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DEVELOPMENT OF A POLYSILICON PROCESS BASED ON CHEMICAL
VAPOR DEPOSITION (PHASE 1)

Fourth Quarterly Progress Report for July 1–September 30, 1980

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December 1980

Work Performed Under Contract No. NAS-7-100-955533

Hemlock Semiconductor Corporation
Hemlock, Michigan

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FOURTH QUARTERLY PROGRESS REPORT

1 JULY TO 30 SEPTEMBER 1980

on

DEVELOPMENT OF A POLYSILICON PROCESS

BASED ON CHEMICAL VAPOR DEPOSITION

(PHASE 1)

prepared by

K. Sharp, Principal Investigator

and

A. Arvidson, and D. Sawyer

December 1980

JPL Contract 955533

"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."

a wholly owned subsidiary of Dow Corning Corporation

12334 Geddes Rd., Hemlock, Michigan 48626

ABSTRACT

The goal of this program is to demonstrate that a dichlorosilane-based reductive chemical vapor deposition (CVD) process is capable of producing, at low cost, high quality polycrystalline silicon. Physical form and purity of this material will be consistent with LSA material requirements for use in the manufacture of high efficiency solar cells.

The feasibility of silicon generation from dichlorosilane (DCS) has been well established in the program to date. The feasibility and optimization portions of the experimental reactor program have been completed, with a number of runs having been conducted over a broad range of conditions in an experimental CVD reactor.

Activities relating to feed of commercially purchased DCS to an intermediate sized reactor and to construction of a Process Development Unit (PDU) to generate and feed DCS to one or more production scale reactors were suspended during the previous quarter because of the receipt of new safety-related information about DCS from Hazards Research Corp.

Experimental data generated by Hazards Research Corp. indicate that DCS/air mixtures possess about four times the explosive severity potential as hydrogen/air mixtures, and that DCS/air mixtures are very readily ignited. As a consequence of this new information, designs and procedures for the intermediate reactor feed and PDU tasks were deemed inadequate and new designs incorporating new safety-related elements are being formulated.

New safety-related design features for the PDU will include: remote plant location; remote operation; no DCS storage; minimal DCS holdup in equipment; and dilution of DCS with hydrogen prior to its transport to decomposition reactors.

In support of PDU activities, a laboratory scale rearranger has been utilized to generate a mixture of redistributed chlorosilanes from trichlorosilane. This mixture was then fed to an experimental reactor in which silicon was deposited and analyzed for electrically active impurities. Both boron and donor impurity levels were less than one part per billion (atomic) in the material.

Silicon grown in the experimental reactor from commercially purchased DCS was converted to single crystal material and sent to Westinghouse for solar cell fabrication and evaluation. Cell efficiencies were nearly identical with baseline efficiencies, and results were cited as "quite encouraging".

A preliminary economic evaluation of the Hemlock Semiconductor process has been completed. The analysis for a plant to generate 1000 metric tonne of silicon indicates a plant investment of \$21.9 M, and a product selling price of \$19.85/kg.

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1.0 Summary

This report describes a process for the low-cost production of polycrystalline silicon from dichlorosilane (DCS) via reductive chemical vapor deposition (CVD) with hydrogen. The DCS is generated from the catalyzed redistribution of trichlorosilane. The by-product silicon tetrachloride may, if desirable, be converted to trichlorosilane via hydrogenation. Objectives of Phase 1 (the current contract) are to demonstrate the feasibility of using DCS as a CVD reactor feed material and to utilize base catalyzed redistribution of trichlorosilane to produce high purity DCS. Phases 2 and 3 of the program will demonstrate the technology readiness of the process at the EPSDU level.

The feasibility and optimization portions of the experimental reactor program has been completed. Over 30 runs with DCS have been successfully conducted over a wide range of reactor operating conditions.

Activities relating to feed of commercially purchased DCS to an intermediate sized reactor and to construction of a Process Development Unit (PDU) to generate and feed DCS to one or more production scale reactors were suspended during the previous quarter because of the receipt of new safety-related information about DCS from Hazards Research Corp.

Experimental data generated by Hazards Research Corp. indicate that DCS/air mixtures possess about four times the explosive severity potential as hydrogen/air mixtures, and that DCS/air mixtures are very readily ignited. As a consequence of this new information, designs and procedures for the intermediate reactor feed and PDU tasks were deemed inadequate, and new designs incorporating new safety-related elements are being formulated this quarter.

New safety-related design features for the PDU will include: remote plant location; remote operation; no DCS storage; minimal DCS holdup in equipment; and dilution of DCS with hydrogen prior to its transport to decomposition reactors.

In support of PDU activities, a laboratory scale rearranger has been utilized to generate a mixture of redistributed chlorosilanes from trichlorosilane. This mixture was then fed to an experimental reactor in which silicon was deposited and analyzed for electrically active impurities. Both boron and donor impurity levels were less than one part per billion (atomic) in the material.

Silicon grown in the experimental reactor from commercially purchased DCS was converted to single crystal material and sent to Westinghouse for solar cell fabrication and evaluation. Cell efficiencies were nearly identical with baseline efficiencies, and results were cited as "quite encouraging".

A preliminary economic evaluation of the Hemlock Semiconductor process has been completed. The analysis for a plant to generate 1000 metric tonne of silicon indicates a plant investment of \$21.9 M, and a product selling price of \$19.85/kg.

2.0 Introduction

The following abbreviations will be used on this section: DCS for dichlorosilane; TCS for trichlorosilane; STC for Silicon Tetrachloride.

2.1 Program Objectives

The objective of this program is to demonstrate that a chlorosilane based chemical vapor deposition (CVD) process can produce a low cost polycrystalline silicon in high volume. Product quality both in terms of purity and form should be comparable to material produced by the existing trichlorosilane CVD process which meets or exceeds requirements for use in the manufacture of high efficiency solar cells.

The overall program covers a 39 month period and consists of a feasibility phase, which is the subject of the current contract, an EPSDU design phase, and an EPSDU construction/demonstration phase. The schedule for the program is shown in Figure 1. Specific Phase 1 project objectives include:

1. Characterization of DCS as a feedstock material for an experimental CVD reactor including quantitative determination of reaction products. (CVD Reactor Feasibility)
2. Design and construction of a DCS CVD reactor which will demonstrate DCS performance in a larger size reactor. (Intermediate Dichlorosilane Reactor Development)
3. Design, construction and operation of a laboratory scale redistribution reactor and a process development unit (PDU) to characterize the TCS-to-DCS redistribution process, determine product purity, and produce sufficient DCS to permit operation of a production sized reactor. (Dichlorosilane Process/Product Evaluation)

4. Conduct preliminary design of a EPSDU based on information collected in the areas previously described and develop supporting information for an economic evaluation of a 1000 metric ton plant. (EPSDU Design)

The general approach taken in meeting the overall program objective consists of: the hydrogenation of STC to produce TCS; synthesis of DCS via redistribution of TCS; high temperature decomposition of DCS to produce poly-crystalline silicon; and recovery of decomposition byproducts. STC a major byproduct of TCS redistribution and minor byproduct of DCS decomposition is recycled into the hydrogenation process.

The basic chemical nature of the various steps in the DCS-based low-cost silicon process have been described in previous Quarterly Reports.^{1,2}

3.0 Technical Status

The following abbreviations will be used in this section: DCS for Dichlorosilane; TCS for Trichlorosilane; and STC for Silicon Tetrachloride.

Phase 1 technical efforts are limited to the four areas discussed in Section 2.1. No efforts are being expended in the area of STC hydrogenation due to JPL support of the Union Carbide program. Neither are efforts being devoted to the development of CVD reactor vent product recovery technology. This technology is closely aligned with recovery system technology currently employed in the TCS CVD process. Phase 1 efforts thus consist of the following:

- CVD Reactor Feasibility
- Intermediate Dichlorosilane Reactor Development
- DCS Process/Product Evaluation
- EPSDU Design

A milestone chart detailing work to be accomplished in these four general areas is shown in Figure 2. The four major areas of technical activity are discussed in Sections 3.1 through 3.5.

3.1 CVD Reactor Feasibility

3.1.1 Objectives

The objectives of the reactor feasibility task are to:

1. Establish feasibility of DCS as a feed material in a high deposition rate CVD reactor.
2. Optimize reactor operating parameters for DCS feed.
3. Develop comparative data regarding DCS and TCS behavior in an experimental CVD reactor.
4. Provide necessary information for scale-up to an intermediate size or full-scale production reactor.
5. Establish vent product composition to permit 1000 metric ton plant design and costing.
6. If necessary, evaluate the performance of a reactor operating on mixed feed (DCS and TCS).

The reactor and support systems employed in this experimental program are described in Section 3.1.2, while experimental results are presented in Section 3.1.3.

3.1.2 Experimental CVD Reactor and Feed System

The previous Quarterly Report detailed the completion of the feasibility and near-completion of the optimization phases of the experimental reactor program. Our intention was to repeat two runs in the experimental design program which constituted the optimization phase of the experimental reactor work. However, the new safety-related information from Hazards Research Corp. (discussed in Section 3.5) received in the interim caused us to reconsider the safety of our feed system to the reactor. A review of the feed system by site safety personnel concluded that extensive revisions in the system would be necessary before experimental runs could be resumed.

It was then decided that such a modification could not be justified to support only two additional runs. The experimental reactor program is accordingly considered completed.

Silicon Purity Data.

Solar cells fabricated by Westinghouse⁴ from material grown in run 394-057 have been evaluated. The twenty-five cells included in the test provided an average uncoated AM1 efficiency of 9.1% compared to the baseline value of 9.28%. Eight of the experimental cells displayed efficiencies in excess of 9.5%, and two exceeded 10%. Westinghouse personnel estimated that with AR coatings, the material would produce cells with AM1 efficiencies of 12-14%, and considered results to date "quite encouraging."

3.2 Intermediate Reactor Development

3.2.1 General

Scheduled activities in this task have also been impacted by the new information relating to explosive potential of DCS. The anticipated feed system for the intermediate reactor involved the placement of commercial DCS cylinders in a heated pipe tunnel below a production reactor building. This arrangement is now deemed inadequately safe, and an alternative feed system is being designed. The new system will feature location of DCS cylinders external to the reactor building, with provisions to safely dilute and vent DCS in the event of a spill. Some cost savings in this task could be realized if the feed system were operated only during the winter months. In that event, formation of a DCS vapor cloud from a leak would be much less likely.

3.3 Dichlorosilane Process/Product Evaluation

3.3.1 General

The objectives of this task are:

1. Establish purity of DCS produced via catalyzed redistribution of TCS.
2. Permit characterization and optimization of the redistribution process through design, construction and operation of a laboratory rearranger unit (PDU support) and a process development unit (PDU), and provide design information for the EPSDU and 1000 metric ton plant.
3. Provide sufficient DCS at a reasonable cost from the PDU to permit regular operation of an intermediate size CVD reactor at high feed rates.

These objectives will be met through dual tasks designed to provide a data base for PDU design and operation, and actual PDU design, construction and optimization. The PDU support effort is discussed in section 3.3.2 while PDU design activities are reviewed in section 3.3.3.

3.3.2 PDU Support

General.

The overall objective of the DCS Intermediates laboratory program is to provide data to support the design, understanding and safe and efficient operation of the PDU (DCS mini-plant). The chemical reactions and principles of operation of this plant have been described elsewhere.^{1,2} Information has been developed in the following general areas:

- Process Stream Analysis
- Catalyst Stability
- Redistribution Kinetics
- Product and Product Stream Safety

A discussion of the objectives and approach in each of these areas has been provided in previous Quarterly Reports.

RESULTS OF EXPERIMENTAL WORK

A. Process Stream Analysis

The gas chromatographic sampling and analytical techniques described in prior Quarterly Reports^{2,3} has been used without difficulty throughout this quarter.

B. Catalyst Stability

No additional direct work was conducted this quarter on the stability of our selected redistribution catalyst--Dowex MWA-1 resin. However, the thermal and conditioning histories of Dowex appear to have a significant effect on its catalytic activity in vapor phase reactions. These results are reported in the following section.

C. Redistribution Kinetics

1. Vapor Phase Trichlorosilane Kinetics

Several additional experimental runs were made in the small vapor phase reactor unit (shown schematically in Fig. 3). These runs were undertaken in order to evaluate the effect of thermal pretreatment of Dowex resin on its catalytic activity. The data are presented in Table 1 and Fig 4. It can be seen that the conditioning method is a factor in the catalytic activity of Dowex. Some degree of thermal pretreatment, i.e., 100 °C, is considered desirable to remove residual moisture, but more drastic treatment, i.e., 165 °C, tends to severely decrease catalytic activity. This behavior is consistent with previous weight loss experiments with Dowex: more rapid loss of weight occurs at temperature exceeding ca. 125 °C.

2. Liquid Phase Trichlorosilane Kinetics

No additional work was done during this quarter on liquid phase TCS redistribution kinetics. As reported previously, the second order kinetic rate constant (k_0/a) was found to be ca 0.20 min^{-1} at 77°C at linear flow rates greater than 25 ft/hr . The rate value k_0/a is defined as the open tube kinetic rate constant divided by the void volume fraction of the catalyst particles. A void volume fraction of 0.5 was assumed for Dowex resin.

3. Product Purity

The purity of material from the laboratory scale liquid phase rearranger was evaluated during this quarter by collecting a 6 liter sample from redistribution of semiconductor grade TCS and depositing a silicon rod by chemical vapor deposition. A 100 inch Dowex bed was used. The deposited silicon rod was analyzed for boron, donor (phosphorus) and aluminum by a zone refining technique and for carbon by Fourier transform infrared (FTIR) analysis. Results are provided in Table 2.

The data indicate extremely low levels of electrically active impurities; all are fully compatible with semiconductor grade material except boron. The boron levels in runs with both heated and room temperature Dowex were a factor of 3 to 4 higher than the control runs made without the presence of the resin. Although this "high" level of boron ($<.7 \text{ ppba}$) is substantially less than the maximum permissible concentration for highly efficient solar cells, some efforts will be directed at reducing this boron contamination during the next quarter.

3.3.3 Process Development Unit Design

Design and proposed operating characteristics of the PDU were discussed in the second Quarterly Report.² Briefly, the design features a redistribution reactor, feed and product storage vessels, and a single distillation column. The

still will be used to purify DCS and, as the last stage in a production cycle, to distill a TCS/STC mixture to provide feed of appropriate composition for the next run. Proposed operating sequences for the unit were provided in the first Quarterly Report.¹

With the arrival of the new information relating to explosion and flammability hazards of DCS (discussed in the following section), extensive reconsideration of design and operating characteristics and location of the PDU were made necessary. The incorporation of new safety features into the design of the facility has constituted a major activity during the past quarter. This activity, as well as the wait for definitive information from Hazards Research Corp. has caused an unavoidable delay in the construction and operation of the PDU.

New safety-related features of the current PDU design are discussed in the section 3.5.

PDU sizing was initially based on a 35 lb/hr production rate of DCS for feed whose composition is 80% TCS and 20% STC. The "mixed feed" approach has since proved undesirable based on laboratory studies. With a pure TCS feed to the redistribution unit, the production rate of the PDU could be a factor of two higher than originally anticipated due to the higher equilibrium yield of DCS.

3.4.0 Preliminary EPSDU Design

3.4.1 General

The EPSDU will consist of the following processes:

- DCS synthesis and purification
- polycrystalline silicon CVD using DCS
- vent product recovery

The production scale of the plant is anticipated to lie between 100 and 200 metric tonne of silicon.

TCS available from existing HSC process streams will be used as feedstock for DCS synthesis and STC will be disposed of in the manner currently employed by HSC. Some integration of the vent product recovery system with the existing HSC recovery system may be possible.

Demonstration of the hydrogenation process is being undertaken by Union Carbide Corporation under a separate JPL contract.

3.4.2 EPSDU Status

Effort in this area was rather limited during the past quarter. Principal activities include refinement of the preliminary material and energy balances for the plant, and consideration of alternatives for plant and reactor location and for possible integration of the EPSDU chlorosilane recovery system with the existing HSC recovery system.

3.4.3 1000 Tonne Plant Design

The bulk of the activity on this task during the past quarter involved preliminary design of a 1000 metric tonne plant. Flow sheets for two design alternatives for the plant have been completed, and are presented in Figures 5-8. The two schemes differ in the extent to which boron removal is accommodated. In the first option (Fig. 7), the TCS streams from reactor recovery and hydrogenation are mixed; in the second (Fig. 8), TCS is segregated from the hydrogenation for distillation and boron removal. The second option requires an additional still. The quantity of chlorosilanes which pass

through the boron removal device per kg silicon generated is approximately five times greater in the first (single TCS still) option.

Design Basis

The 1000 tonne/yr silicon plant design presented in figures 5-8 is discussed in this section. Design basis for the 1000 tonne silicon plant assumes 90% on line time (OLT) for the hydrogenation system, 95% for the stills and recovery unit, and 85% OLT for the decomposition reactors. The decomposition reactor OLT represents 89% OLT for reactors, compounded by 95% building OLT.

Anticipated product yields for the various unit operations are as follows:

1. Hydrogenation

exit concentrations on a non hydrogen basis are

- a. H_2SiCl_2 1% (mole %)
- b. HSiCl_3 29.5%
- c. SiCl_4 69.5%

2. Redistribution

exit concentrations are

- a. H_3SiCl 0.5% (mole %)
- b. H_2SiCl_2 10%
- c. HSiCl_3 78%
- d. SiCl_4 11%

3. Decomposition reactors

exit concentrations on a non-hydrogen basis per mole of H_2SiCl_2 feed are

- a. Si 0.40 in reactor
  - b. H_2SiCl_2 0.10
  - c. HSiCl_3 0.34
  - d. SiCl_4 0.16
  - e. HCl 0.14
- b.-e. in vent

Material flows and compositions utilized assume ideal separations. This approach allows sizing of equipment, but will require modification of tables after separation specifi-

cations are established.

The overhead of the DCS column will be controlled to provide equal amounts of monochlorosilane and TCS in the DCS, thus maintaining a Cl:Si ratio of 2:1 in the decomposition reactor feed stream.

3.4.4 Unit Operation Description for 1000 Tonne Plant

HYDROGENATION UNIT

The hydrogenation unit shown in Figure 5 represents the same unit as presented by Union Carbide Corporation in JPL LSA contract 954334.⁵ The new drawing includes line designation and flows corresponding to HSC requirements.

Exit compositions from the reactor were selected from work of Mui and Seyferth.⁶

Detailed design of this unit as ultimately implemented may differ somewhat from the unit shown, in order to satisfy Hemlock Semiconductor Corporation design and safety criteria.

OPERATION

Metallurgical grade silicon is received in ground form and placed in the silicon storage bin (line 101). The silicon is mixed with copper catalyst (line 102) in the catalyst blender. This mixture is placed in the silicon feed hopper (line 105) where it is purged free from air. Hydrogen is used to pressurize the hopper and force the catalyst and silicon into the hydrogenation reactor (line 107). Hydrogen (line 112) and STC (line 126), in equimolar quantities at just over 500 °C enter the bottom of the hydrogenation reactor, which is operated in a fluidized state. Thermodynamic limitations of the reaction restrict the practical conversion to 29.5 mole % TCS in the effluent.

The products leaving the hydrogenation reactor (line 114) enter the quench contactor. A large quantity of cool liquid is pumped from the waste settler to the quench contactor (line 115) to condense some of the chlorosilanes and wash out metallic salts and silicon metal fines in the reactor product. The salts and metal settle in the waste settler and are removed (line 119).

The cooled product gas then passes to the product condenser (line 117), then to the liquid receiver (line 118). Gravity separation of the liquid and vapor occurs in the receiver. Liquid exits to the crude TCS storage tank (line 121) and hydrogen exits to the hydrogen surge tank (line 122). The liquid product is later sent to distillation.

The hydrogen is compressed and sent to the hydrogen heater (line 124). In the same heater housing, STC from the STC vaporizer is heated to greater than 500 °C. Both streams then feed the hydrogenation reactor.

STC is received as make up material (line 129) as well as recycle from the TCS still (line 207). Make up material is required to replenish the Cl lost in the waste settler effluent and HCl produced in the decomposition reactors (and subsequently sold).

DISTILLATION

A three still system has been selected, as shown in Figure 7. The first still, the stripper column, removes all materials with boiling points lower than DCS from the hydrogenation reactor product.(line 204) DCS, TCS, and STC pass from the bottom of the column (line 206) and enter the TCS column.

The TCS column receives feeds from the bottom of the stripper column (line 206), from the recovery unit (line 201), and from the redistribution reactor (line 214). STC is removed from the bottom (line 207) and is sent to the STC storage tank in the hydrogenation area. The overhead product (line 212) is a mixture of DCS and TCS. DCS goes through the boron removal system, then is fed to the DCS still (line 213).

The DCS still removes DCS overhead (line 219) to storage, with controls used to maintain equal monochlorosilane and TCS concentration in the overhead stream. The bottoms stream from the still (line 215) is comprised of TCS. The TCS goes either directly to the redistribution reactor, or into TCS storage.

Storage in the still area will allow 3 days of operation of the decomposition area with the hydrogenation unit shut

down. This storage is required to maintain high decomposition reactors OLT, thus avoiding unfavorable utility consumption caused by insufficient feed. System capacitance will be supplied as TCS storage located in the line between the bottom of the DCS still and the redistribution reactor. A small amount of DCS storage is provided to ensure process stability.

VAPORIZER

DCS will be vaporized in a kettle type boiler (line 221). It will then be mixed with hydrogen (line 302) to the desired concentration for feed to the decomposition reactors (line 303).

DECOMPOSITION REACTORS

Decomposition reactors will receive DCS and hydrogen as feed (line 303). Each reactor will produce 3000 grams per hour of silicon while in operation. Reactor vent gas will exit to the recovery unit (line 304).

RECOVERY UNIT

The recovery unit receives a mixture of hydrogen, HCl, DCS, TCS, and STC from the decomposition reactors (line 304). The unit separates and purifies the hydrogen for recycle (line 302), recovers HCl for sale (line 310), and recovers chlorosilanes for recycle to the TCS still (line 309).

The recovery unit design shown in fig. 6 is extracted from Ref. 7.

Vent gases from the decomposition reactors (line 304) are cooled to remove some of the chlorosilanes. After the initial refrigeration step, gases are sent to compression and refrigeration (line 305) where most of the remaining chlorosilanes are condensed. Condensed chlorosilanes are removed (line 306 +307) and sent to the distillation area (line 309). The H₂ and HCl, along with traces of chlorosilanes are sent to a charcoal adsorption tower (line 308) for final cleanup of the hydrogen. Purified H₂ is recycled to the decomposition

reactors (line 302). Additional equipment is used to recover the HCl as salable grade (line 310).

WASTE CONTROL

Waste streams requiring treatment are those from the waste settler (line 120) and the stripper column (line 204), and the hydrogen generated in the decomposition reactors (line 311). All other streams are recovered by use of the recovery system.

The vapor streams are washed with water (line 313) in a simple scrubber. The resultant aqueous waste (line 314) will be neutralized with calcium hydroxide and the clean gas will be vented to the atmosphere (line 312).

Liquid wastes (line 120) are disposed of via Dow Corning technology.⁸ The chlorine from this process is neutralized with calcium hydroxide. The silica is collected and appropriate disposition made.

PURITY CONSIDERATIONS

Boron is expected as boron trichloride, generated in the hydrogenation unit. BCl_3 will emerge from the bottom of the stripper column (line 206) and overhead at the TCS still (line 212). From there it will enter the boron removal system. The removal system is a packed column, with selective adsorption of boron trichloride from TCS. This technique has been used to purify STC, with the use of Dowex 3⁹ and other adsorptive media.

Figure 8 is presented to show an alternate technique for boron removal, which would be appropriate if the boron content or adsorption equilibrium is unfavorable for removal via the three still system shown in fig. 7. This system provides a high and low purity stream (with respect to boron) and high and low purity TCS stills. As boron is introduced in the TCS from hydrogenation only, this stream is handled independently of the recycle TCS streams from the redistribution reactor and recovery unit.

Since the TCS from hydrogenation is only about 20% of the

total entering the DCS column, the hydrogenation effluent can tolerate ca. five times the boron concentration as in the three still design described, while maintaining the same boron content in the feed to the decomposition reactors.

Additional data on boron content in the hydrogenation reactor product and adsorption bed behavior are required to determine if the high-low purity system is required.

Phosphorus is expected to accumulate in the STC stream (line 127) until it reaches steady state concentration and is eliminated with the waste settler effluent (line 119). No phosphorus is expected in the DCS still overhead. If necessary, a small column could be added to separate phosphorus compounds from STC prior to feeding the hydrogenation unit, (line 127).

THIN ROD PULLERS

Thin rods or silicon substrates are needed to form the initial circuit for electric current and initial surface area for deposition. The slim rods are produced by heating a larger diameter silicon rod with radio frequency energy, RF, until the silicon melts. A seed is then positioned in the molten silicon and pulled away from the melt. The larger rod is continually pushed into the RF field. Dissimilar rates of pull and feed cause a product that is smaller in diameter and suitable for use as a substrate.

3.5 SAFETY-RELATED DATA ON DICHLOROSILANE

As discussed in the previous Quarterly Report³, a review of the literature relating to combustion and hydrolytic behavior of DCS indicated that under some conditions, DCS/air mixtures spontaneously and violently explode at room temperature. Other accounts have described DCS/air ignition events as unusually forceful, and to some extent unpredictable. These reports are difficult to reconcile with the literature value for autoignition temperature of 100 °C. Moreover, little has

been reported on interaction of DCS and water; there are indications that DCS can ignite when the material contacts a small amount of water.

Accordingly, we have engaged Hazards Research Corp. (HRC) of Rockaway, N.J., to conduct a series of experimental tests relating to the areas of flammability and hydrolytic characteristics of DCS. Four general kinds of tests were conducted; these are listed below and described in more detail in successive sections.

1. Autoignition temperature of DCS/air mixtures.
2. Explosion severity scans of DCS/air mixtures (this experiment involves determining pressure rise rates in a closed vessel subsequent to ignition).
3. Hydrolytic tests. A simulated DCS spill was subjected to a water deluge, and DCS was also spilled into a vessel containing a small amount of water.
4. "Cube test". A DCS/air mixture which exhibited the most powerful explosion in experiment 2. was placed in a wooden cube whose faces were covered with plastic sheeting. The mixture was then ignited with a "squib" inside the cube, and the explosion monitored.

EXPERIMENTAL DATA FROM HAZARDS RESEARCH CORPORATION

Experimental procedures and results from work conducted by Hazards Research Corporation (HRC) on the combustion and hydrolytic behavior of DCS are discussed in this section.

Autoignition Temperature Determination

Autoignition temperature determination was made using a steel autoclave with a volume of 300 ml. A Wood's metal bath was used to provide controlled temperature. A small amount of sample vapor from a reservoir was injected into the thermally

equilibrated vessel at a preselected temperature and the result observed for up to 10 minutes. Temperature of the internal vapor space was monitored using a thermocouple-recorder system. If the result was combustion, a successive trial was performed at a lower temperature; if no combustion was obtained, a higher temperature was used. That procedure was continued until the lowest temperature for combustion and the highest temperature for non-combustion were separated by less than 10 °C. The sample charge was then increased and decreased in subsequent trials to assure that the lowest autoignition temperature as a function of composition had been determined.

Explosion Severity Scan

The explosion severity work was conducted in a ten liter spherical vessel equipped with pressure and temperature monitoring systems. The sphere also contains a rupture disc, a gas injection system and a nichrome coil ignition system. The injection and ignition systems are centrally located.

DCS vapor was admitted to the evacuated sphere to a desired partial pressure, followed by admission of dry air to one atmosphere total pressure. The vessel was allowed to stand to permit mixing for 5 minutes, then the nichrome coil was fused. The pressure history of the reaction was recorded by a fast acting transducer in combination with an oscillograph or, for unusually high velocities, an oscilloscope. Experiments were conducted at ambient temperature.

Hydrolytic Effects

Two tests were conducted to determine the effect of water on DCS. One test consisted of spraying water into a pool of DCS; in the other, DCS was sprayed into a pool of water. Both tests used a pan approximately 12 inches in diameter by 2 inches deep to contain liquid. Five pounds of DCS was placed in a cylinder and released by remote control. Water flow was controlled by remotely located manual valves. Provision was made to ignite vapor clouds resulting from hydrolysis with a

"squib" pyrotechnic igniter.

Explosive Output Trials

In an attempt to simulate an unconfined vapor cloud explosion a cube, 5 feet per side, was constructed of wood and covered with a transparent polyethylene film skin. A small fan within the enclosure provided mixing of the contents and a squib was located within the cube to act as an ignition source. "Bikini" gauges were placed at varying distances from the cube to measure overpressure waves caused by the explosion. High speed photographic equipment was positioned approximately 80 feet away and real time photography was above at roughly a 45° angle to provide visual documentation.

The enclosure started with air inside. The DCS was bled as either vapor or liquid from a cylinder at a remote site to the cube. Cylinder weight change was used to establish when the predetermined amount of DCS had been admitted to the cube. DCS concentration was adjusted to that corresponding to the maximum pressure rise in the explosion severity scans. The process of filling the cube required about 45 minutes.

Results and Discussion

Autoignition temperature for DCS
at a concentration of ca. 23
mole% in dry air: $58 \pm 5^\circ\text{C}$

Four successful (i.e., with ignition) trials were observed between 60 and 70°C . Six unsuccessful trials were observed at 55° over a range of compositions. The autoignition temperature is therefore considered to lie in the range

$58 \pm 5^\circ\text{C}$

Several factors may be responsible for the discrepancy between this figure and the literature value of 100°C . The

vessel used in the current work is somewhat larger than the ASTM standard size AIT vessel; the larger surface area by itself would lead to a lower AIT. The use of steel rather than glass would, however, give rise to a higher AIT value.¹⁰ Finally, if the literature determination did not include a comprehensive scan over a range of compositions, the AIT would have been overestimated.

Irrespective of the precise value of the AIT of DCS in dry air, it is clear that the thermal ignition requirements of such mixtures are very small.

Explosion Severity

DCS/air (17 % DCS)

Expt.	$(dP/dt)_{\max}$ psi/sec	P(static) psig	P(dynamic) psig
1	1.1×10^6	112	280
2	1.1×10^6	125	280
3	0.8×10^6	125	225

Numerous concentrations of DCS in air were investigated to determine what concentration provided the maximum severity. When that concentration was approached (17-20% DCS in air) the oscillographic recording apparatus proved to have inadequate response time for the very high combustion velocities encountered. Data in the above short table result from replicates of a 17% DCS in air mixture whose ignition was monitored on an oscilloscope. In these trials, pressure rise was found to be relatively slow until a pressure of 35 psig was attained (after ca. 3-4 msec). At that point an extremely rapid pressure rise--indicative of a detonation--was evident. Shock wave oscillations at a frequency of 2750 Hz were apparent on the oscilloscope trace. The shock oscillated about a static pressure of 112-125 psig; this was also the final pressure when the shock amplitude attenuated to zero (roughly 12 msec after ignition). The maximum dynamic pressures indicated in the table were attained roughly 100 microseconds after the detona-

tion phase started.

From this test it was established that a detonation can occur with DCS. This is important because detonations are accompanied by blast waves, or overpressure fronts. The blast waves can cause equipment and building damage, whereas in a deflagration there is no blast wave and damage is primarily limited to primary and secondary fires.

These results are obtained from equipment that confines the combustion products. It is possible that the high peak rate of pressure rise is affected by confinement; however, we must assume that partially confined or unconfined vapor clouds of DCS in air could detonate under some conditions.

Hydrolytic Tests

Hydrolytic tests showed no ignition when water was added to DCS, or when DCS was added to water. The hydrolytic reactions were accompanied by copious fuming and left white powdered or rubbery flammable residues. In neither test was spontaneous ignition of the vapor cloud observed.

In the trial in which DCS was discharged into a pool of water, the resultant vapor cloud was successfully ignited with the squib. A flame was also sustained at this point in the pan, even when a water spray was turned on the pan. Reaction both before and after the deliberate ignition was not considered violent.

These results do not conclusively demonstrate that ignition cannot be initiated by addition of water, however. Based on other experience one would conclude that nearly any thermal, mechanical, or chemical reaction energy could be sufficient to ignite a mixture of DCS and air.

Cleanup procedures for the flammable hydrolyzate should prohibit storage in closed containers such as trash cans or bags. If incineration is available as a disposal technique, it would be the best method as it would convert the material into silica.

Explosive Output Studies

Two trials of the explosive output experiment were conducted. The first test, in which DCS was admitted as a gas, proceeded by plan only until the required amount of DCS had been added to the cube. When the valve on the (remote) DCS cylinder was closed, ignition occurred. Unfortunately this unplanned ignition occurred without any photographic or visual documentation. The report was barely audible from the observation stations, and the Bikini overpressure gauges were unaffected.

The second test was run with DCS being admitted as a liquid spray to promote mixing and shorten loading time. Subsequent to loading, the fan was started and run for one minute. Ignition occurred--as scheduled--when the squib was fired. A slight report was heard, but the Bikini gauges did not indicate blast wave overpressures. Both trials produced a rapid deflagration with no detonation. Analysis of the films of the explosion is not yet available.

Based on volume of the report and damage incurred by the cube structure it was estimated that the explosive output was 4-5 times that of the propylene test run as a standard.

This series of tests indicates that DCS is unpredictable in behavior with respect to combustion. Ignition can be induced by something as minor as turning off flow through a line. This fluid dynamic ignition is not understood in detail but is typical of the behavior of silane.¹¹ Although detonation did not occur in this test it is not possible to say that detonation could not occur in a larger vapor cloud. Given sufficient distance the rapid deflagration observed could transform into a supersonic flame front, or detonation.

SUMMARY OF RESULTS FROM HAZARDS RESEARCH CORP.

While the HRC experimental program has not yet been completed, the data obtained thus far have well confirmed our suspicions that DCS/air mixtures are both very easily (and sometimes unpredictably) ignited, and that the explosive poten-

tial from such ignitions is unusually high. Alternative designs and procedures for the PDU and intermediate and production reactors are accordingly being formulated. We are of the firm opinion that, once the hazard potential of DCS is fully appreciated, the material can be safely handled.

Current work underway at HRC includes investigation of combustion characteristics of both DCS/H₂ and redistributed TCS mixtures with air. Results of this work will be described in the next Quarterly Report.

3.6 Preliminary Economic Evaluation of 1000 Metric Tonne Plant

An economic analysis of the HSC DCS-based low cost silicon process has been undertaken. The base conditions for the analysis are those corresponding to a 1000 metric tonne plant, as outlined in section 3.4. Assumptions about process parameters with economic implications are specified in Table 3. Summaries of manufacturing cost and plant investment requirements are presented in Tables 4 and 5.

The estimated product price, including a 20% return on a plant investment of \$21.92 M, is \$19.85/kg silicon. The following section is devoted to a brief discussion of the procedure used for the analysis.

3.6.1 Notes on Procedure used for Economic Analysis

GENERAL

Where possible, the procedure followed has paralleled that developed by C. Yaws and co-workers at Lamar University¹² for evaluating various low-cost solar silicon processes. Capital categories were estimated as percentages of major process equipment cost, direct plant investment costs, etc. When such factors have been used, they have been averages of the corresponding values used by Yaws in his analysis of the standard Siemens¹³ and Union Carbide¹⁴ low-cost silicon technologies.

MAJOR PROCESS EQUIPMENT

Purchased cost will be used for all equipment except for CVD reactors. For these, we will utilize installed reactor cost as a portion of the reactor building cost, since it can be estimated quite accurately. Appropriate factors will be used to convert purchased into installed cost for other equipment. All figures have been converted into 1980 dollars.

The cost figures for hydrogenation and (in part) distillation and redistribution-related equipment have been derived from the cost analysis of Union Carbide Corp., as presented in their LSA Quarterly Reports.⁵ In these cases, equipment has been appropriately scaled or otherwise modified for HSC operation.

OTHER DIRECT PLANT INVESTMENT COSTS - Installed utilities was treated as a separate category. An estimate was made for installation of a substation capable of generating 80 M kwh of electrical power per year.

REMAINDER OF INVESTMENT ESTIMATES - were made on the basis of factors used by Yaws, as described above.

4.0 Conclusions and Recommendations

With the successful conclusion of the experimental reactor program, the next activities scheduled for Phase 1 of the contract were the cylinder-based dichlorosilane (DCS) feed to an "intermediate" sized reactor, and the construction of the Process development Unit (PDU) whose function it will be to generate DCS from redistribution of TCS, and feed the DCS to one or more production scale reactors. These two activities were suspended during the last Quarter, while safety-related data for DCS were being developed by Hazards Research Corp. These data provided new, fundamental information relating to combustion and hydrolytic properties of DCS. To accommodate the new information, the design and operating characteristics of the PDU and intermediate reactor feed systems were extensively revised during this reporting period. These modifications have been approved by the appropriate Corporate Safety Audit teams.

The new safety data do not invalidate the technology of producing high-quality silicon from DCS; rather, they require design modifications to minimize the hazards associated with handling DCS.

The feasibility of generating high quality silicon from dichlorosilane via CVD was further established during this reporting period by fabrication of solar cells (by Westinghouse) from (commercially supplied) DCS-grown silicon, and by growth of silicon containing < 1ppba electrical impurities from redistributed TCS. The efficiencies of the solar cells was very nearly equal to the baseline efficiency established by Westinghouse.

5.0 Program Schedule/Plans

Except in the areas of intermediate reactor DCS feed and PDU construction, as noted above, the program is proceeding according to or exceeding plan. Efforts planned in the various

task areas in accordance with the Program Schedule shown in figure 1 are summarized below.

1. Experimental Reactor Feasibility/Optimization (3.1)
has been completed.
  2. Intermediate Dichlorosilane Reactor Development (3.2)
Finalize new design, operating procedures to
accommodate new safety-related information.
Ascertain feasibility of utilizing unmodified (HSC)
production reactor for this task.
  3. Dichlorosilane Process/Product Evaluation (3.3)
Finalize new design, operating procedures to
accommodate new safety-related information.
Order PDU major equipment items.
Initiate PDU construction.
  4. Preliminary EPSDU Design (3.4)
Identify plant, reactor locations.
Modify Process Flow Diagrams to indicate changes in
PDU design.
- 6.0 New Technology
No new technology was developed during this quarter.
- 7.0 References
1. Hemlock Semiconductor Corp., "First Quarterly Report",
Low-Cost Silicon Solar Array Project, DOE/JPL Contract
No. 955533, January, 1980.
  2. Hemlock Semiconductor Corp., "Second Quarterly
Report", Low-Cost Silicon Solar Array Project, DOE/JPL
Contract No. 955533, May, 1980.
  3. Hemlock Semiconductor Corp., "Third Quarterly Report",
Low-Cost Silicon Solar Array Project, DOE/JPL Contract

No. 955533, September, 1980.

4. Westinghouse R&D Center, "Monthly Technical Progress Report No. 59", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 954331, September, 1980.
5. Union Carbide Corp., "Final Progress Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 954334, 3/79.
6. J. Mui and D. Seyferth, Massachusetts Institute of Technology, "Fifth Quarterly Progress Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 955382, July, 1980.
7. JPL Publication 79-110, "Silicon Materials Outlook for 1980-85 Calendar Years.
8. The proprietary nature of this technology precludes its disclosure in this report.
9. British patent no. 834,380, issued to F.A. Pohl and I. T. Hauskrecht, April 16, 1958.
10. W. Cruice, Hazards Research Corp., Rockaway, N. J.
11. E. Ziemer and H. Balboni, International Business Machines, Essex Junction, VT, private communication.
12. Lamar University, Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 954343.
13. Lamar University, "Eighth Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 954343, September, 1977.
14. Lamar University, "Thirteenth Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 954343, 12/78.

8.0 Acknowledgements

We are grateful to D. Goffnett, L. Jones and H. Smith of Hemlock Semiconductor Corp. for their able assistance in the experimental aspects of this projects, and to D. Mueller for the data reduction of the experimental design program. We further wish to acknowledge contributions in the area of project safety by R. Siegel, R. Salinger, T. Elvey and D. Parsels of Dow Corning Corp.

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APPENDIX A

TABLE 1
KINETIC DATA FROM VAPOR PHASE REDISTRIBUTION
OF TRICHLOROSILANE DOWEX RESIN

Conditions				Products (Mole %)					
Ref No	Dowex	Preparation	Bed Size	Linear Flow (Ft/Hr)	Res Time (sec)	MCS	DCS	TCS	STC
30B	Air	(Inert) Purge	B	10312	0.35	0.0	4.7	90.6	4.6
30C	"	"	"	6714	0.54	0.1	6.2	87.5	6.2
30D	"	"	"	2398	1.5	0.2	8.8	81.8	9.2
30A	"	"	"	720	5.0	0.4	9.7	79.9	10.2
30E	"	"	"	120	30	0.7	12.9	72.8	13.5
18B	100 °C	Vac Bake	A	10312	0.17	0.0	1.0	97.8	1.2
16A	"	"	"	6714	0.29	0.0	1.3	96.8	1.7
16B	"	"	"	2398	0.75	0.0	2.9	93.7	3.3
16C	"	"	"	720	2.5	0.0	5.1	88.6	6.2
18A	"	"	"	120	15	0.5	11.0	76.1	12.4
37C	100 °C	Vac Bake	B	10312	0.35	0.0	1.1	97.9	1.0
37A	"	"	"	6714	0.54	0.0	1.3	97.6	1.1
37B	"	"	"	2398	1.5	0.0	2.7	94.7	2.6
37E	"	"	"	720	5.0	0.2	7.6	84.2	8.0
37D	"	"	"	12	30	0.5	10.8	75.4	12.9
46B	100 °C	Vac Bake	C	3592	0.17	0.0	1.7	97.3	1.0
46E	"	"	"	2338	0.26	0.0	1.8	96.8	1.3
46A	"	"	"	834	0.72	0.1	2.9	94.7	2.3
46C	"	"	"	250	2.4	0.1	5.5	89.4	5.0
46D	"	"	"	42	14	1.4	11.0	74.9	12.7
53C	100 °C	Argon	C	3592	0.17	0.0	0.6	98.8	0.6
53A	"	"	"	2338	0.26	0.0	0.3	99.4	0.3
53D	"	"	"	834	0.72	0.0	1.8	96.3	1.9
53E	"	"	"	250	2.4	0.0	4.6	90.9	4.5
53B	"	"	"	42	14	0.2	8.5	82.9	8.3
44D	165 °C	Vac Bake	C	3592	0.17	0.0	0.5	98.7	0.8
44A	"	"	"	2338	0.26	0.0	0.8	98.2	0.9
44E	"	"	"	834	0.72	0.0	0.9	97.8	1.3
44B	"	"	"	250	2.4	0.0	2.1	95.7	2.2
44C	"	"	"	42	14	0.0	3.9	91.8	4.3

Catalyst : Dowex Ion Exchange Resin

Bed Sizes : A = 1/4" x 6"
(Tubing Wall = 0.035") B = 1/4" x 12"
C = 3/8" x 2"

Bed Temperature : 85 °C

Feed : 40 mole % Trichlorosilane in Helium

Note : Linear flow and residence time have been corrected for the void volume of the catalyst, e.g., $\alpha=0.50$

TABLE 2
 PURITY EVALUATIONS FOR SILICON GROWN FROM REDISTRIBUTED
 TRICHLOROSILANE

Ref No	Silicon Rod Feed Source	Boron (ppba)	Donor (ppba)	Aluminum (ppba)	Carbon (ppma)
022	77 °C Dowex	Bed Conditioning Run - No Rod Grown			
023	Control	0.19	1.1	0.30	0.5
024	24 °C Dowex	0.48	1.4	0.09	0.5
025	Control	0.15	1.7	0.18	0.3
026	77 °C Dowex	0.69	1.2	0.06	0.4

TABLE 3
BASE CONDITIONS FOR ECONOMIC ANALYSIS OF
HSC DICHLOROSILANE-TO-SILICON PROCESS

1. General

Plant size: 1000 metric tonne of silicon
Product: High purity silicon
Process: Production of DCS (SiH_2Cl_2); generation of silicon from reductive chemical vapor deposition (CVD) of DCS

2. Production of TCS (SiHCl_3)

From hydrogenation of STC (SiCl_4)
Fluidized bed reactor, containing metallurgical grade silicon
Reactor operates at 500 °C and 500 psig
Equimolar feed of STC and H_2
Product composition approximately

STC	70%
TCS	29%
DCS	1%

3. Removal/recycle of STC

Distillation (TCS still)
Separation of TCS + DCS from STC
Overhead stream to DCS still
Bottoms stream to hydrogenation reactor

4. Boron removal

Removal of BCl_3 via complexation with nitrogen or oxygen
base on non-volatile support
Fixed bed reactor
Feed from TCS still
Effluent to DCS still
No chlorosilane material loss

5. DCS production

Redistribution of TCS with Dowex ion exchange resin
Pure TCS feed
Liquid phase operation @ 70 °C, 80 psig
Products to TCS still
Product composition as follows

DCS:	10.5% (includes <1% SiH_3Cl (MCS))
TCS:	78.5%
STC:	11.0%

6. DCS purification

Distillation (DCS still)
Feed from TCS still
Separate DCS (+MCS) from TCS
Overhead stream to CVD reactors
Bottoms stream to redistribution reactor

TABLE 3 (continued)
 BASE CONDITIONS FOR ECONOMIC ANALYSIS OF
 HSC DICHLOROSILANE-TO-SILICON PROCESS

7. Silicon production
  - Siemens CVD reactor (modified as appropriate)
  - DCS/H₂ feed
  - Molar conversion to silicon of 40%
  - Deposition rate of 3000 g/hr
  - Vent composition (per mole of DCS fed)

HCl	.14
DCS	.10
TCS	.34
STC	.16
8. Operating ratio
  - 85% utilization (on line time) of CVD reactors
  - 7445 production hours/yr
9. Waste treatment
  - Waste gas streams to water scrubber. Resultant HCl neutralized with Ca(OH)₂.
  - Waste liquid streams processed with current Dow Corning technology. Chlorosilanes neutralized with Ca(OH)₂; silica generate sold or disposed of depending on quality.
10. Storage requirements
  - DCS: 2 hours
  - TCS: 3 days
  - STC: 3 weeks
  - Silicon: 14 days
  - Ca(OH)₂ 14 days
11. Recycle streams
  - Hydrogen from CVD reactors returned to same reactors
  - Chlorosilanes, HCl from CVD reactor vent to recovery system
  - Recovery system condenses, separates HCl, chlorosilanes;
  - HCl sold for credit (\$.12/lb), chlorosilanes returned to TCS still
  - Recovery system modified Siemens technology
12. Slim rod pullers
  - Pull rate average of 470 cm/hr (for each of five machines)
  - Thin rod diameter of 6 mm

TABLE 4.

HEMLOCK SEMICONDUCTOR CORPORATION
 LOW-COST SILICON PROCESS
 MANUFACTURING COST
 PRELIMINARY ESTIMATE

1980 Dollars
 (Manufacturing capital = 21.92M)

	\$/kg silicon
1. Direct Manufacturing Cost	
1.1 Raw Materials	2.78
1.2 Direct Operating Labor	.72
1.3 Utilities	2.97
1.4 Supervision/Clerical	.18
1.5 Maintenance and Repair	2.00
1.6 Operating Supplies	.40
1.7 Laboratory Charge	.18
	\$9.23
2. Indirect Manufacturing Cost	
2.1 Depreciation	2.19
2.2 Local Taxes	.44
2.3 Insurance	.22
3. Plant Overhead (12.3% of 1+2)	1.49
4. Total Manufacturing Cost	13.57
5. General Expenses	
5.1 Administration	.76
5.2 Distribution and Sales	.76
5.3 R&D	.38
6. Product Cost	15.47
7. Profit (20% ROI)	4.38
8. Product Price	<u>19.85</u>

TABLE 5.

HEMLOCK SEMICONDUCTOR CORPORATION
 LOW-COST SILICON PROCESS
 MANUFACTURING CAPITAL
 PRELIMINARY ESTIMATE

1980 Dollars

1.	Direct Plant Investment (battery limit)	
a.	Major Process Equipment (except CVD rxs)	\$2653.1 M
b.	Installation/Instrumentation/Buildings (not including the reactor building (100% of 1a.)	2653.1
	Subtotal	5306.2
c.	CVD Reactors (installed)/Reactor Building	5401.0
d.	Total Battery Limit Investment	10707.2
2.	Other Direct Plant Investment Costs	
a.	Utilities, installed	2300.0
b.	Other direct (general offices, shops etc.) (55% of 1a.)	1266.0
3.	TOTAL OF DIRECT PLANT INVESTMENT	14273.2
4.	Indirect Plant Investment Costs	
a.	Engineering overhead (10% of 3.)	1427.3
b.	Normal contingency (18% of 3.)	2569.2
	TOTAL INDIRECT PLANT INVESTMENT	3996.5
5.	TOTAL DIRECT & INDIRECT INVESTMENT	18269.7
6.	Overall Contingency (20% of 5.)	3653.9
7.	FIXED CAPITAL PLANT INVESTMENT (5+6)	<u>21923.6</u>

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APPENDIX B

Time (Mo.) Project	3	6	9	12	15	18	21	24	27	30	33	36	39
Phase 1 DCS Reactor Feasibility TCS-DCS Redistri- bution Feasibility EPSDU Plant Design Intermediate Rx Design and Construction													
Phase 2 EPSDU Design Production Rx. Eval- uation													
Phase 3 EPSDU Constnction /Demonstration													

FIGURE 1. SCHEDULE OF EFFORTS BY PHASES

FIGURE 2. PROGRAM PLAN MILESTONE SCHEDULE

FIGURE 2. PROGRAM PLAN MILESTONE SCHEDULE (cont.)

FIGURE 3. LABORATORY GAS PHASE REARRANGER UNIT

1- 7µm Sintered Filter

2- Micrometer Valve

3- Rotameters (2)

4- 300 ml Trichlorosilane Cylinder

5- Pressure Gauges (2)

6- Preheater Coil (S.S. Tubing)

7- Catalyst Bed (S.S. Tubing)

8- Oil Bath

FIGURE 4.

KINETIC DATA FROM VAPOR PHASE REDISTRIBUTION OF TRICHLOROSILANE
BY DOWEX MWA-1 RESIN (85°C)

FIGURE 5.
HSC PROCESS
SILICON TETRACHLORIDE
HYDROGENATION SYSTEM

Hamlock Semiconductor Corporation			
PROJECT: 4-40000000		SILICON TETRACHLORIDE HYDROGENATION SYSTEM	
DESIGN: 1-10-80		FLOW SHEET	
REVISED: 1-10-80		SCALE: NONE	
DRAWN BY: [Signature]		HWI-3855	

FIGURE 6.

HSC PROCESS

DICHLOROSILANE DECOMPOSITION
REACTORS AND RECOVERY UNIT

RECOVERY UNIT
PER Y&WS FIG 3-2
JPL PUBLICATION 79-410
"SILICON MATERIALS OUTLOOK
STUDY FOR THE 80-85 CALENDAR YEAR"

Hemlock Semiconductor Corporation									
DICHLOROSILANE DECOMPOSITION REACTORS AND RECOVERY UNIT									
FLOW SHEET									
DESIGNED BY	DATE	REVISED BY	DATE	REVISED BY	DATE	REVISED BY	DATE	REVISED BY	DATE
W. J. HARRISON	10/1/80								
CHECKED BY	DATE	APPROVED BY	DATE	APPROVED BY	DATE	APPROVED BY	DATE	APPROVED BY	DATE
G. J. HARRISON	10/1/80								
SCALE	NONE	HWI-3857							

FIGURE 8.

HSC PROCESS

DCS PRODUCTION UNIT WITH HIGH
AND LOW PURITY TCS STREAMS

REVISIONS										REVISIONS									
1										1									
2										2									
3										3									
4										4									
5										5									
6										6									
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Hemlock Semiconductor CORPORATION

DESIGNED BY
A. H. H. H. H.
CHECKED BY
J. H. H. H.
APPROVED BY
J. H. H. H.

DCS PRODUCTION UNIT WITH HIGH
AND LOW PURITY TCS STREAMS
FLOW SHEET

SCALE: NONE HWI-3858