$0.56 per watt. The details are presented in Section 3.0.

2.0 TECHNICAL PROGRESS

2.1 Documentation

The following reports were submitted during this quarter:

- 22 Quarterly Report No. 4
- 23 Technical Progress Report No. 13
- 24 Technical Progress Report No. 14
- 25 Technical Progress Report No. 15

2.2 Spray Dopant Optimization

The Emulsitone Phosphorofilm spray dopant used on this program has proven its capability to produce good cells - but, not reproducibly. Its chief problem has been, notably, its limited shelf life which appears to be about one month for any useful application (see October and November 1981 Monthly Technical Reports).

The shelf life problem manifests itself in the reduced ability of the dopant to wet the surface of the wafers and the presence of an acetic acid smell. A proposed hypothesis for this degeneration is an attack by the phosphoric acid on the acetate groups which remain on the polyvinylalcohol from its original

manufacture by hydrolysis (typically incomplete) of polyvinyl acetate. This attack of the acetate groups would produce acetic acid with its characteristic smell and a change in the wetting characteristics of the polymer.

An inhouse attempt to develop a formulation without these problems was begun, but unfortunately, had produced no results at the time the Stop Work Order was received.

The problem of the short shelf life of the dopant is a serious one. During the month of February two separate batches of the dopant were found to have deteriorated beyond use. Any future evaluations of its use should consider the possibility of on-site preparation of the dopant solution.

2.3 Edging Experiments

The candidate oxide forming materials at the end of last Quarter's work were:

- Titanium Isopropoxide
- Titanium Chloride
- Niobium Chloride
- Chromium Trioxide

All the previous tests involved hand painting a thin layer of the paste onto a wafer. Since this is hardly an automatable process the following experiments were performed by screen printing a narrow ($\approx 3/16$ ") annulus on the periphery of the front surface.

It was decided at this time to eliminate the Chromium Tri-oxide because pastes prepared with it were not stable. They polymerized in a matter of minutes and consequently created problems in screen printing.

Cells were fabricated using:

- Titanium Isopropoxide
- Titanium Chloride
- Niobium Chloride

Experimental controls were fabricated with no annulus.

The controls and the cells produced using Titanium Chloride and Niobium Chloride were shunted. The oxide made with the Titanium Isopropoxide however showed some promise of being a good diffusion mask.

The experiment above was performed with the annulus protected by Colonial resist ink. An additional group of cells was fabricated with an exposed annulus to determine whether it would be

plated by the nickel bath. The annulus did not plate even after etching with fuming HF.

Continuing efforts in this area were to include optimization of a Titanium Isopropoxide paste formulation with attention given to screenability of the paste and uniformity of the resulting oxide.

2.4 AR Coating Experiments

Experiments this quarter including defining the temperature range and time needed to produce a satisfactory AR coat using hand-sprayed titanium isopropoxide. The initial problems were identifying a suitable heating unit and finding an adequate means to measure and control its surface temperature.

Three temperature-measuring methods were evaluated. The "Heat Spy" detector measures IR emission from surfaces, and was both inaccurate (non-reproducible) and much too sensitive to the nature of the surface (its emissivity). A simple bimetallic sensor was adequate for temperatures below 300°C, but was inaccurate above 350°C, with an error of +10 to +25%. The best method was found using a chromel-alumel (type K) thermocouple encased in a stainless steel probe, with a Keithley LCD readout. This method gave stable readings and reproducible results as long as the

probe was in good contact with the surface.

Several hotplates were evaluated for uniformity of temperature, with the large Corning hotplate showing the most even temperature ($\pm 5^{\circ}\text{C}$ reproducibility was possible at 405°C across an area 5×6 "). Both the small Corning hotplate and the Fisher hotplate had hot spots of up to 50°C hotter than the surrounding surface. An aluminum platen, ($3/8$ " thick) was used to attempt to even out these hot spots, but the effect was to reduce the attainable surface temperatures below 300°C , which does not produce an adequate AR coat.

The time-temperature matrix originally proposed was based on temperatures measured with the bimetallic surface thermometer. Using the chromel-alumel thermocouple, however, the target range was lowered. The matrix developed ranged from 280 - 425°C in temperature, and from 5 seconds to 90 seconds. The conditions which produced an acceptable AR coat were achieved within fairly narrow limits: Temperature range - 400 - 410°C , with 405°C optimum; time - 45 second wafer preheat (with wafer in place on heated substrate) followed by 5 second (approx) spray time, with no post-spray heating.

The most typical results of spraying at too low a temperature and/or insufficient preheat time was the presence of uneven and

spotty second-order droplets interspersed with variable-sized regions of first-order AR. The most typical result of spraying at too high a temperature and too much preheat time was a smooth, second or third order oxide (gold, green), which was partially or totally impervious to fuming HF.

Finally, the condition of the spraying apparatus was important. The airbrush (Badger 200-HD) must be absolutely clean. Replacement of the teflon needle bearing and head bearing is required at fairly frequent intervals to produce a smooth, fine spray stream.

2.5 Metallization

The Solarex-built foam fluxer (see Figure 1) was completed and installed on the wave soldering apparatus. Its performance was evaluated using about 25 wafers from this program and about 300 wafers from one of the production lines. Its performance was equal to or better than the previously used brush or dip methods with the added benefits of increasing throughput and by adjusting the level of the foam column, the amount of solder on the backs of the cells can be minimized.

The fluxer is simple in construction being made of 1/4" thick PVC stock welded into a tank approximately 8"x8"x12" wide. In

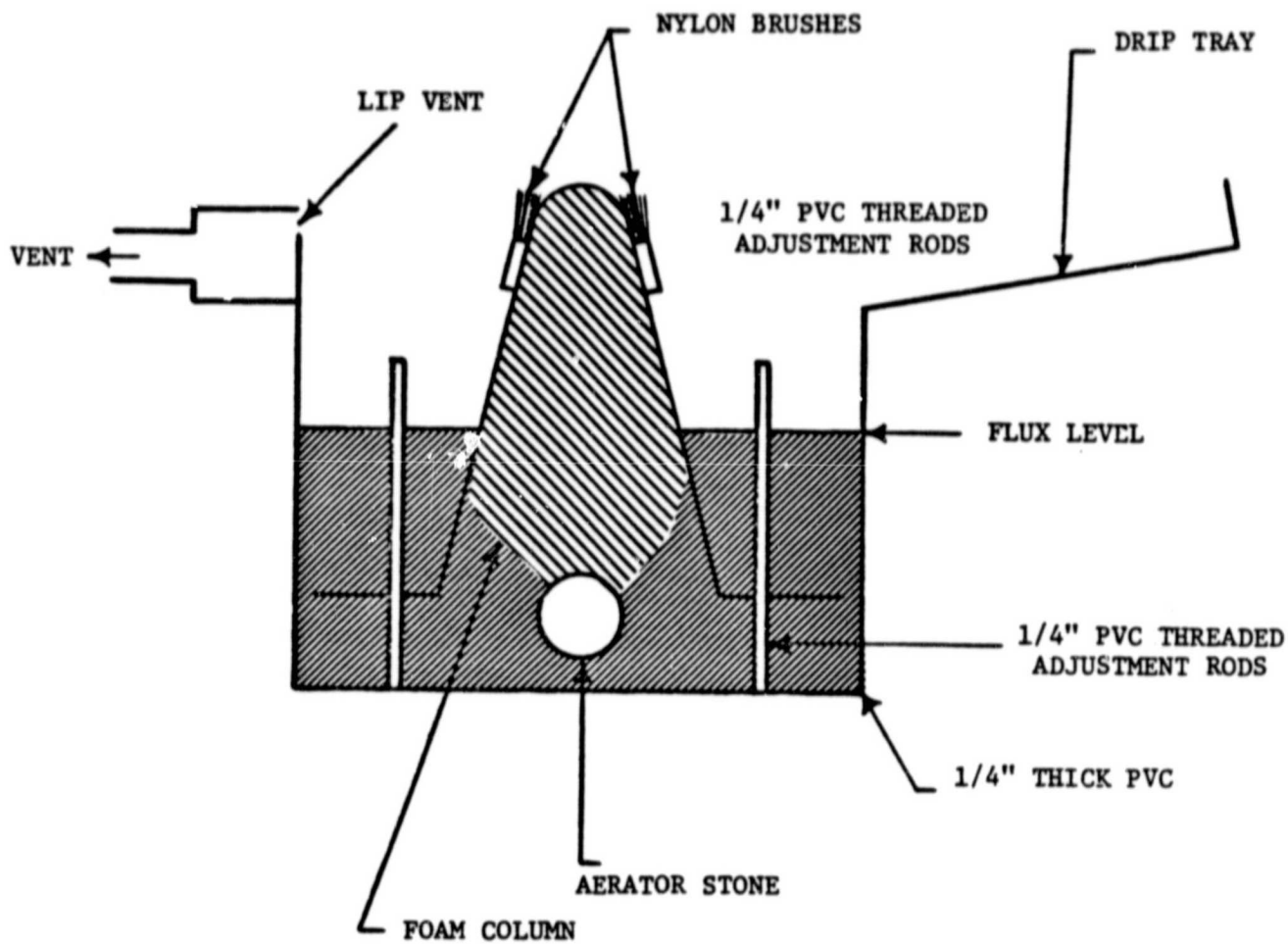


FIGURE 1

FOAM FLUXER - CROSS SECTION

the bottom of this tank the aerator stone is installed (Electro-vert #6-0903-003-02-1). This is then covered by a flattened cone of PVC whose top aperture is 1"x5" (wide enough to flux the entire cell in one pass). The top of the cone is fitted with nylon bristled brushes on either side to help maintain the column of foam.

In operation, the fluxer tank is filled approximately half full and the aerator stone is pressurized with air at \approx 2-3 psi. A column of foam will then develop over the aerator stone, rise and flow out of the top of the flattened cone. The whole assembly is placed under the wave transport system so that a wafer on the transport system will pass through the foam and be completely wetted.

The fluxes used were Alfa #'s 850-41 and 850-25. The former did not foam well enough, but the latter, when diluted to the lowest specific gravity within the manufacturers recommendations, foamed sufficiently and performed well as a flux. The 850-25 flux was used on the previously mentioned test wafers.

2.6 Ion Milling

Experimentation with ion milling was reported in July and August 1981. All sample groups exhibited a low shunt resistance.

Samples, after having their edges sawn, still had the same low shunt resistance. Later evaluation of the silicon used for those experiments proved the silicon to be at fault. It was, therefore, decided to run this set of experiments again - this time with silicon of known performance.

Processing was started on cells for these experiments but was delayed considerably by failure of the spray dopant and consequently the experiments were incomplete at the time of the Stop Work Order.

3.0 SAMICS COST ANALYSIS

A cost basis has been determined for the 6.6 MW/yr output MEPSDU plot line. The calculations are based upon:

- 10% efficient cells
- 80% yields
- 3 shifts/day
- 345 days/yr operation
- Production of 1,000 good cells/hr

Table #1 lists the required materials, the amounts needed per wafer, the cost of the materials, the calculated cost per wafer, the number of wafers processed per year and the total item cost per year.

Table #2 lists the required utilities, their usage rate, the amount needed per wafer, the cost of the utilities, the cost per wafer, the number of wafers processed per year and the total item cost per year.

Table #3 presents equipment costs and lifetimes, square footage used, the number of workers needed, the direct labor costs, the total materials cost (from Table #1) and the total utilities cost (from Table #2).

Table #4 presents the cost data in a format which reflects the IPEG2 equation:

$$\text{cost} = \frac{(C_i^* \times \text{EQPT}) + (109 \times \text{SQFT}) + (2.1 \text{ DLAB}) + (1.2 \text{ MATS}) + (1.2 \text{ UTIL})}{\text{QUAN}}$$

(*Coefficients C_i are presented in Table #5) and tabulates the costs by category and process step.

The total cost of the 6.6 MW/yr MEPSDU cell pilot line has been determined by IPEG2 to be \$.5567/watt (exclusive of silicon).

TABLE #1

	AMT/WAFER	COST/AMT \$	COST/WAFER \$	# WFRS/YR	COST/YR \$
ETCH NaOH	.01 Kg	.18 Kg	1.8 E-3	8.25 E-6	14850
HCl	.0011 liter	1.13/l	1.2 E-3	8.25 E-6	10255
DIFFUSION Dopant	.64 cm ³	.0092/cm ³	6 E-3	8.15 E-6	48900
BSF Al Paste	.65 m ³	.002/gm	3.5 E-3	8.05 E-6	28175
Screens	5 E-5	55/ea	2.75 E-3	8.05 E-6	22138
Squeegee Blades	3.75 E-4	2.4/ea	8.3 E-4	8.05 E-6	6704
BACK CLEANUP Glass Beads	2 E-4 lbs	1/lb	2 E-4	7.95 E-6	1590
HF 70%	1. ml	.67/lb	2 E-3	7.95 E-6	15900
AR COAT Titanium Isopropoxide	.65 cm ³	.0143/cm ³	9.3 E-3	7.86 E-6	73098
PATTERN PRINT Colonial Ink	2 cm ³	.0131/cm ³	2.6 E-2	7.76 E-6	203310
Screens	1.7 E-4	55/ea	9.4 E-3	7.76 E-6	72556
Squeegee Blades	3.75 E-4	2.4/ea	8.3 E-4	7.76 E-6	6441
Ni PLATE HF 70%	1.0 ml	.67/lb	2 E-3	7.67 E-6	15340
Electroless Ni	.005 gal	5./gal	2.5 E-2	7.67 E-6	191750
Trichloroethylene	4.5 E-4 gal	21.73/gal	9.8 E-3	7.67 E-6	75000
ION MILLING Filaments	1.25 E-4	5./ea	6.2 E-4	7.5 E-6	4650
Oil	1.4 E-6 gal	100/gal	1.4 E-4	7.5 E-6	1050
Argon	4E-7 tank	60/tank	2.4 E-5	7.5 E-6	180
WAVE SOLDERING Solder	.1 cm ³	5.18/lb ³	8.6 E-3	7.33 E-6	63038
Flux	.5 cm	.002/cm	1 E-3	7.33 E-6	7330

TABLE #2

	USGAGE RATE	AMT/WAFER	COST/AMT \$	COST/WAFER \$	# WAFERS/YR	COST/YR \$	ELEC COST/YR \$	VENT COST/YR \$	AIR COST/YR \$	COOLING H ₂ O COST/YR \$	TAP H ₂ O COST/YR \$	DI H ₂ O COST YR \$
ETCH	Elect	.016 KW hr	.047/KW hr	7.5 E-4	8.25 E6	6204.	6204.					
	Vent	96 ft ³	.000047/ft ³	4.5 E-4	8.25 E6	3741.		3741.				
	Tap H ₂ O	.0064 ft ³	.0098/ft ³	6.3 E-5	8.25 E6	517.					517.	
	DI H ₂ O	.0079 ft ³	.263/ft ³	2.1 E-3	8.25 E6	17424.						17424.
DIFFUSION	Elect	.034 KW hr	.047/KW hr	1.6 E-3	8.15 E6	13024.	13024.					
	Vent	77.7 ft ³	.000047/ft ³	3.6 E-4	8.15 E6	2993.		2993.				
	Air	.729 ft ³	.00039/ft ³	2.8 E-4	8.15 E6	2290.			2290.			
BSF	Elect	19.5 KW	.0157 KW hr	7.4 E-4	8.05 E6	5940.	5940.					
	Vent	600 cf/m	29.2 ft ³	1.4 E-4	8.05 E6	1105.		1105.				
	Air	10 cf/m	.486 ft ³	1.9 E-4	8.05 E6	1526.			1526.			
BACK CLEANUP	Elec	2 KW	.00166 KW hr	7.8 E-5	7.95 E6	620.	620.					
	Vent	3000 cf/m	149 ft ³	7.0 E-4	7.95 E6	5567.		5567.				
	Air	272 cf/m	13.5 ft ³	5.2 E-3	7.95 E6	41856.			41856.			
	DI H ₂ O	9.6 cf/h	.008 ft ³	2.1 E-3	7.95 E6	16695.						16695.
ABC	Elect	31 KW	.125 KW hr	1.2 E-3	7.86 E6	9235.	9235.					
	Vent	1200 cf/m	59.8 ft ³	2.8 E-4	7.86 E6	2209.		2209.				
	Air	15 cf/m	.75 ft ³	2.9 E-4	7.86 E6	2299.			2299.			
RES PRINT	Elect	11.25 KW	.00956 KW hr	4.5 E-4	7.76 E6	3486.	3486.					
	Vent	600 cf/m	36.6 ft ³	1.4 E-4	7.76 E6	1116.		1116.				
	Air	10 cf/m	.51 ft ³	2.0 E-4	7.76 E6	1543.			1543.			
M1	Elect	34 KW	.012 KW hr	5.6 E-4	7.67 E6	4325.	4325.					
	Vent	7200 cf/m	372 ft ³	1.8 E-3	7.67 E6	13410.		13410.				
	DI H ₂ O	.26 cf/m	.0134 ft ³	3.5 E-3	7.67 E6	26979.						
	Cooling H ₂ O	.5 cf/m	.0258 ft ³	7.0 E-5	7.67 E6	534.				534.		
ION MILLING	Elect	7.5 KW	.0066 KW hr	3.1 E-4	7.5 E6	2326.	2326.					
	Air	.01 cf/m	.005 ft ³	1.9 E-7	7.5 E6	1.			1.			
	Cooling H ₂ O	.134 cf/m	.0070 ft ³	1.9 E-5	7.5 E6	141.				141.		
WAVE	Elect	14.25 KW	.0128 KW hr	6.0 E-4	7.33 E6	4409.	4409.					
	Vent	600 cf/m	32.4 ft ³	1.5 E-4	7.33 E6	1116.		1116.				
	Air	2 cf/m	.108 ft ³	4.2 E-5	7.33 E6	308.			308.			
	Tap H ₂ O	7 cf/m	.0063 ft ³	6.1 E-5	7.33 E6	452.					452.	
TEST	Elect	3 KW	.0028 KW hr	1.3 E-4	7.17 E6	943.	943.					
	Vent	100 cf/m	5.52 ft ³	2.6 E-5	7.17 E6	187.		187.				
TOTAL					5.6 E6	194521.	50512.	31444.	49823.	675.	969.	61098.

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TABLE #3

	EQUIP	EQUIP LIFETIME YRS	FT ²	WORKERS/SHIFT	DLAB	MATS/YR	UTIL/YR
ETCH	65,000	5	200	1	70400.	\$ 25105	\$ 27886
DIFFUSION	173,000	10	504	1	70400.	48900	18307
BSE FORM	83,000	10	328	1/2	33000.	57017	8571
BACK CLEANUP	138,000	3	288	1	70400.	17490	64738
AR COAT	104,000	10	224	1/2	33000.	73098	13743
RESIST PRINT	126,000	10	392	1	70400.	282307	6145
Ni PLATE	50,000	5	378	1	70400.	282090	45248
ION MILL	135,000	10	200	1	65686.	5880	2468
WAVE SOLDER	100,000.	5	400.	1	65686.	70368	6285
TEST	<u>55,000</u>	10	<u>150</u>	<u>2</u>	<u>150543.</u>	<u>0</u>	<u>1130</u>
	\$1,029,000		3064 ft ²	10	\$699,915./yr	\$862,255/yr	\$194,521/yr

TABLE #4

	Ci x EQUIP		FT ² x 109		2.1 x DLAB		1.2 x MATS		1.2 x UTIL		TOTALS
	6.6 E-6		6.6 E-6		6.6 E-6		6.6 E-6		6.6 E-6		
ETCH	.0064		.0033		.0224		.0046		.0050		.0417
DIFFUSION	.0136		.0083		.0224		.0089		.0033		.0565
BSF FORM	.0065		.0054		.0105		.0104		.0016		.0344
BACK CLEANUP	.0174		.0048		.0224		.0029		.0118		.0593
AR COAT	.0082		.0037		.0105		.0133		.0025		.0382
RESIST PRINT	.0099		.0065		.0224		.0513		.0011		.0912
Ni PLATE	.0049		.0062		.0224		.0513		.0082		.0930
ION MILL	.0106		.0033		.0209		.0011		.0004		.0363
WAVE SOLDER	.0098		.0066		.0209		.0128		.0011		.0512
TEST	.0043		.0025		.0479		.0000		.0002		.0549
	.0916		.0506		.2227		.1566		.0352		.5567

ALL COSTS ARE EXPRESSED IN DOLLARS

TABLE #5

EQUIPMENT LIFETIME	3	5	7	10	15	20
COEFFICIENT, C_i	.83	.65	.57	.52	.48	.46

4.0 SCHEDULE AND PLANS

The month of March will be spent preparing a new Program Plan and Cost Proposal to reflect the change of scope, name and budget.

A summary report will be prepared on Phase I to describe the progress and status of the MEPSDU Program.

Experimentation in line with the new scope will be renewed in late March.

REFERENCES

1. Solarex Corporation, A Module Experimental Process System Developmental Unit (MEPSDU), First Quarterly Report, February 1981, JPL Contract No. 955902.
2. Solarex Corporation, A Module Experimental Process System Developmental Unit (MEPSDU), Second Quarterly Report, May, 1981, JPL Contract No. 955902.
3. Solarex Corporation, A Module Experimental Process System Development Unit (MEPSDU), Third Quarterly Report, August, 1981, JPL Contract No. 955902.
4. Solarex Corporation, A Module Experimental Process System Development Unit (MEPSDU), Fourth Quarterly Report, November, 1981, JPL Contract No. 955902.