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LOW COST CZOCHRALSKI CRYSTAL GROWING TECHNOLOGY:
NEAR TERM IMPLEMENTATION OF THE FLAT PLATE PHOTOVOLTAIC
COST REDUCTION OF THE LOW COST SOLAR ARRAY PROJECT

Third Quarterly Progress Report for October 1—December 31, 1979

By
E. G. Roberts
R. L. Lane

MASTER

Work Performed Under Contract No. NAS-7-100-955270

Kayex Corporation
Rochester, New York

U-63 B

U.S. Department of Energy

Solar Energy

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THIRD QUARTERLY PROGRESS REPORT
OCTOBER 1ST - DECEMBER 31ST, 1979
PROGRAM MANAGER: R. L. LANE
PRINCIPAL INVESTIGATOR: E. G. ROBERTS

KAYEX CORPORATION
1000 MILLSTEAD WAY
ROCHESTER, NEW YORK 14624

"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

During the quarterly reporting period, the RF induction heated generator and the contractor owned CG2000 RC crystal puller have been installed and commissioned.

Two crystal growth runs have been made utilizing the crystal puller in the standard resistance heated mode. The first run was completed by growing 16.7 kg of zero dislocation 4" diameter ingot from 18 kg of charged polycrystalline silicon chunk.

The second run was undertaken at the request of the JPL technical monitor as part of contract #954888. A total of 100.3 kg of ingot was grown from 104.5 kg of charged polycrystalline silicon chunk.

Fabrication of the redesigned polycrystalline rod recharging system is overdue by the subcontractor. Delivery of the weight transducer is awaited. Delivery has been promised for 12/28/79. Delivery of the total system is scheduled for 1/4/80.

Redesign, fabrication and assembly of the RF feedthru and work coil system was completed to interface with the commissioning of the generator. During commissioning, some localized overheating of the RF feedthru was evident. The feedthru system has been modified and is currently being re-assembled. This will be completed by 1/4/80.

The quotation received for the supply of the cold crucible boat and work coil has been analyzed and rejected. A total system incorporating the melt transfer tube system plus the RF feedthru has been designed in-house. Various suppliers have been asked to quote and the quotations are currently awaited.

Installation of the microprocessor control system is almost complete, with overall completion scheduled for 12/28/79.

All necessary supplies have been received together with 512 kg of polycrystalline silicon rod.

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1.0 INTRODUCTION

The purpose of the program is to demonstrate the growth of up to 150 kilograms of 6" diameter single crystal silicon ingot from one crucible by the Czochralski (CZ) method.

The method being developed relies upon conventional CZ technology combined with new equipment and process designs and concepts. These concepts alternate cycles of crystal growth and hot melt replenishment and are designed to be ultimately suitable for use in a high volume production facility.

A Hamco Model CG2000 RC crystal grower will be utilized for the project. Modifications will be comprehended into the puller which will allow a special chamber to be fitted. This chamber will allow for the storage of polycrystalline silicon rod to be used in the accelerated melt and crucible recharge cycle. A vacuum tight isolation valve will be incorporated into the system to allow retrieval of grown crystals and crucible melt replenishment without contamination.

Additional modifications designed into the system allow for accelerated methods of melting both polycrystalline silicon rods and polycrystalline silicon chunks by the use of RF induction heating. Rods will be melted directly into the crucible by the use of a conical shaped induction heated copper work coil. Power to the work coil will be fed from a 50 kw RF generator via a remote switching system to a Hamco designed and fabricated RF feedthru system. The feedthru will be sited in one of the spectator viewports and will couple directly onto the work coil. It is designed to be infinitely variable through the vertical plane such that it will be utilized initially to melt rod, and can then be vertically adjusted to act as a heat sink for the accelerated growth program. The principle of the heat sink arrangement is that it will absorb the energy released by the heat of fusion during crystal growth.

A secondary accelerated melt program will be developed using polycrystalline

silicon chunks as feedstock. These chunks will be melted and fed into the growth crucible by the utilization of a cold crucible premelter. The premelter will also be heated by RF induction heating fed from the RF generator via a secondary remote switching arrangement. Silicon chunk feedstock will be fed into the cold crucible arrangement by means of a controlled feed mechanical auger system. Silicon will be melted and the melt levitated and poured through the cold crucible by utilizing the induction heating multiturn work coil surrounding the crucible. Transfer of the molten silicon into the quartz growth crucible will be achieved by pouring the molten silicon through a transfer tube. The temperature of the molten silicon in the transfer tube will be maintained by the use of a secondary RF multiturn coil surrounding the transfer tube. The total system, incorporating the ancillary RF feedthru assembly, is designed to interface with the puller through a port in either the furnace tank wall or furnace tank cover. The system is designed such that the transfer tube assembly system can be retracted after discharge of the molten silicon, so that it does not interfere with normal crystal growth.

The CG2000 RC crystal grower will be interfaced with a microprocessor based control system. This system will be programmable to the required sequencing and control requirements.

The program, therefore, deals with the process concepts as described. These concepts deal primarily with process improvements which will lower the costs of the meltdown and the crystal growth steps. Additionally, it deals with improved automation for the process to reduce labor costs and increase yields.

2.0 PROGRESS

The 50 kw RF induction heating generator has been installed and commissioned. The remote switching station for the accelerated melt program has been installed and commissioned. The secondary remote switching station for the cold crucible premelter will be fitted along with the cold crucible premelter when it becomes available.

The contractor-owned CG2000 RC crystal puller has been totally installed and commissioned. To date, two crystal growth runs have been made utilizing the equipment in the standard resistance heated mode. The first run was made during October, when 18 kilograms of re-cycled crystal was charged. Total weight charged versus total weight pulled equates to a 92.8% yield.

The second run was made at the request of the project technical monitor as part of JPL Contract #954888. The request was to attempt a 100 kilogram run using a standard unmodified Hamco CG2000 RC puller. The run was successfully completed using a standard 12 inch crucible and ancillary piece parts. A total of 100.3 kilograms was pulled from a total charge weight of 104.5 kilograms. This represents a pulled yield of 96%. A total of 63.9 kilograms of the pulled crystal weight was monocrystalline. This represents a 63.7% yield. The total run time was 108 hours, giving a throughput of 0.93 kg/hr. A series of mechanical problems impacted considerably on the run time and so affected the throughput. Mechanical problems encountered were as follows: (a) crucible rotation stopping due to the drive belt losing several teeth, (b) a broken crystal lift cable due to operator error.

Overall, however, the results obtained confirmed our view that a standard Hamco CG2000 RC puller has the capability of producing 100 kilograms of 5" diameter crystal from one crucible utilizing the hopper silicon recharging device developed on the JPL Contract #954888 program.

The polycrystalline rod recharge mechanism has been totally redesigned and is being built by a subcontractor. The system is complete except for the fitting of the weight transducer. The supplier is scheduled to deliver 12/28/79 and the subcontractor is scheduled to deliver the recharge mechanism 1/4/80.

The RF feedthru system for the accelerated melt program has been fabricated in-house and installed together with the work coil in the crystal grower. Two problems became evident when RF power was applied to the system:

a) Some localized overheating occurred in a stainless steel section of the feedthru assembly. This can be overcome by substituting the stainless steel with teflon.

b) Some generator output efficiency loss occurred due to the design of the feedthru system. This can be overcome by shortening the length of the co-axial feedthru and fitting a copper backing plate to the total assembly. This will improve the efficiency loss and provide an improved match between the generator, feedthru and the work coil. This will also reduce the heat load on the feedthru system.

All of the modifications are complete and the system is being reassembled. Completion of the reassembly is 1/11/80.

Some air leakage occurred on the initial fitting of the RF feedthru to the spectator viewport. This has been overcome by modification of the seal housing arrangement to accommodate an additional seal.

The RF feedthru and coil have been designed to be infinitely variable through the vertical plane. This will allow the work coil to be repositioned after completion of the rod melting cycle such that it can be utilized as a heat sink arrangement for the accelerated growth program.

During the previous reporting period, it was reported that a quotation had

been received for building of the cold crucible premelter. This quotation was evaluated and rejected, as it made no provision for melt transfer and RF feedthru systems to allow the premelter to interface with the crystal puller. Consequently, a total package was designed in-house and submitted for quotation. Replies to the quotation request are scheduled for 12/31/79. A visit is planned to another sub-contractor facility on 1/3/80 for discussion on fabricating the total package.

The installation of the microprocessor control system is almost complete. The data storage is approximately 95% complete and is scheduled for completion 1/3/80. Interfacing of the microprocessor wiring with the crystal puller is approximately 88% complete and is scheduled for completion 1/3/80. The initial run utilizing the microprocessor is now scheduled for the week commencing 1/7/80.

All necessary piece parts have been delivered together with 512 kilograms of nominally 5" diameter polycrystalline silicon rod. The rod supplier was not able to guarantee that the material was "crack free", but has supplied on a "best effort" basis with visual inspection prior to shipment.

The various programs relating to the overall contract are now reported separately.

2.1 Accelerated Melt Program

Meltback of Polycrystalline Silicon Rod Using an RF Coil

Polycrystalline silicon rod will be utilized as feed stock for the CZ growth program. It will be melted directly into the CZ growth crucible using an RF induction heated work coil. The rod will be stored and alternately melted in order to recharge the crucible after completion of each pulling cycle.

Discussion

A single turn conical-shaped copper work coil will be utilized for the accelerated melt program. Power is fed to the coil by means of a 50 kw RF induction heated generator operating at a frequency of 450 KHz. The power is fed by

co-axial cable to a remote switching box located on the puller framework. Power is then fed from the remote box via a specially constructed RF feedthru system sited in the spectator viewport of the puller. Power is then fed from the generator to the work coil via the remote box and feedthru. The system is illustrated in Figure 1.

The work coil is fabricated from copper and is water cooled. The coil is cone-shaped with a nominal ID of 6.5 inches. The cone is angled at approximately 30° to the top wall ID with a total band width of approximately 4". The coil will have a capability of both melting and pulling of crystals of up to 6 inches in diameter. The work coil is illustrated in Figure 2.

Delays have been encountered in the fabrication of the RF feedthru. Due to subcontractors refusing to quote, it was necessary to fabricate in-house. After fabrication and installation, some problems became evident when power was applied to the system.

a) Some localized heating occurred on a stainless steel section of the feedthru. This can be overcome by substituting the stainless steel with a teflon section.

b) Some generator output efficiency loss occurred due to the design of the system. This can be overcome by shortening the length of the feedthru and fitting a copper backing plate to the total assembly.

The modification to item (a) is completed and the modification to item (b) is completed and is currently being reassembled.

Some air leakage which occurred around the RF feedthru where it entered the viewport has been eliminated by modification to the seal housing to accommodate an additional seal.

These delays have resulted in a delay in the commencement of the accelerated melt program. Also, the delivery of the modified polycrystalline recharge mechanism has been delayed due to the subcontractor being unable to

Figure 1
7

Figure 2
8

secure delivery of one gear reducer. This was shipped 12/31/79 and was not received until 1/7/80. Delivery has now been scheduled for 1/15/80.

During the fabrication and modification of the RF feedthru and RF work coil, two crystal growth runs have been undertaken utilizing the crystal puller in the standard resistance heated mode. Details of these runs will be reported separately.

All initial accelerated melt and growth runs will be undertaken using 12" purified graphite piece parts and pulling 5" diameter crystals. When the necessary parameters have been established, 14" purified graphite piece parts will be installed and 6" diameter crystals will be pulled as required by the program.

515 kilograms of polycrystalline silicon has been purchased from Montedison USA. This material is nominally 5" diameter and has been supplied on a "best effort crack-free" basis.

2.2 Accelerated Growth Rate

Accelerated growth rate is an integral part of the program related to reduction of cost. By utilizing a suitable heat sink arrangement to absorb the energy released by the heat of fusion generated during the growth cycle, it is anticipated that improved growth rates can be achieved.

The copper water-cooled RF work coil used in the accelerated melt program has been designed to be adjustable through the vertical plane. After completion of the accelerated melt program, the work coil will be isolated from the RF generator and adjusted through the vertical plane to act as the heat sink.

Establishment of the operating parameters will be ongoing as part of the accelerated melt program.

2.3 Induction Premelting of Lump/Granular Silicon

A cold crucible system will be developed as part of the program phase immediately following the establishment of the accelerated melt/growth parameters.

The cold crucible will have the capability of being fed with either lump or granular type silicon feedstock, melted, and the resultant melt fed into the Czochralski growth crucible free of contamination.

An initial quotation of \$2800 was received from a subcontractor to build a cold crucible boat arrangement and ancillary multiturn copper work coil. This quotation was evaluated and rejected. It was rejected on the basis that a total design package was required incorporating the silver plated cold crucible boat, work coil, transfer tube and ancillary heating coil and feed-thru system. This system has been designed in-house and will form the basis of a technical feasibility meeting to be held at the subcontractor's facility on 1/8/80. A conceptual design sketch (Figure 3) is included in this report.

The cold crucible system will be operable on a continuous melt and transfer principle during the crucible replenishment process. A storage/feed arrangement for silicon lumps will be interfaced with the cold crucible, allowing lump silicon to be continuously fed into the cold crucible. The cold crucible boat will be endorsed in a quartz tube under argon pressure and will be heated by a multiturn RF work coil around the quartz tube. The RF coil will melt the silicon and, due to the induced magnetic field, the melt will be levitated and allowed to flow through the cold crucible boat. The material will then flow into a transfer boat of similar construction. This boat will be heated by means of a secondary coil, which will be built within the quartz transfer tube - refer to Figure 4. This total system will be fed through the tank cover into the work crucible. It will be built so as to be totally retractable, which will

DESIGN SKETCH

LEVITATION TRANSFER TUBE

Figure 4

allow it to be adjusted after charging of the crucible such that it will not interfere with normal crystal growth.

2.4 Microprocessor Controls

The contractor-owned CG2000 RC crystal grower will be interfaced with a microprocessor control system. Data storage and interface wiring for the interfacing of the microprocessor control to the puller is complete with the exception of the optic isolation system. This does not prevent operation of the control system on the CG2000 RC puller and will be completed after initial control evaluations are complete. De-bug and test of the programming is on-going. An initial run utilizing the microprocessor system is scheduled for 1/28/80.

3.0 DETAILS OF CRYSTAL GROWTH RUNS - NOS. 1 & 2

Both of the crystal growth runs were completed using 12" diameter crucibles and graphite piece parts.

Crystal Growth Run No. 1

Run No. 1 was undertaken as part of the acceptance trial of the equipment. A total of 18 kilograms of re-melt silicon was charged into the crucible. A total of 16.7 kilograms of zero dislocation 4" diameter material was pulled from the melt. The growth rate was increased to 4.9" per hour until the crystal rotation became eccentric. The growth rate was reduced progressively until the crystal became concentric again. The crystal, however, retained its zero dislocation structure. The crystal is illustrated in Figure #5.

Crystal Growth Run No. 2

Run No. 2 was undertaken at the request of Dr. A. Kachare, JPL Technical Project Monitor, as part of JPL contract #954888. The request was to investigate whether a new standard Hamco CG2000 RC crystal grower was capable of growing 100 kilograms of silicon from one crucible.

Figure 5
14

The run was completed using a 12" crucible and 12" graphite piece parts. Crucible recharging was accomplished using the lump charging device developed under JPL contract #954888. Details of the lump recharging device is as follows:

LUMP RECHARGING DEVICE

The lump recharging device presently being used is based on a self-dumping hopper design that can be seen in Figures 6 and 7. Chunks of silicon are loaded into the hopper when it is in the furnace pull chamber. The isolation valve is closed and the pull chamber is at atmospheric pressure. It is necessary to keep the bottom funnel portion of the hopper closed (tight to the bottom opening) so that silicon will not fall through the bottom opening while loading the hopper. This is accomplished by raising the hopper enough to keep its weight on the funnel. When the hopper is loaded with the desired amount of silicon, it is raised to a sufficient height for the isolation valve to be opened. Before the isolation valve can be opened, however, it is necessary to equalize the vacuum in both the pull chamber and hot zone sections of the grower. This is accomplished by first purging the pull chamber of air by pumping in argon and simultaneously pulling a vacuum on the pull chamber. Once the pull chamber has been purged and the vacuum pressure equalized, the isolation valve is opened and the hopper can be lowered to discharge the silicon into the crucible.

During the loading and purging cycles, the residual melt has been partially frozen into the crucible. Also, the crucible has been brought to a position that will minimize the distance the silicon will fall and, at the same time, prevent the silicon from piling up beyond the bottom of the funnel.

The discharge of the silicon is accomplished by stopping the hopper travel with the use of a support ring that rests on the flange of the isolation valve. The rod and funnel portion of the hopper is allowed to travel further, thus creating an opening between the funnel and stationary portion of the hopper.

Figure 6

Figure 7
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When the desired amount of silicon has been discharged, the rod and funnel are raised, closing the opening. Once the funnel contacts the stationary portion of the hopper, the entire hopper can be raised into the pull chamber past the isolation valve. At this point, you can either prepare for another recharge cycle, if necessary, or move the hopper back out of the way and lower the seed to grow another crystal.

During this reporting period, it was found that the capacity of the present hopper is approximately 18 kilograms of lump silicon. Since the hopper and its prototype were designed and tested when 12" diameter crucibles were being used, the ability to discharge 18 kilograms of lump silicon into a 12" diameter crucible with a residual melt is only possible by the use of a two step method. However, this two step method does not require two complete recharge cycles.

Based upon recharge results to date, it appears that particle size for reliable operation will be about 1" maximum. It is not known what the minimum acceptable particle size will be, but it has been observed that what we refer to as fines have a tendency to be blown around to a small degree.

Problems existed throughout Run #2 due to mechanical problems within the puller, i.e. continual problems due to the crucible rotation stopping, broken seed lift cable, and the teeth stripping off the crucible rotation drive belt. All of these problems impacted on the thruput due to the length of time required to complete the 100 kilogram cycle (109 hours). The quantity of high quality material also suffered as a result. 63.7% of the 100.3 kilograms grown was classified as high quality material (monocrystalline or better). However, after 50 hours of run time, 84.3% of the crystal grown to that time was high quality material (4 crystals weighing 46.4 kilograms).

A summary of the run is as follows:

Crystal ingot diameter	12.7 cms
Initial melt charge	18 kgms
Crucible diameter	30.5 cms
Total wt. of silicon melted	104.5 kgms
Total ingot pulled	100.3 kgms
Pulled yield	96%
Total wt. of mono crystal	63.9 kgms
Yield of mono crystal	63.7%
Number of ingots	9
Thruput	0.93 kgms
Total run time	108 hrs
Recharge material	100% lump

The crystals grown during Run #2 are illustrated in Figure 8.

Figure 8
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4.0 PLANS

- a) Complete installation of RF work coil and ancillary RF feedthru.
- b) Receive, install, and commission modified recharge mechanism.
- c) Commence accelerated melt and growth program and develop operating parameters.
- d) Interface microprocessor control system with Hamco CG2000 RC crystal puller and commence growth program.
- e) Complete cold crucible technical discussions and place order.
- f) Install and commission cold crucible assembly and determine operating parameters.

5.0 PROGRAM PLAN

The program plan is illustrated in Figure 9 and is updated to December 31, 1979.

CZ GROWTH METHODS

CONDITIONS	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Crucible Size (inches)	14" x 11-1/2"	14" x 11-1/2"
Crystal Diameter (cms)	15.25	15.25
Growth Rate (cm/hr)	15.0	15.0
Total Poly Melted (kg)	160	160
Total Crystal Pulled (kg)	150	150
Pulled Yield (%)	93.75	93.75
Yield After CG (%)	85.0	85.0
No. Crystals/Crucible	5	5
Cycle Time (hrs)	59.8	59.1
Throughput (kg/hr)	2.25	2.28

TABLE 1

SAMICS/IPEG INPUT DATA & COST CALCULATION

FOR LOW COST CZ (ROD FEED)/LOW COST CZ (POLY LUMP FEED)

CONDITIONS (PER CYCLE)	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Total Si Melted (kg)	160	160
Crystal Weight	30	30
No. of Crystals/Crucible	5	5
Diameter of Crystal (cm)	15.25	15.25
Growth Rate (cm/hr)	15.0	15.0
Cycle Time (hrs)	59.8	59.1
Crucible Size	14" x 11-1/2"	14" x 11-1/2"
% Yield (Total in Spec. CG Ground)	85%	85%
Thruput (kg/hr)	2.25	2.28
Input Data (1980 \$)		
Capital Equipment Cost (EQPT)	219,000	209,000
Manufacturing Floor Space (SQFT)	100	100
Annual Direct Labor Salaries		
Prod. Operator (0.65 Persons/Yr)	8,100	8,100
Elect. Tech. (0.3 Persons/Yr)	1,425	1,425
Inspector (0.1 Persons/Yr)	<u>1,068</u>	<u>1,068</u>
Total DLAB =	10,593	10,593

TABLE 2

	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Direct Used Materials & Supplies:		
85% Usage Per Year		
Cycles/Yr Hrs/Cycle	124.4/59.8	125.9/59.1
Poly-Kg/Yr (Charged)	19,904	20,144
Seed (\$5.82)	\$ 722	\$ 733
Dopant (Not Costed)		
Argon (100 Ft ³ /Cycle-Hr @ 0.02/Ft ³)	\$ 14,878	\$ 14,881
Crucibles (14" = \$291)	36,084	36,666
Miscellaneous (including graphite:		
\$3.5/Cycle-Hr)	<u>26,037</u>	<u>26,042</u>
Materials Total (MATS)	\$ 77,721	\$ 78,322
Utilities (Process):		
Electricity		
(65kw x 0.035/kw)(Cycle Time - 3 hrs)		
(# Cycles)	\$ 16,075	\$ 16,354
Cooling Water		
(65kw)(\$0.0074)(Cycle Time - 2 hrs)		
(# Cycles)	<u>3,458</u>	<u>3,457</u>
Utilities Total (UTIL)	\$ 19,533	\$ 19,811

TABLE 3

	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
IPEG Price:		
C1 EQPT = \$0.49/Yr - \$EQPT	\$ 107,310	\$ 102,410
C2 SQFT = \$97/Yr - \$SQFT	9,700	9,700
C3 DLAB = \$2.1/Yr - \$DLAB	22,245	22,245
C4 MATS = \$1.3/Yr - \$MATS	101,037	101,818
C5 UTIL = \$1.3/Yr - \$UTIL	<u>19,533</u>	<u>19,811</u>
Annual Cost	\$ 259,825	\$ 255,984
Quan. (Total Charge x % Yield)(kg)	16,918 kg	17,122 kg
Throughput	2.25 kg	2.28 kg
Add-On Cost (\$/kg or \$/m ²)		
(Assume 1 kg = 1 m ²)	\$ 15.36 (1980)	\$ 14.95 (1980)

TABLE 4

PROGRAM PLAN LOW COST CZ CRYSTAL GROWTH JPL CONTRACT NO. 955270

Total Man Months

60.75 60.75

START

DESIGN SPEC.

COMPLETED

COMPLETE

TASK

Figure 9
26