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LOW COST SOLAR ARRAY PROJECT. TASK I, SILICON MATERIAL

Gaseous Melt Replenishment System

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MASTER

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ABSTRACT

During this quarterly period we have completed assembly of 95% of the power control and gas handling systems. 95% of the reaction chamber parts are made and ready to be assembled.

As new problems have become apparent, we have made specific design changes and additions. Specifically, thermal expansion, quartz to graphite connections, and recycle gas flow control have posed problems that have been eliminated by design.

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, guarantee 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 1) design, construction and testing of a melt replenishment system, 2) a continuous 24 hour run, 3) design and construction of a melt replenishment system to produce 1/2 kg/hr. at 18% conversion of TCS to silicon, 4) design and construction of a valve/delivery tube to connect a melt replenishment system to a Czochralski crystal puller and 5) GMR production of silicon from TCS which is transferred to a Czochralski unit for crystal production continuously for 96 hours.

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.
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

Figure 1.

Schematic Diagram of
"Poly" Reaction Chamber

(Scale $\approx 5:1$)

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. DISCUSSION OF TECHNICAL PROGRESS

During the past quarter we have continued to receive and assemble parts of the reaction chamber, power control box, and gas handling system. Some of the assembled equipment has been tested.

A. Procurement and Assembly

We have received the jacketed steel chamber, TCS evaporator, L&N controllers, gas chromatograph, top circular aluminum chamber extension, two charcoal tank heaters and a 15 gallon compressor surge tank. Delivery of a drum of activated charcoal, the chamber "top hat", two flow control valves, a by-pass valve, and the gas chromatograph prepacked columns is expected within two weeks.

The L&N controllers were installed, and the power control box was completed. The sample lines and manifold for the gas chromatograph, the TCS evaporator, and compressor surge tank were installed and tested for leaks. The liquid scrubbing system was completed and has been run continuously for several hundred hours. Four small weigh scales were installed under the halo-silane storage drum to indicate when the drum needs to be replaced with an empty drum. The activated charcoal tanks with heaters were mounted and plumbed into the recycle system. When the activated charcoal arrives, the tanks will be filled, leak tested and then insulated.

The water lines to the coolers and jacketed chamber were completed. Assembly of the main heater, U-tube heater, the heater electrodes, and the electrode current feeds were begun.

B. Additional Design

Additional design and fabrication was completed concerning horizontal expansion of the graphite support box, quartz to graphite connections, and gas flow control.

The graphite box which supports the quartz deposition vessel was originally supported from the steel heat shield plate by rigid graphite posts. Due to thermal expansion of the box these posts would be susceptible to breaking, therefore a "custom" support was designed. The column has 1 inch diameter ends with a 1/2 inch hollow mid section. The bottom is secured to the steel heat shield. The top is grooved for a roller bearing; a similar groove is on all four of the corners of the support box's bottom plate. The slots are aligned on the diagonals of the square bottom plate; thus, allowing movement on the bearing but not allowing the box to move off center. The cross sectional area of each support post is as small as possible to minimize heat flow. The small contact area of the rollers also minimizes heat transfer. Around each of the posts is a shorter graphite collar which supports the graphite heat shield.

The quartz to graphite connections will be made with a Grafoil[®] gasket and circular compression rings which will press the flanged quartz tube to the graphite heat exchanger header.

Due to the low strength of quartz at high temperature and the relatively large surface area of the reaction chamber walls (100 square inches each), the gas pressure and flow to and from the chamber must be steady. Two additional vee-stem valves were installed in the gas cycle, one before and one after the chamber. These valves are in addition to two regulating valves already in the system. Relatively large pressure drops across the two vee-stem valves will attenuate the pressure variations to the regulating valves and therefore in the quartz chamber.

C. Testing

During the past quarter we have tested all liquid pumps, the TCS evaporator, the compressor, liquid scrubber, water coolers, and condensers. With a few modifications all work satisfactorily. The system has been checked several times for leaks and is nearly "leak tight".

IV. PROJECTED FOURTH QUARTER ACTIVITIES

During the next three months we intend to complete the pressure equalitation system, and reaction chamber. When these are assembled and tested, we will begin testing silicon deposition rates with respect to gas composition, temperature, chamber configuration, and density. Upon approval we will begin to design and fabricate a silicon delivery tube that will integrate the GMR system with a Czochralski unit.