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

Quarterly Report No. 11, April-June 1980

By
Dorothea Walters

MASTER

July 1980

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Siltec Corporation
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 #11

April - June 1980

Program Manager: George Fiegl

Prepared by: Dorothea Walters

July 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 phase of the continuous liquid feed program is the design and development of equipment and processes in order to ultimately demonstrate the continuous growth of crystals, by the use of the Czochralski method, suitable for producing monocrystalline silicon for use in solar cells. This involves the growth of at least 150 kg 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.

Demonstrations using the silicon polyrod feed mechanism continued this quarter providing continuous melt replenishment to the melt-down chamber, subsequent transfer of this melt, and the simultaneous growth of silicon ingots in the growth chamber.

The frame to the CLF furnace was extended in order to accommodate 50 kg crystal ingots above the gate valve, hence enabling us to accomplish our goal of 150 kg throughput.

Development work continued on various aspects of the melt transfer system.

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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-

INTRODUCTION (Cont'd)

sidered during design and development of the furnace.

During this past quarter, activities and events were centered around:

- . Extending the existing CLF furnace frame in order to increase crystal pull capacity, thus producing greater ingots and improving the economics of the CLF system.
- . Modifications incorporated into the silicon polyrod feed function -- primarily a result of the extension to the CLF furnace frame.
- . Developing the transfer tube system, including additional thermal profiles as well as the investigation of alternative materials for its construction.
- . A new design applied to the quartz tube in order to prevent melt from being drawn into the heater/insulator areas of the transfer system. This design is still under evaluation for efficacy and practicality.
- . Consistent runs in line with the current schedule demonstrating melt transfer and simultaneous continuous crystal growth.

2.0 RESULTS

2.1 Extension to CLF Furnace Frame

Early in the quarter, the existing CLF furnace frame was extended 27 inches over the growth chamber (see Figures 1 and 2). In this way, we have extended crystal pull capacity considerably -- from 24 inches to an overall length of 48 inches above the vacuum gate valve, thereby increasing cycle efficiency in the growth of crystals.

Fig. 1. Upward view of placement of CLF extension on existing frame.

Fig. 2. Horizontal view of extension to basic CLF frame.

Following the installation of the extension to the basic CLF furnace frame, it was immediately evident that the S.S. bellows located directly over the growth chamber would not traverse

Extension to CLF Furnace Frame (Cont'd)

the total distance to the extended top of the frame. As the bellows used on the CLF furnace are of special design and therefore custom ordered, immediate purchase of a modified replacement was impossible. (The spare bellows originally ordered for backup had recently been used over the meltdown chamber when the decision was made to use silicon polyrods to replenish the melt supply for these initial demonstration runs as opposed to silicon lumps or particles. This particular bellows now houses the polyrods during the continuous feed process.)

To remedy the situation of inadequate bellows, we decided to remove a section from the bellows over the meltdown chamber; the missing portion was replaced with a S.S. cylinder (approximately 8 inches in diameter and 3 feet in length), connected to the cable pull mechanism located on top of the furnace frame which controls the motion of the suspended polyrods. The S.S. cylinder diameter is ample for either accommodating polyrods or for the possibility of growing silicon ingots if circumstances warrant. The removed portion of the bellows was in turn fixed to the existing bellows over the growth chamber.

It is extremely critical in the CLF process that all equipment components meet the specified tolerable leak rate.

Extension to CLF Furnace Frame (Cont'd)

Because of the lack of specific technical expertise in working with S.S. bellows, remodification work was not done in-house, but was instead handled by a bellows specialist on the East Coast.

2.2 Demonstration of CLF System

2.2.1 Standard Demonstration Runs

Demonstration runs occurred on a weekly basis during the quarter. Runs were smooth with relatively few difficulties experienced. Use of the recently extended seed lift mechanism, made possible through elevating the frame, aided significantly in these tests as seen by the increased size and weight of the ingots produced. Of all ingots grown, sizes were from 155 to 160 mm in diameter with weight ranging from 25 to 30 kg. Of the total material pulled, 50 percent was monocrystalline in structure. We achieved an average and consistent pull rate of 2 kg/hr.

2.2.2 Nonstandard Demonstration Runs

Demonstrations of continuous melt transfer were basically routine when compared to the final run at the end of May. After transferring melt successfully most of the day, the transfer tube was unintentionally dipped too far into the melt contained in the growth crucible. Although melt transfer was recovered, we could not be certain at this point that the heater or other internal portions of the transfer tube system

Nonstandard Demonstration Runs (Cont'd)

were not damaged.

With more than an hour of melt transfer as an indicator, it was assumed that the process was resuming as normal; it was not until early in the evening when melt transfer ceased (some 4 hours after the transfer tube shell was brought in contact with the silicon melt) that we were assured some damage had in fact occurred to the tube.

As the ingot itself was slightly larger in diameter than in previous runs (approximately 160 mm), the decision was made to remove it, then recover the melt contained in the growth crucible by growing another ingot. However, in raising the initial ingot, which had been growing for about 10 hours, either due to a flaw in the seed itself or as a result of an unprecedented roughness in the seed lift mechanism, the ingot broke where connected within the seed chuck. The ingot, weighing approximately 20 kg, fell through the gate valve back into the melt, cracking the growth crucible and splattering molten silicon throughout the furnace interior.

It was definitely fortuitous that almost all parts and

Nonstandard Demonstration Runs (Cont'd)

components within this portion of the CLF furnace were either salvaged or quickly duplicated by stock items on hand -- save for the bellows, which was riddled with miniscule holes made by droplets of molten silicon. There was no other recourse but to send the bellows back to the East Coast for immediate repair.

In the event repair was not possible or practical, we had worked out an alternate plan of action in order to maintain the schedule outlined in the Program Plan. Fortunately, there has not been a necessity to resort to another method, and we expect shipment of the repaired bellows within the next few days.

We feel the problems related to multiple ingot growth, as described in this report, can be eliminated by growing one single ingot. The growth of a large ingot can definitely be achieved through the incorporation of continuous melt replenishment with a significant impact on crystal add-on cost. Siltec will pursue this objective.

2.3 Melt Deflectors for the Transfer Tube

The incidence of inadvertently dipping either of the tube ends too far into the melt has been noted. It occurs both during tube priming (in order to initiate the transfer process) and/or while the melt transfer function is in progress, when transfer would accelerate too quickly, filling one crucible more rapidly than can be normally anticipated and thus submerging one end of the transfer tube to a point where the melt comes in contact with the outer shell. From past experience, we determined (by means of transfer tube dissections) that when this dipping occurred, the molten silicon would come in contact with the tube heating element, as well as being drawn up into the internal multilayers of the tube system, hence shortening its life and leading ultimately to the termination of the transfer process.

As the actual melt transfer process is an extremely precise and delicate operation, some method had to be devised in order to prevent this drawing of melt from happening.

A quartz tube was designed with cup-like deflectors/ferrules located near each end (approximately 1 inch from the tip) of the transfer tube as integrated portions of the tube itself (Figure 3). The O.D. and I.D. are unaltered by the addition of the deflectors; the outer shell of the transfer tube fits snugly around them. Melt flow is not impeded in the slightest.

Melt Deflectors for the Transfer Tube (Cont'd)

Fig. 3. Illustration of quartz deflectors which prevent melt from being drawn into the transfer tube heater area.

NOTES

1. UNLESS OTHERWISE SPECIFIED,
ALL DIMENSIONS ARE IN INCHES

It is quite probable that the use of this redesigned quartz tube, if used on the demonstration run at the end of May when the inadvertent overdip occurred, would have prevented the melt from seeping into the critical internal portions of the transfer tube system. However, we are still in the

Melt Deflectors for the Transfer Tube (Cont'd)

process of examining and evaluating the variables, advantages and disadvantages involved in the use of this modification.

Although from one perspective the deflectors are a sound idea in theory in deterring the melt from entering into the thermal insulation of the tube, they indeed have some significant disadvantages:

- . The deflector "cups," after several hours of exposure to the melt, could possibly separate from the quartz tube -- a result of dissolution and erosion by the melt. This occurrence would terminate crystal growth abruptly.
- . Visibility of the tube ends, important in monitoring melt transfer, is greatly impaired.
- . A heat sink is generated in a critical unheated portion of the tube.
- . This modification makes the tube considerably more expensive.

It is because of these disadvantages that the redesigned quartz tube has not been tested on any of the runs conducted to date. We hope, however, to demonstrate our melt deflection theory in the near future.

2.4 Additional Transfer Tube System Development

Alternative materials for the rigid graphite heating element are being investigated. Under consideration are: tungsten, molybdenum and molybdisilicide. These materials are all suitable for the 1450°C temperature range (under vacuum) in which the furnace and transfer tube system are operated. The primary goal in this effort is to reduce cost through both simplicity of the design as well as the ability to use the element for at least 15 consecutive runs.

Again with the objective of lowering the total cost factor of the transfer tube system, we attempted to substitute ceramic tape for the more expensive silica tape used in tube preparation to date. However, the ceramic tape displayed too high an impurity level and therefore could not successfully be used as an electrical insulator.

Additional thermal profiles were conducted in order to further comprehend the effects of the heat zones in the main heater on the temperature of the transfer tube. Recent profiles have been performed at lower temperatures, thus causing minimal deterioration of the quartz transfer tube so that the completely assembled transfer tube system can be reused in a CLF demonstration at a later date.

As seen in the profiles illustrated in Figure 4, temperature

Additional Transfer Tube System Development (Cont'd)

data plotted at lower temperature levels are more uniform primarily as a result of more effective thermal insulation. In addition, the influence of the main heater for the crucibles is minimized.

Fig. 4. Drawing of transfer tube system (half-section)
indicating the most recent thermal profile data.

3.0 CONCLUSIONS

3.1 Monocrystalline Structure

Although we are achieving 50 percent monocrystalline structure in the material produced, we would like to see this yield increased considerably. We intend to investigate all possible areas involved in crystal quality in order to achieve this goal.

3.2 Redesigned Quartz Tube

In theory, the quartz transfer tube, redesigned in order to prevent molten silicon from entering critical heater/insulation areas of the transfer system, has merit. However, this modification still bears some evaluation of the advantages versus the disadvantages (outlined earlier in this report). We will continue our investigations in this area.

4.0 PROJECTION OF ACTIVITIES

Plans for the next reporting period involve the following areas:

- . Receipt of the repaired S.S. bellows and subsequent installation on the CLF furnace.
- . Performance of growth demonstrations, including:
 - the growth of at least one 50-kg, 150-mm diameter silicon crystal.
 - The growth of first 100 kg, then 150 kg monocrystalline ingots (150 mm in diameter) from one crucible incorporating continuous liquid feed.
- . Continued development of the chunk feed mechanism.
- . Continued evaluation and development of the transfer tube system.

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

Program Plan (Milestones)

PROGRAM PLAN
(Revision 71)

JPL Contract No. 954886, Modif. 11

CLF Furnace - Phase II Scale-Up Feasibility Demonstration

TASK DESCRIPTION	April					May				June				July					August			
	1	7	14	21	28	5	12	19	26	2	9	16	23	1	7	14	21	28	4	11	18	25
(A) <u>Operate the CLF furnace & demonstrate:</u>																						
(i) Growth of one 25 kg, 15 cm dia crystal.	▼						▼															
(ii) Growth of 100 kg of silicon single crystals 15 cm dia from one crucible using CLF.																	▼					
(iii) On one occasion growth of one 50 kg, 15 cm dia silicon crystal.																			▼			
(iv) On two occasions growth of 150 kg (three 50 kg ingots) 15 cm diameter single silicon crystal.																				▼		▼
(v) On one occasion growth throughput of 50 kg approx. 15 cm diameter single crystal from one crucible using CLF with a chunk feed mechanism.																					▼	
(B) <u>Modify the CLF furnace:</u>																						
(i) Installation and development of silicon chunk feed mechanism																					▼	
(ii) Extend crystal lift mechanism to allow processing of 50 kg ingots above gate valve.

Start-Finish

Approved:

G. Fiegl, Program Manager
SILTEC Corporation
3717 Haven Avenue
Menlo Park, CA 94025