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LSA LARGE AREA SILICON SHEET TASK CONTINUOUS LIQUID FEED
CZOCHELSKI GROWTH

Quarterly Report, July–September 1979

By
Dorothea Walters

October 1979

Work Performed Under Contract No. NAS-7-100-954886

Siltec Corporation
Menlo Park, California

MASTER

U.S. Department of Energy

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Solar Energy

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LSA LARGE AREA SILICON SHEET TASK
CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

QUARTERLY REPORT #8

July - September 1979

Project Manager: George Fiegl

Prepared by: Dorothea Walters

October 1979

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

This project, currently in its second phase, is directed toward the design and development of equipment and processes to demonstrate continuous growth of crystals by the Czochralski method, suitable for producing single silicon crystals for use in solar cells. "Continuous" is defined as the growth of at least 150 kg of monosilicon crystal, 150 mm in diameter, from one growth container. The approach to meeting this goal is to develop a furnace with continuous liquid replenishment of the growth crucible, accomplished by a meltdown system with a continuous solid silicon feed mechanism and a liquid transfer system, with associated automatic feedback controls.

In order to establish process parameters for conventional CZ growth of 150 mm ingots, several runs with 12-kg charges were performed, and solidification rates of 2.7 to 3.5 kg/hr were achieved. This series of runs is providing a control group which will be used in future runs with continuous melt replenishment.

Significant progress has been made in the development of a new melt transfer system. Basic material problems relating to the high temperature environment were solved. The cost of the new transfer mechanism is lowered substantially through multiple use of the major system components.

Manufacturing of parts and installation of the polyrod feed mechanism for continuous recharging of the meltdown chamber were completed together with the feedback control system which uses the melt level sensor belonging to the growth crucible as input.

The design for a continuous particle feeder into the meltdown chamber has been completed, and the fabrication of this system is currently in progress.

Several short melt replenishment runs with 5 to 8 kg of continuous melt transfer were performed in an effort to develop feedback control systems for solid rod and particle feeding as well as to establish the permissible pressure region in which melt transfer can be assured.

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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" is defined as a throughput of silicon that produces 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 meltdown chamber.
- . Design, development and fabrication of the continuous liquid silicon feed system with a reuseable heater and automatic feed-back controls.
- . Demonstration of continuous crystal growth as defined above, with an after growth yield >80%.
- . Definition of process control variables for continuous Czochralski growth.
- . Theoretical analysis of silicon sheet growth to be considered during design and development of the furnace.

During this past quarter, activities were directed primarily toward:

- . Amelioration of leaks to an acceptable leak rate suitable for operation under reduced pressure.
- . Performance of several standard 12-kg runs, growing 150 mm crystals under reduced pressure.

INTRODUCTION (cont'd)

- . Development of a new melt transfer system.
- . Development of components for the polyrod feed mechanism.
- . Design of a continuous particle feeder into the meltdown crucible.
- . Finally, the performance of the first transfer run conducted under reduced pressure.

2.0 RESULTS

2.1 Growth Under Low (Reduced) Pressure

Problem areas noted in the last quarterly report, specifically silicon monoxide (SiO) build-up in the furnace interior over long periods of furnace operation, were dramatically reduced due to a number of factors. Modifications to the argon gas introduction system and evacuation ports were made in order to optimize the carrier gas purge action during low pressure growth.

To elaborate, early this quarter, it was noted that monocrystalline quantities produced were not quite up to expectation. This was determined to be caused in part by the inadequacy of the oil vacuum pump in use at the time for the growing operation under low pressure. The available pump speed was not sufficient to remove the required gas volume necessary for efficient purge action of SiO vapors, in the desired operating range of 30 to 40 torr, inside the furnace chamber. In addition, filter clogging with SiO after 15 to 20 hours of furnace run time led us to the decision of installing a large capacity 300 SCFM liquid ring vacuum pump. It was installed in September, and an improvement in the quantity of monocrystalline ingots obtained was noted immediately.

Growth under reduced pressure has not only reduced these SiO deposits, but, in critical parts of the furnace interior, has also significantly reduced the amount of argon consumption. Occasionally structure losses occur, but this is no longer a result of SiO particle interference with the solid liquid interface of the growing crystal.

A large number of crystals, 150 mm in diameter, were grown by the standard CZ technique from 12-kg charges to provide a control group for future continuous melt replenishment runs as well as to establish

Growth Under Low Pressure (cont'd)

the process parameters for 150 mm ingot growth with reduced pressure. Solidification rates of 2.7 to 3.5 kg/hr were achieved during the first few inches of growth. Maintenance of these solidification rates can be expected throughout ingot growth with liquid replenishment as a result of the more stable thermal condition of the melt.

The series of tests performed reconfirmed the fact that monocrystalline growth cannot be sustained for more than two to three inches beyond the point of "structure loss" (e.g., introduction of dislocations before a shift to polycrystalline growth occurs). We also determined from these runs that to separate these 150 mm diameter ingots from the melt without a complete roundoff causes internal fractures which usually are only revealed during the slicing process of the ingot.

2.2 Transfer Tube System

Basic material problems related to the high temperature environment of the transfer system were solved. The portion of the transfer system closest to the melt surface deteriorated rapidly in previous runs, however, this problem was completely resolved through the application of a special coating to the outside shell of the transfer tube.

The heating element, previously formed by grafoil strips, was replaced with a rigid graphite heater permitting multiple run use, thereby dramatically reducing the cost of the heating element per kg of Si produced.

All parts of the new transfer system are designed for multiple use as well as ease of assembly. Suitable electrode connections are presently under development.

Transfer Tube System (cont'd)

Short transfer runs were performed under a low pressure environment at 35 to 40 torr. Both crucibles were charged with 12-kg melts. "Priming" of the tube was accomplished through a pressure differential of 40 torr. Continuous melt replenishment during crystal growth was possible in the 35 to 40 torr range. The possible pressure region in which melt transfer is assured is a function of the hydrostatic pressure generated by the silicon column in the transfer tube.

During these test runs, monocrystalline ingots were grown from replenished melts.

2.3 Continuous Solid Replenishment

2.3.1 Polyrod Feed Mechanism

A Hamco bead chain assembly, provided by JPL, was installed in the CLF system early this quarter. The model we received was modified by replacement of the motors. This addition will enable us to "feed" a polyrod, thus accomplishing one method of continuous solid replenishment of molten silicon (see Figure 1). The polyrod will ultimately be lowered through a chamber consisting of a stainless steel bellows identical to the bellows which serve to house and cool the new-grown ingot.

A feedback control system has been designed for polyrod feeding with temperature and melt level sensor input.

We have four polyrods, 150 mm in diameter, 30 kg each, on hand, and await receipt of a cable and clevis (chuck) from which to suspend the polyrod before initiating demonstration runs.

Figure 1. Diagram of polyrod feed mechanism for CLF furnace.

2.3.2 Lump Feed Mechanism

A vibratory feeder for transporting silicon lumps from a supply hopper to the meltdown crucible, via a canted chute, has been ordered, and the overall design of the feeder has been completed (see Figure 2). Design of subsystems is currently in progress.

Figure 3 illustrates the feed mechanism as part of the servo-system which assures constant melt level in the growth crucible.

2.4 Extension to Frame of CLF Furnace

An extension was designed for the CLF furnace which will add 27 inches to the overall height of the frame, thereby extending crystal pull capability. The present configuration limited the pulling capacity to 24 inches of crystal above the gate valve. This addition will enable us to grow crystals with an overall length of up to 48 inches above the gate valve, increasing cycle efficiency in the growth of crystals. All material has been ordered and received, and plans are to complete this extension as soon as priorities permit.

2.5 Hot Zone Study

An in-house study has been initiated to examine the hot zones of crystal growth systems within Siltec R&D. This study will, of course, include the performance of the hot zones within the CLF furnace system. We hope to provide a thermal profile in the next quarterly report.

2.6 Crystal Ingot Quality/Quantity

During a few of the demonstration runs early this past quarter, the

Figure 2. Silicon Lump Feed Mechanism

As depicted, silicon lumps are transported from the storage/supply hopper, via a vibratory feeder, into a tube, canted in such a manner that it will both absorb the energy of the free-falling lumps and guide these pieces into the melt with minimum disturbance.

Figure 3. Diagram of lump/particle feed mechanism for CLF furnace.

Crystal Ingot Quality/Quantity (cont'd)

silicon crystals displayed a bluish discoloration resulting from exposure to oxygen in the growth chamber. A considerable amount of time was spent checking for leaks using a mass spectrometer. All leaks have subsequently been reduced to an acceptable level, and the discoloration phenomenon has not recurred.

There is very little in the way of theoretical knowledge or practical experience on a production scale in the silicon industry regarding the growth of ingots 150 mm in diameter and larger. As all of the ingots grown in our demonstration runs have fallen into this 6-inch size category, and considering the statement made above, we have experienced some difficulty in maintaining monocrystalline structure. There is a vast difference between the growth process of 100 mm diameter ingots and those that are 150 mm, and we continue to accumulate knowledge and experience in an effort to improve both reproducibility of monocrystalline material as well as efficiency of operation. With this objective to improve consistency of monocrystalline growth, experiments have been conducted varying the crystal growth speed, with the most significant changes occurring in the shouldering technique.

3.0 CONCLUSIONS

3.1 Growth Under Low Pressure

Lack of monocrystalline ingot production in sufficient quantities prompted us to replace the oil vacuum pump with a liquid ring water pump in order to increase efficiency in pump speed while reducing the incidence of filter clogging with SiO₂ after 15 to 20 hours of furnace run time. Immediate improvement in both quality and quantity were evident, indicating the superiority and practicality of this new water pump.

3.2 Equipment Control

The CLF furnace, as presently configured, is almost entirely manually controlled. While this has been suitable for demonstrating technical features of the system, many of the functions of the furnace must be handled automatically before technical readiness can be achieved.

4.0 PROJECTION OF ACTIVITIES

4.1 Solid Recharging

A continuous solid recharging mechanism is needed to fully utilize the advantages of the CLF furnace as well as to avoid incidence of quartz chipping and/or breakage of the meltdown crucible as a result of thermal cycling or being struck by silicon lumps. Two methods are currently under investigation: continuous polyrod, and lump/particle replenishment.

Components for the polyrod feed mechanism have been fabricated, and we plan to have the system in operation early next quarter.

Conceptual designs for the lump/particle feed mechanisms are complete, and the design of subsystems is currently in progress. We plan to demonstrate these methods approximately January 1980.

4.2 Transfer Runs

Many demonstration runs utilizing the transfer tube under low pressure are planned. We have already conducted several successful runs and have learned a great deal from each experience.

4.3 Reuseable Transfer Tube Heater

As we have experienced problems with heater lifetime when in use over a period of 60 to 70 hours, a new rigid graphite heater has been designed and fabricated. A suitable electrode connection is presently under development, and we anticipate conversion to this heater in October.

4.4 Program Plan

In most respects, activities are flowing along in accordance with the Program Plan. It is our intention to maintain this schedule of activities.

APPENDIX I

New Technology

No new technology was generated during this reporting period.

APPENDIX II

Summary of Characterization, Design, and Economic Analysis Data

No data were generated this quarter to analyze.

APPENDIX III

Milestones

PROGRAM PLAN

TASK DESCRIPTION	PERFORMANCE SCHEDULE									
	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN	
(1) Modify the liquid feed (CLF) furnace										
(A) Overall design	▲	▲	▲							
(B) Reduced pressure operation 10 to 30 Torr	▲	▲	▲	▲						
(C) Polypod 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 four 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 three 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 and annual workshop				▲				▲		
(B) Performance review meetings			▲			▲			▲	
(9) Provide documentation	▲	▲	▲	▲	▲	▲	▲	▲	▲	
(10) Procure parts, material and services (as required)										

Milestone Legend ▲ Due Date ▲—▲ Start-Finish