DR. 827

DOE/JPL/955270-2

440
2/19/80

LOW COST CZOCHRALSKI CRYSTAL GROWING TECHNOLOGY

Near Term Implementation of the Flat Plate Photovoltaic Cost Reduction
of the Low Cost Solar Array Project

Second Quarterly Progress Report, July 1–September 30, 1979

By
E. G. Roberts

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

Kayex Corporation
Rochester, New York

MASTER

U.S. Department of Energy

Solar Energy

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Paper Copy ~~\$4.00~~

Microfiche ~~\$3.00~~

5.00

50

LOW COST CZOCHRALSKI CRYSTAL GROWING TECHNOLOGY

NEAR TERM IMPLEMENTATION OF THE FLAT PLATE PHOTOVOLTAIC
COST REDUCTION OF THE LOW COST SOLAR ARRAY PROJECT

SECOND QUARTERLY PROGRESS REPORT
JULY 1 - SEPTEMBER 30, 1979
PROGRAM MANAGER: R. L. LANE
PRINCIPAL INVESTIGATOR: E. G. ROBERTS

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

MASTER

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."

REPRODUCTION OF THIS DOCUMENT IS UNLIMITED eb

ABSTRACT

During the quarterly reporting period, the R.F. induction heating generator and the contractor-owned CG2000 RC crystal puller have been received and are currently being installed. Installation is approximately 90% complete. Completion is scheduled for October 3, 1979. All functions of the crystal grower have been tested out and a 7 kilogram crystal has been pulled in the equipment testing department as part of a standard customer acceptance analysis.

Total redesign of the crystal recharge system has been completed and the fabrication has been subcontracted. Problems interfacing the R.F. work coil with the R.F. feed-thru system has necessitated a redesign of the coil connection assembly. This is scheduled for completion October 10, 1979.

The R.F. feed-thru has been redesigned due to extensive lead times for specialized materials. The system is currently being fabricated in-house. The redesign now allows us to utilize the spectator viewport for the feed-thru rather than having to machine the port in the tank cover assembly.

All necessary piece parts for crystal growth have been received, together with 159 kilograms of nominally 5" diameter polycrystalline silicon rod.

A quotation has been received for fabrication of the cold crucible assembly and work coil. This is currently being evaluated.

CONTENTS

	<u>PAGE</u>
ABSTRACT	i
1.0 INTRODUCTION	1
2.0 PROGRESS	2
2.1 ACCELERATED MELT REPLENISHMENT	3
2.2 ACCELERATED GROWTH RATE	5
2.3 INDUCTION PREMELTING OF LUMP/GRANULAR SILICON	6
2.4 MICROPROCESSOR CONTROLS	8
3.0 PLANS	8
4.0 PROGRAM PLAN	8

FIGURES

FIGURE 1 PROGRAM PLAN	9
-----------------------	---

1.0 INTRODUCTION

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

The method being developed relies on conventional CZ technology combined with new equipment designs which alternate cycles of crystal growth and hot melt replenishment by methods suitable for use in a high volume production facility.

A Hamco Model CG 2000 Crystal Grower will be modified with a special chamber for the storage of a supply of polycrystalline silicon and a vacuum tight isolation valve to permit retrieval of crystals and melt replenishment without contamination.

A number of additional modifications to the facility have been comprehended in the overall design. These modifications will allow for the feed-thru systems for both the accelerated melt and accelerated pulling programs and for the feeding of silicon from the cold crucible to the quartz crucible used in the crystal pulling process. The initial thrust of the project will be to develop the polycrystalline silicon rod accelerated melt program. The R.F. feed-thru for this program will be fitted through the spectator viewport in the tank cover assembly. The R.F. induction work coil will be connected to the R.F. feed-thru and is designed to be infinitely variable through the vertical plane. This coil will be utilized for both the accelerated melt program and also as the heat sink for the accelerated growth program.

The cold crucible will be utilized to continuously melt and feed polycrystalline silicon chunk material directly into the opaque quartz crucible. Material will be fed into the cold crucible by means of an auger feed system. The material will be melted and levitated by utilization of induction heating magnetic field phenomena. The resulting melt will flow directly into the quartz crucible through a suitable feed-thru system sited in the chamber wall.

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

2.0 PROGRESS

The 50 kw R.F. induction heating generator has been received. All electrical supplies to the generator have been completed, together with all necessary plumbing.

The contractor-owned CG2000 RC crystal puller has been tested and fault-checked through the standard customer acceptance testing department. A 7 kg charge has been successfully melted and a crystal pulled as part of the standard acceptance procedure. The puller has now been transferred to the designated JPL contracts' area and the installation is currently nearing completion. Completion is scheduled for October 3, 1979. Customer testing are then scheduled to pull a crystal from a 17 kilogram charge as the final acceptance check.

The crystal recharge mechanism has been totally redesigned and is currently being built by a sub-contractor. The redesign was considered necessary as the bulk and overall weight of the existing system was found to be a major source of air leakage, resulting in structure loss.

The R.F. feed-thru system is currently being machined in-house and is approximately 90% complete. Problems arose due to the sub-contractor's reluctance to fabricate the feed-thru arrangement. A contractor agreed to fabricate and deliver by September 1, 1979. After retaining the drawings for one month, they declined to fabricate, stating they would not guarantee the system due to the high heat load. In order to avoid further delay, it was decided to fabricate in-house.

Some minor redesign was undertaken so that the feed-thru could be fitted into

the spectator viewport. The advantage of the re-design is two-fold, i.e. (a) it allows the sections in the feed-thru to be spaced further apart, which will ensure less probability of insulation break-down, and (b) by utilizing the spectator viewport, it prevents the tank cover assembly having to be machined to accommodate the feed-thru.

The R.F. induction single turn work coil is available and will be fitted to the R.F. feed-thru when the feed-thru becomes available. Completion is currently estimated as October 18, 1979. The work coil has been designed to be infinitely variable through the vertical plane. It will be consequently utilized as the heat sink, which will remove the heat of fusion during crystal growth. This will form the basis of the accelerated growth program.

Technical discussion has been ongoing with a subcontractor to build both the cold crucible and the ancillary R.F. induction heating coil for the contamination-free melting of polycrystalline silicon chunk material. A quotation of \$2800 has been received and is currently being investigated.

The microprocessor system is currently undergoing program and software evaluation. This is approaching completion, which will allow the system to be interfaced with the CG2000 RC crystal puller. The necessary wiring modifications will be undertaken after completion of the crystal puller installation.

All piece parts with the exception of the heat shield have been received. The delivery of the heat shield has been promised for October 1, 1979.

159 kilograms of nominally 5" diameter polycrystalline silicon rod has been received out of a total order of 515 kilograms.

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

2.1 Accelerated Melt Replenishment

Meltback of Polycrystalline Rod Using an R.F. Coil

Polycrystalline silicon rod will be melted using an R.F. induction heated copper work coil. The rod will be stored and alternately melted in order to recharge the crucible after completion of the pulling cycle.

Problems encountered during the design and fabrication stages were:

1. Coil configuration and matching of the coil to the R.F. feed-thru.
2. Design and construction of the R.F. feed-thru.

Discussion:

A single turn machined copper fabricated work coil will be utilized for the accelerated melt program. Power is fed to the coil by means of a 50 kw R.F. 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 R.F. feed-thru 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 feed-thru.

The work coil has been fabricated from machined copper, but will require some modification to allow it to be connected to the R.F. feed-thru. By fabricating from machined copper, the wall thickness is thicker than conventional tubing. The coil is water cooled; the increased wall thickness therefore allows the coil more protection against fracture should arcing occur.

The coil is cone-shaped with an ID of nominally 6.5". The cone is angled at 30° to the top wall ID with a total band width of approximately 4". The coil will therefore have the capability of melting and pulling of crystals of up to 6" diameter. Melting trials have indicated that, by having a slightly cone shaped coil, the melt can be effectively "squeezed", such that a pouring effect of the melt can be achieved.

Major problems have been encountered in locating a subcontractor who had the

capability and the willingness to fabricate the R.F. feed-thru. The one contractor who originally agreed to build finally returned the drawings after one month and declined to build. They stated that they could not guarantee the fabrication because of the anticipated high heat load. The feed-thru is now being fabricated in-house and is presently 90% complete. Extreme difficulties were experienced in locating material suitable for fabrication, i.e. machinable copper.

These delays have resulted in a delay in the commencement of the accelerated melt program. However, it will not prevent the standard products testing department from pulling a crystal from a 17 kilogram silicon charge using the equipment in its standard form. This will be followed by a 100 kilogram run as part of JPL Contract #954888 program. This has been requested by Dr. Kachare, JPL Project Monitor. These runs will allow the R.F. feed-thru and work coil installation to be completed.

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

A suitable supply of polycrystalline silicon rods was located at Montedison USA at a price of \$65 per kilo. This was price advantageous over Hemlock Semiconductor Corporation by \$21.65 per kilo. To date, a total of 159 kilograms has been received against a total order quantity of 515 kilograms. The material is nominally 5" diameter and, although an initial request was made for "crackfree" material, suppliers could not guarantee it to be so. Material has therefore been supplied on a "best effort" basis.

2.2 Accelerated Growth Rate

Accelerated growth is an integral part of the program related to reduction

of cost. By utilizing an appropriate heat sink arrangement to effectively absorb the energy released by the heat of fusion generated during the growth cycle, it is anticipated that growth rates of up to 15 cms per hour can be achieved.

The copper water-cooled R.F. work coil used in the accelerated melt program has been so designed such that it can be adjusted through the vertical plane. This work coil will be adjusted after the rod melting cycle so that it can be utilized as the heat sink arrangement.

Establishment of the optimum parameters for achieving maximum growth rates 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 automatically fed with either lump or granular type silicon feed stock, melted, and the resultant melt fed into the Czochralski growth crucible free of contamination.

Technical discussions have been ongoing with a subcontractor for the building of a cold crucible system as detailed in Figure 1. A quotation was received September 26, 1979 for the design and construction of an arrangement incorporating the cold crucible and the work coil for a total cost of \$2800. The subcontractor has been asked to quote the design and subcontract separately so that a decision whether or not to fabricate can be made after the design phase. The cost quoted does not include silver plating.

The design quotation will allow fabrication of a cold crucible "boat" type system as opposed to a "skull type" design. It will consist of a copper boat incorporating a 4" diameter scoop-shaped device in which the shell of the

scoop will be formed of individual 1/2" copper tubes. The tubes will be brazed to a master manifold and deadended. A second master manifold (for intake water) will mate with the first manifold and will have individual 1/4" copper tubing outlets which will penetrate individually into each individual 1/2" copper tube. Water entering the intake manifold will enter the individual copper tubes via the 1/4" inserts. Water can then flow on the outside of the 1/4" tubes via the 1/2" tubes and return to the outlet manifold. This will ensure adequate cooling to the entire assembly.

A high voltage multi-turn work coil will be fabricated. This will be designed to load as heavily as possible the output of the generator into the crucible boat. The coil will be formed of multi-turn copper tubing with leads which will match the output tank circuit and the insulating stud mount assemblies. This will prevent arcing. Approximate dimensions of the cold crucible are 10" long x 4" diameter.

It is considered that the cold crucible arrangement be operated on a continuous feed melt and deliver principle during the crucible replenishment process. It is envisaged that a storage hopper/auger feed system be utilized to feed the silicon chunk/granular material into the cold crucible. The R.F. coil arrangement will melt the silicon and, due to the induced magnetic field, the melt will be levitated and so flow over the cold crucible arrangement. The melt will be transferred from the cold crucible to the Czochralski pulling crucible by means of a heated transfer tube. This transfer tube will be so designed as to retain the temperature of the molten silicon above the temperature of solidification.

An application has been made to JPL to utilize already developed technologies for polycrystalline silicon feeding and for the transfer of molten silicon via a heated transfer tube. Being able to utilize developed

JPL contract technologies would result in both time and cost savings. To date, no authorization has been granted.

2.4 Microprocessor Controls

The contractor-owned CG2000 RC crystal grower will be interfaced with a microprocessor control system. The system is currently undergoing program and software evaluation. This is almost complete, which will then allow the system to be interfaced with the crystal puller. Wiring modifications will then have to be undertaken to complete the installation for evaluation.

3.0 PLANS

- a) Complete installation of CG2000 RC puller and R.F. induction heating generator.
- b) Complete fabrication of R.F. feed-thru system and R.F. work coil.
- c) Complete 17 kg final acceptance crystal growth evaluation.
- d) Complete 100 kg crystal growth run for JPL Contract #954888.
- e) Complete fabrication of modified design recharge mechanism and install on crystal puller.
- f) Evaluate cold crucible quotation and place order.
- g) Conduct bench trials utilizing cold crucible and determine operating parameters.

4.0 PROGRAM PLAN

Figure 1 is the Program Plan updated to September 30, 1979. Labor hours and incurred costs are updated to August 31, 1979.

PROGRAM PLAN LOW COST CZ CRYSTAL GROWTH J.P.L. CONTRACT NO 955270