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**LSA LARGE AREA SILICON SHEET TASK: CONTINUOUS LIQUID FEED
CZOCHELSKI GROWTH**

Quarterly Report No. 10 for January—March 1980

By
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April 1980

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Menlo Park, California

U.S. Department of Energy

Solar Energy

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LSA LARGE AREA SILICON SHEET TASK
CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

QUARTERLY REPORT #10

January - March 1980

Program Manager: George Fiegl

Prepared by: Dorothea Walters

April 1980

SILTEC CORPORATION

Menlo Park, California

The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate 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.

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ABSTRACT

The purpose of this specific phase of the continuous liquid feed program is the design and development of equipment and processes in order to demonstrate the continuous growth of crystals, by use of the Czochralski method, suitable for producing monocrystalline silicon for use in solar cells. This involves the growth of at least 150 kgs of monocrystalline silicon ingots, 150 mm in diameter, obtained from a single growth container. Our approach to meeting this goal is to develop a furnace with continuous liquid replenishment to the growth crucible.

The most significant event occurring this quarter was the repeated demonstration of the polyrod feed mechanism, providing continuous melt replenishment to the meltdown chamber, subsequent transfer of this melt, and the simultaneous growth of silicon ingots in the growth chamber.

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1.0 INTRODUCTION

The overall purpose of this program, which began in October 1977, is to demonstrate continuous crystal growth via the Czochralski method; "continuous" being defined as the throughput of silicon producing 150 kg of single crystal material, 150 mm in diameter, from one common container.

The program under Phase II includes:

- . Furnace conversion for low pressure operating conditions.
- . Design, development and fabrication of continuous solid feed systems for silicon, in various forms, for the melt-down chamber.
- . Design, development and fabrication of the continuous liquid silicon feed system with a reusable heater and automatic feedback controls.
- . Demonstration of continuous crystal growth as defined above, with an after growth yield greater than 80%.
- . Definition of process control variables for continuous Czochralski growth.
- . Theoretical analysis of silicon sheet growth to be con-

sidered during design and development of the furnace.

During this past quarter, activities were directed primarily toward:

- . Repeated demonstration of the polyrod system, providing continuous melt replenishment to the meltdown chamber, transferring this melt, and simultaneously growing silicon ingots in the growth chamber.
- . Data were collected from several thermal profiles, providing valuable information toward the improvement in efficiency of CLF technology.
- . Improvements were made to the transfer tube system, further assuring unimpeded melt transfer.
- . An improved melt level sensor was installed on the CLF furnace.

2.0 PROGRESS

2.1 Transfer Tube System

After several unsuccessful transfer attempts with a smaller (6 mm) I.D. quartz tube, it became apparent that through the decrease in diameter, silicon solidification within the tube after priming was a major problem. It was necessary to decrease the diameter of both I.D. and O.D. of the quartz tube to allow enough space for both the complete transfer tube system in the 12-inch crucible as well as for the grown 150 mm ingot (from the same crucible).

The decision was made early this quarter to return to the larger 8 mm I.D. tube size. However, as these tubes were not immediately available for testing, we continued using the 6 mm I.D. tubes until the larger tubes on order finally arrived. In an effort to avoid the problem of solidification in the tube ends when using this smaller I.D., we enlarged the tube ends to 8 mm. This step proved to be quite a successful, if only temporary, remedy in preventing solidification of the silicon in the transfer tube.

Both rigid graphite and grafoil strip heaters were tested early in the quarter; testing was solely with the strip heaters during the latter part of the quarter. The return to grafoil strip heaters was originally the result of a delay in the shipment of the rigid graphite heaters. However, use

Transfer Tube System (Cont'd)

of the strip heater has managed to provide much valuable information while enabling us to continue with transfer experiments without the problem of temperature variations evident when using the rigid heater.

2.1.1 MoO₃ Buildup on Electrodes

During several of the recent runs, the formation of molybdenum trioxide became evident in the vicinity of the heater electrodes, thereby increasing resistance. In an effort to check this buildup, a new design was created, substituting carbon electrodes for the molybdenum (see Figure 1). As we have temporarily discontinued use of the rigid heaters and are using grafoil strips, we have not had an opportunity to test these new electrodes to date. (The buildup only seems to occur when using the rigid heaters.) However, they are on hand and will be tested in the near future.

Figure 1. Drawing of graphite electrodes on rigid heater.

2.2 Melt Transfer Process

The actual procedure of priming the tube was improved through means of a more efficient sealing method on the mounting flange (a grafoil strip seal) of the melt transfer system. Figure 2 depicts the current priming technique. A pressure pulse of 3 inches Hg initiates the melt transfer process. As a result, the melt level in the growth crucible rises, making contact with the end of the transfer tube and continues to rise according to the amount of $\Delta h/2$ until both melt surfaces reach the same level.

Figure 2. Illustration of improved priming technique.

2.3 Thermal Profiles of the Transfer Tube System

We experienced some difficulty last quarter in maintaining uniform temperature distribution along the path of melt transfer, manifesting itself in the form of melt solidification near the tube ends. In an effort to improve this function, modifications were made to the ends of the rigid graphite heater. In this manner, the temperature is increased at the tube ends, compensating for the increased loss of heat observed in these areas prior to the modifications.

But this modification was not the entire solution, so thermal profiles were conducted again this quarter using platinum/rhodium thermocouples by means of the following two methods:

- . Moving the junctions in small increments of 0.25 and 0.5 inch through the tube (this method could only be applied when temperatures were kept below 1400°C).
- . Fixing the thermocouple junctions in critical areas of the tube, especially at the end and near the electrodes, thereby enabling measurement of actual operating temperatures (above 1400°C). Figure 3 shows the Type B multijunction array at the recording point on the exterior of the furnace.

Thermal Profiles of the Transfer Tube System (Cont'd)

Figure 3. Type B multijunction array on exterior of CLF furnace chamber.

Temperature data collected from both methods were displayed on a Eurotherm temperature sensor and was subsequently plotted on a graph which indicated specifically the areas along the melt transfer system where temperatures were increased or diminished. Although a significant drop-off was evident near the tube end -- even with the modified rigid heater ends used in these experiments -- this was not considered to be serious. During the actual melt transfer process (in contrast to the "dry runs" used for the thermal profiles), the quartz tube ends, which would be submerged in the molten silicon and would transmit enough heat to the

Thermal Profiles of the Transfer Tube System (Cont'd)

areas unexposed to either heater or melt, would make the temperature gradient in this small region considerably less severe. This critical area, involving approximately 0.375 to 0.5 inch of tube length, must reach temperatures above 1420°C (overall) in order to avoid melt solidification.

2.4 Temperature of Transfer Tube

Earlier in the quarter, when conducting the numerous thermal profiles in order to establish tangible data reflecting temperature distribution along the path of the melt transfer system primarily to avoid incidence of melt solidification in the tube, we observed some arcing between the heater and the thermal insulation. In fact, this phenomenon was most often the final negative event occurring prior to total heater breakdown. Steps were taken to achieve a uniform temperature distribution along the entire length of the melt transfer tube, taking all facets of heat contribution (including the heat emanating directly from the molten silicon) into consideration.

The area surrounding the electrodes of the transfer tube displayed a major increase in temperature resulting from increased contact resistance. The electrode contact area to the heating element was enlarged to lower contact resistance.

The same instrumentation (chart recorder) on the console which records the melt level positions is also equipped to record variations in the temperature of the transfer tube system. These data are of great value in determining various melt transfer characteristics.

2.5 Polyrod Feed System

This quarter marked the first of several subsequent runs using the polyrod feed mechanism. All tests ran in successful harmony in that solid polysilicon in the form of a rod was continuously fed into the meltdown chamber and the melt continuously transferred from the meltdown to the growth chamber while an ingot was simultaneously grown. Melt levels in both crucibles were maintained at approximately 12 kgs per crucible -- monitored by the melt level sensor. The largest ingot produced totaled approximately 34 kgs at a growth rate averaging 3 to 4 kgs per hour. Of this total, 50% of this material was monocrystalline in nature.

In greater detail, the amplified signal from the melt level sensor of the growth chamber with direct proportional control was used to feed the polyrod at a precise predetermined rate. After the growth of approximately 20 kgs of ingot, the usual pull speed fluctuations typically observed in conventional CZ growth were considerably reduced to a level of ± 0.2 inch per hour. This is primarily a result of the attainment of thermal equilibrium in the growth chamber as well as operating near steady state conditions.

After the initial polyrod (weighing approximately 20 kgs)

Polyrod Feed System (Cont'd)

has been fed into the melt, growth of the ingot continues uninterrupted while melt replenishment is achieved by reducing melt volume in the meltdown crucible. In the meantime, a new polyrod is introduced via the vacuum gate valve over the meltdown chamber.

This process was successfully demonstrated in several runs, permitting unlimited continuous melt replenishment without effecting growth conditions for the crystal.

Figure 4 illustrates this procedure.

Figure 4. Diagram of polyrod feed mechanism for CLF furnace.

2.6 Melt Level Sensor

We have added an improved melt level sensor to the CLF furnace replacing the breadboard previously used. The new system is comprised of a printed circuit board, optical filter and position sensor, packaged in a heat-reflective housing.

The sensor circuit is currently monitored via a chart recorder located on the console. With the installation of this new circuitry, we find the system extremely sensitive in accurately recording data in precise detail. Using this instrumentation, we can immediately detect any melt stagnation within the tube -- providing the necessary edge in preventing melt transfer failures.

3.0 CONCLUSIONS

3.1 Equipment Control

In its present state, many functions of the CLF furnace are still manually controlled. While this has been suitable for demonstration of the technical features of the system, several of these functions must be handled automatically before full technical readiness is achieved.

3.2 Growth Demonstrations

Through data accumulated from thermal profiles, it is evident that the temperature distribution is remaining quite uniform along the melt transfer path. However, we will continue with numerous growth demonstrations in order to improve this and all other areas of the melt transfer system in order to reach the optimum design.

4.0 PROJECTION OF ACTIVITIES

4.1 Solid Polysilicon Recharging

Plans are to continue demonstration runs using the polyrod feed mechanism for recharging the CLF melt transfer system. All near-future transfer runs will incorporate the polyrod recharge system in order to accomplish our goals as outlined in the Program Plan.

4.2 Furnace Frame Extension

Activities were initiated during the final days of the quarter to extend the frame of the CLF system 27 inches directly over the growth chamber. This extension will be completed in April, thereby enabling us to pull a greater quantity of crystal material -- from 24 inches to an overall length of 48 inches above the gate valve, thus increasing cycle efficiency in the growth of crystals. Plenty of material is on hand for these projected runs.

4.3 Program Plan

Activities will continue toward accomplishment of the goals set forth in the Program Plan.

APPENDIX I

New Technology

No new technology was generated during this reporting period.

APPENDIX II

Summary of Characterization, Design, and Economic Analysis Data

No characterization, design or economic data were generated this quarter.

APPENDIX III

Milestones

TASK DESCRIPTION	PERFORMANCE SCHEDULE											
	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	
1. Modify the liquid feed (CLF) furnace												
A. Overall design	▲	▲	▲									
B. Reduced pressure operation 10 to 30 torr	▲	▲	▲									
C. Polyrod feed mechanism with feedback control system	▲	▲	▲									
D. Extend crystal lift mechanism to 60 inches above gate valve									▲			
2. Transfer tube system												
A. Design and development	▲	▲	▲	▲	▲							
B. Fabrication		▲	▲	▲	▲	▲						
3. Develop particle feed system												
A. Overall design (mechanical and electronic)	▲	▲	▲	▲	▲	▲						
B. Fabrication				▲	▲	▲	▲	▲				
4. Growth demonstrations												
A. Growth of one crystal 25 kg from one set-up with melt transfer							▲					
B. Growth of 4 crystals 25 kg mass each from one set-up with melt transfer								▲				
C. Growth of one crystal 50 kg mass from one set-up with melt transfer									▲			
D. Growth of 3 crystals 50 kg mass each from one set-up with melt transfer										▲	▲	
5. Economic analysis							▲					▲
6. Provide representative samples						▲	▲			▲	▲	
7. Support design and performance reviews			▲	▲								
8. Provide support personnel												
A. Project integration meetings, DOE-sponsored meetings & annual workshop				▲				▲				
B. Performance review meetings			▲			▲			▲			
9. Provide documentation	▲	▲	▲	▲	▲	▲	▲	▲	▲	▲		▲
10. Procure parts, material and services (as required)												

Milestone Legend: ▲ = Due Date; ▽=▲ = Start-Finish