430
5-27-80
JPL

DOE/JPL/955269-79/2

Dr. 1499

LOW COST SOLAR ARRAY PROJECT, TASK I—SILICON
MATERIAL. GASEOUS MELT REPLENISHMENT SYSTEM

Second Quarterly Progress Report, July 18—October 17, 1979

By
D. N. Jewett
H. E. Bates
D. M. Hill

Work Performed Under Contract No. NAS-7-100-955269

Energy Materials Corporation
Harvard, Massachusetts

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.

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."

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 \$5.00
Microfiche \$3.50

DRL-113

DRD-SE-4

Low Cost Solar Array Project
Task I - Silicon Material

Gaseous Melt Replenishment System

Second Quarterly Progress Report
18 July - 17 October 1979

JPL Contract 955269

by

D.N. Jewett
H.E. Bates
D.M. Hill

Energy Materials Corporation
Ayer Road
Harvard, Massachusetts 01451

The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photo-voltaic 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.

Table of Contents

<u>SECTION</u>	<u>PAGE</u>
ABSTRACT.....	1
I. INTRODUCTION.....	2
II. PROCESS DESCRIPTION.....	4
III. TECHNICAL PROGRESS.....	7
IV. PLANNED THIRD QUARTER ACTIVITIES.....	11

ABSTRACT

During the second quarterly reporting period we have continued design activities on a number of components of the reactor furnace system and heat exchangers. Numbers of purchased components have been received, mainly for the gas handling system. Assembly of the gas handling system is under way.

I. INTRODUCTION

This program is intended to develop a system which incorporates both silicon formation and melt replenishment into the same piece of equipment. The processes and equipment are scaled such that a modest investment can make available to the Czochralski crystal grower a low cost source of silicon. In addition, the smaller scale of operation means that the systems can be put into operation without large capital investments, guaranty of markets, etc.

The chemical reactions, H_2 reduction of $SiHCl_3$, are those in commercial usage now. The major innovation is in reactor design which allows a high productivity of silicon. The reactor has been conservatively sized on the basis of epitaxial deposition rates. The conclusion of this calculation is that a reasonably sized system can produce silicon rapidly enough to keep pace with either 10 cm or 12 cm diameter Czochralski crystal growth operating in a semi-continuous mode.

The program plans call for design, construction and testing of a prototype system and demonstration, over a twenty-four hour period, of a 0.5 kg/hr rate of Si production with 18% conversion efficiency. Based on initial findings with the prototype system,

a second generation system will be built and demonstrated over a 96 hour continuous run. This system will also include a valve/delivery tube system to allow replenishment of a crystal growth system. Demonstration of the complete system performance in operation with a Czochralski crystal puller over a 96 hour period will comprise the final phase of the program.

II. PROCESS DESCRIPTION

A schematic cross-section of the reactor system is shown in Figure 1. Polycrystalline silicon will be deposited on the inside walls of a resistively heated, multi-walled fused silica reaction chamber by H_2 reduction of $SiHCl_3$. After sufficient silicon has been produced, the reactor is flushed with argon and the silicon melted out of the reactor into a Czochralski crystal growth crucible. The reactor is then returned to the deposition stage. The reaction chamber and the crystal growth system are separated by a heated delivery tube. The "U" tube acts as a valve by adjusting the temperature above or below the melting point of silicon. A more detailed description of a process cycle is given below:

1. The reactor is brought up to temperature under inert gas flow.
2. A small amount of silicon is melted in the "U" tube to form a positive gas seal. The "U" tube temperature is dropped to about $1200^{\circ}C$.
3. At the selected reaction temperature $SiHCl_3$ and H_2 are introduced into the chamber and their flow rates set to optimize the maximum mass deposition of silicon.

Figure 1.

Schematic Diagram of
"Poly" Reaction Chamber

(Scale $\approx 5:1$)

4. The reaction is allowed to run for several hours until the desired amount of silicon has been deposited. This is determined by the desired cycle time and the reactor vessel size.
5. At this point the reactant gases are flushed out of the reactor with Ar.
6. Keeping the "U" tube at 1200°C, the reactor is raised to about 1450°C to melt down the silicon produced.
7. The gas pressure between the reactor and the delivery tube is equilibrated.
8. The "U" tube and delivery tube are raised to about 1415°C. When the silicon is melted in the "U" tube, the reactor will empty due to gravity, causing liquid silicon to flow out through the delivery tube to either the Czochralski crucible or an intermediate replenishment crucible. Due to the equilibrated pressures and a vent to prevent siphoning, the "U" tube will remain full after the reactor has been emptied.
9. The reactor is returned to reaction temperature and the "U" tube and delivery tube are returned to 1200°C.
10. The reactant gas flow is then re-admitted to the chamber to start the cycle again.

III. TECHNICAL PROGRESS

A. Gas Handling System

During this quarter the major components of the gas handling system have been received. (See Figure 2.) These include the following: hydrogen recycle compressor, by-product condensers, filters, pressure regulators, and pumps. An insulated tank and pump have been received to circulate methanol, cooled by a dry ice-methanol bath, through the first low temperature by-product condensor.

A support rack and bench for the majority of the gas handling equipment have been assembled. A small metal building, 10 X 14 feet has been erected immediately outside the main building to house the recirculating compressor, exhaust gas scrubber, and by-product chlorosilane collection drums. Major piping connecting the by-product condensers, recycle compressor, pressure regulators, filters and flow meter has been assembled and leak-tested. Initial operation tests of the recycle compressor indicated too much vibration was being transmitted through the piping connected to the compressor. Stainless steel flexible couplings were procured, and will be installed to isolate the compressor.

B. Design Activity

Detailed mechanical design and drawings have been com-

FIGURE 2. SCHEMATIC DIAGRAM OF PROCESS EQUIPMENT AND FLOW SYSTEM.

pleted in this period for the following: reactor vessel support/enclosure, U-tube valve support, reactor heating elements, U-tube heater, primary heat exchanger, and finned tube gas cooler. This latter item is a water-jacketed, finned Inconel tube intended to cool the product gas stream to improve the efficiency of the subsequent chlorosilane condensers and alleviate problems of thermal stress on the tube sheets of the shell and tube heat exchangers. Fabrication of the various support structures, heating elements and the primary heat exchanger is nearly complete, and most of the pieces have been received. All of these are graphite.

Further design and fabrication is required for housing, supporting, and connections to the primary heat exchanger. The heat exchanger must not impose any significant loads on the reactor vessel or tubulations to prevent deformation of the quartz vessel at high temperature. Also, the heat exchanger must not be rigidly mounted such that it will constrain the connecting tubulations as the various members supporting the reactor vessel increase in length with temperature. We plan to use flexible stainless steel bellows to connect to the cold end of the exchanger, and a counterweight support system to nullify its weight.

C. Reactor Furnace

Major components of the reactor furnace power supply have been received. These include power controllers, transformers,

cabinet, switches and contacters. Delivery of the Leeds & Northrup temperature controllers is scheduled for mid-November. Assembly and wiring of the power supplies has begun and is planned for completion in mid-November.

Delivery of the reactor furnace chamber, originally scheduled for early September, has been significantly delayed. The fabricator has experienced delays in receiving the steel preforms needed to build the chamber. Delivery of the finished chamber and trichlorosilane evaporator is now being promised for early November.

D. Reactor Vessel

Fabrication of the prototype quartz reactor vessels is awaiting delivery of material to the vendor. He has suggested in discussions, some minor modifications to make assembly easier, but which have no effect on the basic configuration.

E. Gas Chromatograph

We have ordered, for on-line product stream analysis, a Varian Model 920 gas chromatograph. Delivery is scheduled for early December.

IV. PLANNED ACTIVITIES

Planned activities for the third quarter (October-January) include:

- Complete construction and assembly of the prototype system.
- Start-up operation of the prototype system.
- Installation and calibration of the gas chromatograph system.

PROGRAM PLAN - Milestone Chart - (JPL Contract 955269)

8-1-79