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EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR
PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE
SILANE-TO-SILICON PROCESS

Quarterly Progress Report, October—December 1979

Work Performed Under Contract No. NAS-7-100-954334

Union Carbide Corporation
New York, New York

U.S. Department of Energy

Solar Energy

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QUARTERLY PROGRESS REPORT

OCT — DEC 1979

low cost solar array project

EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR
PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE
SILANE-TO SILICON PROCESS

**UNION CARBIDE
CORPORATION**

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ABSTRACT

This report covers the work performed in October, November, and December, 1979 on JPL/DOE Contract 954334, Phase III. This phase consists of the engineering design, fabrication, assembly, operation, economic analysis, and process support R & D for an Experimental Process System Development Unit (EPSDU).

EPSDU equipment specification, vendor development, and evaluation of quotations represent a significant effort which will permit the start of ordering EPSDU equipment during the next quarter. Initial mini-burner test results are encouraging. It appears that a fairly simple and cost-effective waste treatment system may be possible. Engineering design packages such as the P & I, electrical one-line, and layout are also progressing well.

The free-space reactor PDU was assembled and is undergoing checkout. The modified seal and shaft of the reactor scraper ran with limited success. This problem will be resolved in January. A computer modeling of the PDU has been prepared using a two-dimensional axisymmetric model with recirculation. Some test runs have been made.

Work on determination of the critical silane decomposition in a fixed bed has been completed. The data generated follow the extension of the Murthy's data. This data, along with the deposition rate data to be acquired, will be used in the fluid-bed reactor design. Present test data show that capacitive heating of the fluid bed and particle separation in a boot are very promising.

The chromatographic method of detecting low levels of boron-in-silane does not seem practical.

SECTION I. INTRODUCTION

This report covers the work performed in October, November, and December, 1979 on JPL/DOE Contract 954334, Phase III.

The overall objective of the LSA Silicon Material Task is to establish a chemical process for producing silicon at a rate and price commensurate with the production goals of the LSA project for solar-cell modules. This material must be suitable for utilization in the large-area sheet process and in the automated process for the fabrication of solar cells having satisfactory physical and electrical performance characteristics.

As part of the overall Silicon Material Task, Union Carbide developed the silane-silicon process and advanced the technology to the point where it has a definite potential for providing high-purity polysilicon on a commercial scale at a cost of \$14/kg by 1986 (1980 dollars). This work, completed under Phases I and II of the contract, provided a firm base for the Phase III program (initiated in April 1979) aimed at establishing the practicality of the process by pursuing the following specific objectives:

- Design, fabricate, install, and operate an Experimental Process System Development Unit (EPSDU) sized for 100 MT/Yr to obtain extensive performance data to establish the data base for the design of commercial facilities.
- Perform supporting research and development to provide an information base usable for the EPSDU and for technological design and economic analysis for potential scale-up of the process.
- Perform iterative economic analyses of the estimated product cost for the production of semiconductor-grade silicon in a facility capable of producing 1000 MT/Yr.

This process for preparing semiconductor-grade silicon in the EPSDU from metallurgical-grade (M-G) silicon is based on a well-integrated arrangement of purification steps that provides a cost-effective process system.

The three basic steps entail converting M-G silicon to trichlorosilane, redistributing the trichlorosilane to produce silane, and thermally decomposing the silane to form polycrystalline silicon powder. The powder is then melted and the molten silicon is cast for subsequent fabrication of solar cells.

The technical progress presented in this report is arranged according to the Work Breakdown Structure (WBS) shown in Table I.

TABLE I WORK BREAKDOWN STRUCTURE

1.1 DESIGN & PROCUREMENT	1.4.4 INITIAL FEASIBILITY DEMO
1.1.1 PROCESS DESIGN	1.4.4.1 Preparation
1.1.1.1 Process Design Update	1.4.4.2 Steady-state 15-day Run
1.1.1.2 Support for Engineering Design	1.4.4.3 Sample Preparation & Delivery
1.1.1.3 Support for Equipment Design/Specs	1.4.5 DATA GENERATION & ANALYSIS
1.1.2 ENGINEERING DESIGN	1.4.5.1 Hydrogenation
1.1.2.1 Control Systems	1.4.5.2 Distillation/Redistribution
1.1.2.2 Layout	1.4.5.3 Pyrolysis/Consolidation
1.1.2.3 Electrical	1982 LSA Goals
1.1.2.4 Safety	1.4.5.4 Waste Treatment & Auxiliaries
1.1.3 EQUIPMENT SPECIFICATION/PROCUREMENT	1.4.6 SILICON CONSOLIDATION R & D, 1986 GOALS
1.1.3.1 Mechanical	1.4.6.1 Hydrogen-Atmosphere Operation
1.1.3.2 Electrical	1.4.6.2 Large-Diameter Rod Casting
1.1.3.3 Instruments & Controls	1.4.6.3 Sample Preparation & Deliveries
1.1.3.4 Other Instruments	1.4.7 OFF-SITE QC ANALYSIS
1.1.4 INSTALLATION DRAWINGS	1.4.7.1 Sample Preparation & Deliveries
1.1.4.1 Site	1.4.7.2 Analysis
1.1.4.2 Civil	1.4.8 FINAL PERFORMANCE RUN
1.1.4.3 Mechanical & Piping	1.4.8.1 Preparation
1.1.4.4 Electrical	1.4.8.2 Optimized Performance Run
1.1.4.5 Instrumentation	1.4.8.3 Sample Preparation/Delivery, Data Anal.
1.1.5 ENVIRONMENTAL	1.4.9 EQUIPMENT DURABILITY ASSESSMENT
1.1.5.1 Environmental Impact Study	1.4.9.1 Hydrogenation, Distillation, Redistribution
1.1.5.2 Noise	1.4.9.2 Silane Pyrolysis & Silicon Consolidation
1.1.5.3 Permits/Special Studies	1.4.9.3 Waste Treatment & Auxiliaries
1.1.6 COST ESTIMATE	1.4.10 SHUTDOWN
1.2 EPSDU-EQUIPMENT FABRICATION/DELIVERY	1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS
1.2.1 MECHANICAL	1.5.1 PROCESS DESIGN
1.2.1.1 Compressors	1.5.1.1 Functional Package
1.2.1.2 Pumps	1.5.1.2 Functional Package Update No. 1
1.2.1.3 Unclassified	1.5.1.3 Functional Package Update No. 2
1.2.2 ELECTRICAL	1.5.2 ENGINEERING DESIGN
1.2.2.1 Motor-Control Center	1.5.2.1 Engineering Package
1.2.2.2 Power Wiring	1.5.2.2 Engineering Package Update No. 1
1.2.2.3 Unclassified	1.5.2.3 Engineering Package Update No. 2
1.2.3 INSTRUMENTS & CONTROLS	1.5.3 COST ESTIMATING
1.2.3.1 Field Instruments & Controls	1.5.3.1 Baseline Estimates
1.2.3.2 Panel Instruments	1.5.3.2 Cost Estimate Update No. 1
1.2.3.3 Data Collection System	1.5.3.3 Cost Estimate Update No. 2
1.2.3.4 Unclassified	1.5.4 ASSESSMENT & REPORTING
1.2.4 OTHER EQUIPMENT	1.5.4.1 Baseline Assessment
1.2.4.1 Tanks/Bins	1.5.4.2 Assessment Update No. 1
1.2.4.2 Heat Exchangers	1.5.4.3 Assessment Update No. 2
1.2.4.3 Reactors	1.6 PROCESS SUPPORT R & D
1.2.4.4 Columns	1.6.1 FREE-SPACE REACTOR DEVELOPMENT
1.2.4.5 Filters	1.6.1.1 PDU Experiments
1.2.4.6 Solids Handling	1.6.1.2 Theoretical Analysis
1.2.4.7 Unclassified	1.6.2 MELTING/CONSOLIDATION
1.3 EPSDU-INSTALLATION & CHECKOUT	1.6.2.1 Melter System Design
1.3.1 SITE EVALUATION	1.6.2.2 Hydrogen-Atmosphere Tests
1.3.2 INSTALLATION	1.6.2.3 Large-Diameter Rod Casting
1.3.2.1 Site	1.6.2.4 Shotting Feasibility Assessment
1.3.2.2 Civil	1.6.3 FLUID BED DEVELOPMENT
1.3.2.3 Structural	1.6.3.1 Fixed-Bed Deposition
1.3.2.4 Mechanical	1.6.3.2 Fluidization-Experiments with Inert Gas
1.3.2.5 Electrical	1.6.3.3 Evaluation-Fixed Bed/Fluidization
1.3.2.6 Instruments & Controls	1.6.3.4 Fluid-Bed Pyrolysis
1.3.2.7 Other Equipment	1.6.3.5 Fluid-Bed Process Design for EPSDU
1.3.2.8 Piping	1.6.3.6 Fluid-Bed Model Update
1.3.3 CHECKOUT	1.6.4 QUALITY CONTROL
1.3.3.1 Safety	1.6.4.1 Silane Analysis - Slim-Rod Deposition
1.3.3.2 Operational	1.6.4.2 Silane Analysis - Direct Measurement
1.4 OPERATION	1.6.4.3 Silane Analysis - Epitaxy
1.4.1 PREPARATION	1.6.4.4 Float-Zone Refining
1.4.1.1 Operating Manual	1.7 MANAGEMENT AND DELIVERABLES
1.4.1.2 Training	1.7.1 PROJECT MANAGEMENT
1.4.1.3 Inspection & Calibration	1.7.1.1 Administration
1.4.2 START-UP	1.7.1.2 Contract Maintenance
1.4.2.1 Waste Treatment	1.7.2 PROGRAM MANAGEMENT
1.4.2.2 Hydrogenation	1.7.2.1 Technical Direction
1.4.2.3 Distillation/Redistribution	1.7.2.2 Cost/Schedule/Documentation Services
1.4.2.4 Pyrolysis/Consolidation	1.7.3 DELIVERABLES
1.4.3 MODIFICATION	1.7.3.1 EPSDU Facility
1.4.3.1 Design	1.7.3.2 Material Samples
1.4.3.2 Procurement	1.7.3.3 Data
1.4.3.3 Installation	

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SECTION II. TECHNICAL ACTIVITIES (BY WBS NUMBER)

1.1 EPSDU - DESIGN/PROCUREMENT

1.1.1 Process Design

During this quarter, several reviews of engineering work have been accomplished in order to improve the EPSDU process and to reduce the capital and operating costs. Items reviewed include the process and instrumentation diagram, process equipment requirements, layout, electrical one-line, and the waste treatment system.

A review was made of the data collection points and the engineering parameters to be measured. This information is being incorporated into the EPSDU data collection schedule along with duration and frequency of each data-taking and sampling. A review of the process and instrumentation diagram took into account the data collection schedule including sampling for the quality analysis.

Process equipment requirements were reviewed and certain pieces of non-critical equipment were deleted or simplified for increased facility reliability and cost effectiveness. A review of vendor response to process equipment was also made and found that some vendors did not understand our requirements or were unsuitable. Additional vendor development is being pursued.

The existing facility layout was reviewed and several minor improvements were suggested, generally to functionally simplify operation. The electrical one-line diagram was reviewed to ascertain what the motor-control center must provide and how control improvements can be made.

Modifications were suggested for the waste-treatment area which would reduce equipment costs by not recovering by-products as we

would do in a commercial facility. There are four cost-cutting schemes under examination. The most attractive scheme will be selected in January.

A visit to our Sistersville facility was made to discuss equipment-related items as to materials of construction, equipment sealing, and to discuss quality assurance techniques. The results will be reviewed by engineering personnel and appropriate action will be taken.

The first stage of the mini-burner test, which consisted of flame-hydrolyzing STC vapor, was ended. While initial data seemed encouraging, an insufficient amount of STC was vaporized to collect enough data to model. It was concluded that the inability to vaporize sufficient STC was because of the low ambient temperature and was not worth correcting at this time. If questions about vapor STC reaction still exist, the equipment can be moved indoors after the liquid STC and sludge testing is complete. Testing with liquid STC will now commence. Sampling train improvements were made to allow a mass balance closure, as well as improve data quality. The burner test set-up was photographed and is presented in Figures 1 and 2. Modifications have been made to the burner so that liquid silicon tetrachloride can be effectively flame-hydrolyzed to silica. An improved atomization and flame-receiver design has greatly enhanced the flame reactions. Further improvement in the atomizer design shall be attempted before completing the liquid silicon tetrachloride studies. The current atomizer breaks up the liquid effectively, but imparts too high a linear velocity, so that the droplet does not have sufficient residence time in the flame zone for complete hydrolysis. A wider spray angle will be tried.

1.1.2 Engineering Design

1.1.2.1 Control Systems

An internal Engineering team review of the process and instrumentation diagram was made, and recommended changes were noted on the master copy. The P&I diagram is not complete in the waste treatment pending selection of

Figure 1 Waste Burner Test Apparatus

Figure 2 Waste Burner Assembly

a simplified scheme in accordance with Section 1.1.1, Process Design, above. The pyrolysis subsystem is also on hold, pending free-space reactor PDU test results in accordance with Section 1.6.1.1 PDU Experiments. These will be developed in the next quarter.

Instrumentation and control components will be purchased using several itemized equipment lists. Specifications were initiated for the automatic valve list, local instrumentation list, and panel instrumentation list.

The requirements for the computerized data acquisition system were defined and a preliminary equipment list was developed. Specific software requirements will require additional review as the data acquisition plan develops.

1.1.2.2 Layout

A total of six drawings was released which incorporate all current facility design information. The drawings include a site plan with underground utilities noted, and initial plans and elevations of the various EPSDU facility processing areas, including a switchgear/control panel layout. Figure 3 represents the overall EPSDU facility layout based on current design philosophy.

The EPSDU site plan was submitted to UCC/East Chicago operations personnel for review. Waste disposal ponds which now occupy a portion of the EPSDU site will be decommissioned and back-filled in late Spring 1980.

1.1.2.3 Electrical

A revised electrical one-line diagram was issued showing current expected equipment power loads based on the most recent equipment summary update. Alternates are being evaluated for use of either one-2,000 KVA transformer arrangement or two-1,000 KVA transformers.

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1 - Fuel Oil Storage Tank	22 - Pyrolysis H ₂ Compr.
2 - Hot Oil Furnace	23 - Silane Shift Tanks
3 - STC Storage Tank & Pumps	24 - Argon Storage Tanks
4 - TCS Storage Tank	25 - Melting & Consolidation
5 - STC Recycle Vaporizer	26 - Undefined Area for Future Melter Tests
6 - Muriatic Acid Storage Tank	27 - Sanitary Sewer Water Lift Station
7 - Waste Burners	28 - Quality Control & Analysis Building
8 - Waste Gas Induction & Agglomerator Blowers	29 - Shower & Locker Room Building
9 - Silica Venturi & Muriatic Acid Pumps	30 - Offices
10 - Weak & Strong Muriatic Scrubber & Pump Tank	31 - Acid Water Containment Trench
11 - Hopper Building	32 - Gas Superheater
12 - Refer Skid	33 - Cooling Tower
13 - Waste Silica Neutralizer	
14 - Lift Station	
15 - Leaping Weir Station	
16 - SW Gear & Controls Building	
17 - Transformers	
18 - H ₂ Storage Tank	
19 - H ₂ Compressor	
20 - Columns & Reactor Gantry	
21 - DCS Distal., TCS Distal., Quench Cond'r, & Quench Cond'r. wash pumps	

Figure 3 PRELIMINARY EPSDU FACILITY LAYOUT (UPDATE 1)

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The local power company (NIPSCO) was contacted to examine the possibility of an alternate, more cost-effective supply of primary power to the EPSDU facility. NIPSCO submitted a proposal for supply of 12.5 KV power. A technical and economic comparison of the NIPSCO power supply arrangement versus the currently proposed UCC in-plant supply arrangement will be completed in the next quarter. This study includes additional design work covering tie-in of the EPSDU facility power system into the existing East Chicago plant power system.

The EPSDU requires an emergency power supply should the primary power fail from technical and safety considerations. Work is underway to define the extent of the emergency power requirements.

1.1.2.4 Safety

The revised layout drawings were reviewed by the safety engineer and recommended changes were made prior to release. Periodic safety reviews will continue to occur throughout the design period.

1.1.3 Equipment Specifications/Procurement

Equipment power requirements were reviewed in conjunction with the NIPSCO supply proposal above. A total connected load of 1700 KVA and an estimated demand of 1100 KW is predicted.

A summary listing of vendor responses to quotation requests was prepared to identify specific equipment items where sufficient bid information is lacking. Based on this listing, additional candidate vendors have been identified and requests for quotation (RFQ) have been issued. Some favorable followup quotations have already been received.

Preparation of RFPs (Requests for Purchase) was initiated, based on technical, cost, and schedule evaluation of submitted vendor quotations. Equipment purchases were prioritized to identify long-lead items and items requiring vendor-supplied drawing information needed prior to initiation of the detailed installation design effort. Initial equipment orders will center on process-related items including tanks, bins, reactors, heat exchangers, and hydrogen compressors. Follow-up quotations were also processed for several items with new or revised specifications, based on continuing process development efforts.

Pieces of custom process equipment have been identified for further mechanical design prior to reissue of RFQs. Continued activity in this area is expected for the next quarter.

1.1.4 Installation Drawings

Design support was provided to cost engineering on preliminary piping and manual valve specifications, electrical specifications, and equipment foundation requirements.

1.1.5 Environmental

The engineering memorandum documenting all environmental aspects of the EPSDU has been issued. The air discharge process diagram has received initial review by appropriate Indiana State Air Quality (ISAQ) officials. Additional information requested by ISAQ was supplied based on the originally proposed waste treatment scheme. Permit application information for wastewater discharge was submitted to the East Chicago sanitary district officials.

While use of one of the proposed, simplified waste treatment schemes requires resubmittal of air and water discharge information, we do not expect any delays in obtaining the necessary permits.

1.1.6 Cost Estimate

Detailed cost estimating activity was initiated this quarter including preliminary designs necessary to establish installation costs for the hopper structure and process equipment structure. Detailed material and labor installation cost take-offs were completed for major process piping, instrumentation tubing, power wiring, control wiring, and equipment foundations. Detailed equipment cost summaries were also completed. The above information will be used to assemble the detailed cost estimate for the EPSDU. The cost estimate will then be incorporated into the baseline budget and the Performance Measurement System (PMS) documents under preparation.

1.3 EPSDU-INSTALLATION AND CHECKOUT

1.3.1 Site Evaluation

A contract was awarded to Dames and Moore, Chicago, and work completed for soil boring and sample analysis work at the East Chicago, Indiana EPSDU site. The contract also included survey work to establish topographic features to define site preparation activities and associated costs. The contractor's findings, contained in their detailed report, favor use of auger-cast type pilings to support concentrated loads including structures and heavy equipment items.

1.6 PROCESS SUPPORT R & D

1.6.1 Free-Space Reactor Development

1.6.1.1 Process Development Unit (PDU) Experiments

Mechanical assembly of the PDU is virtually complete and the system checkout has begun. Figures 4 through 7 show the free-space reactor, the silicon powder hopper, and other pieces of equipment which comprise the PDU. Figure 8 shows the induction heater motor-generator, and Figure 9 shows the control panel.

The new long power cables have been installed for the induction heater and the heater has been retuned for optimum performance on the new inconel reactor. The operational settings are significantly different from the settings used at Parma with the monel reactor and the shorter cables. In a heating test, using a 20 KW power supply input to the coils, a temperature of 700°C was achieved within 45 minutes. The reactor was in place on the hopper but no cooling water was used in the reactor top. The maximum temperature of the reactor top was approximately 150°F. This performance is considered adequate and we anticipate no problems in achieving 800°C in the pyrolysis zone within an acceptable amount of time.

Procurement of the supplies required for the first 24-hour run has been completed. Three spare quartz liners have been acquired as back-up for the reactor, in the event that heavy silicon deposition results in overstressing and cracking of the quartz liner. Sufficient silane has been accumulated for shake-down and the first run.

A safety review meeting was held in November. Major hazards such as silane handling, hydrogen handling, waste gas disposal, electrical hazards, and fire control were addressed. The actions taken to control these hazards on the project were deemed adequate.

Startup of the PDU was delayed in December because the reactor scraper shaft failed to seal properly. Initial inspection revealed pitted surfaces on

Figure 4 Free-Space Reactor, Top Flange Assembly

Figure 5 Free-Space Reactor and Silicon Powder Hopper

Figure 7 Gas Cannon on the Silicon Powder Hopper

Figure* 8 Induction Heater Motor-Generator for Free-Space PDU

Figure 9 Control Panel for the Free-Space PDU

the shafts and a possible shaft misalignment which probably contributed to the cause of the seal failure. A short-term program is now underway to improve seal performance. To minimize the sealing and eliminate the alignment problem, a single shaft will be used. A shaft vendor has been found who will guarantee the shaft surface to our finish specifications for this special application. In addition, Ribbon-Pak formed-in-place Grafoil rings will be used (instead of pre-formed rings) to compensate for minor alignment problems.

A longer lead time solution of using a flexible metal bellows seal is being pursued to allow operation of the PDU, if attempts at improving stuffing-box performance are not successful. Since a bellows will be used on the EPSDU free-space reactor, testing one on the PDU could be useful, although it is undesirable from a cost viewpoint.

The remainder of the PDU is nearing operation status. The automatic sampling chromatograph is currently being set in the instrument shop and the hygrometer probe holders are being modified to mate with our piping system. Both items should be in place by early January. Leak testing of the system, using both soap and a helium detector, has revealed several leaking welds and fittings. These items have been removed and sent to the fabrication shop for rework. The vacuum pump is being relocated to minimize pressure drop in the line connecting it to the hopper. Several minor changes to the purge systems will permit greater use of nitrogen and minimize the argon required during startup. Nitrogen is readily available and is desirable from the cost standpoint, but argon must be used when purity is required. Successful completion of these areas could result in operational startup during January, assuming that the new shaft seals are effective.

1.6.1.2 Theoretical Analysis

Intracompany engineering memorandum (No. 6506; SGS No. 32) entitled "Free-Space Reactor Modeling" was issued on October 4, 1979. This report documented our investigation of the Galloway model of the free-space reactor, the JPL model of a plug flow silane reactor, and various confined-jet models.

The conclusion of the report was that a two-dimensional model capable of accurately portraying recirculation in the free-space reactor should be investigated.

A computer program "FREESPACE" based on "TEACHT" capable of modeling the fluid mechanics and heat transfer in the free-space reactor has been developed. Runs at various Reactor Radius/Nozzle Diameter (R/d) ratios and silane throughputs were conducted.

The program solves the conservation equations for momentum, mass, and energy. The time-averaged forms of the Navier-Stokes equations are used, along with the K-E turbulent energy dissipation effective viscosity model. The fluid is assumed homogeneous and its density, thermal conductivity, and heat capacity are assumed constant. The chemical reaction is assumed to occur in a specified temperature range. Test cases for laminar and turbulent heat transfer in pipe flows have compared favorably with analytic solutions. Nusselt number variations along the pipe length for confined-jet runs at small R/d ratios have compared favorably with experimental results published in the literature.

Two runs were made to determine the predicted effects of cooling the reactor lid. The first case was run assuming the reactor lid to be insulated, and the second run was made holding the lid temperature constant at room temperature. As Figure 10 shows, when the lid is not cooled, the hot zone where the reaction is assumed to occur extends very near to the nozzle tip. This prediction is verified by the experimental observations of silicon deposition on the nozzle when the lid was not cooled. The run in which the lid was cooled shows the hot zone has moved down the reactor away from the nozzle, yet it is far enough away from the walls to prevent wall reaction. The fluid streamlines suggest that all the fluid will pass through the hot zone before contacting the wall or passing through the reactor, so complete conversion should be attained.

Detailed modeling of the kinetics is extremely difficult and probably beyond the scope of our modeling effort. Several factors account for the difficulty:

- The resulting equations become "stiff" due to the difference

Figure 10 PREDICTED EFFECT ON LOCATION OF THE HOT ZONE WHEN REACTOR LID IS COOLED OR INSULATED

in magnitude between the time scales of the fluid mechanics and the chemical reactions.

- The reaction rate of silane decomposition is an extremely strong function of temperature and is also a function of silane concentration. Since turbulent fluctuations of both temperature and concentration are too great to accurately determine the reaction rates, these turbulent fluctuations would have to be determined (extremely difficult) or a suitable model from turbulent combustion such as Spaulding's eddy breakup model used (difficult).
- The accuracy of the available experimental data on rates of silane decomposition does not warrant the use of such methods.

However, the program is being expanded to include the radiation heat transfer, the heat released due to the chemical reaction, and the species conservation equation for silane. More specifically, the amount of heat released by complete conversion of silane in the reactor equals the mass throughput of silane times the heat-of-reaction of the silane decomposition $\text{SiH}_4(\text{g}) \rightarrow \text{Si}(\text{s}) + 2\text{H}_2(\text{g})$. Preliminary estimates obtained from the Galloway model indicate that in the experimental reactor the amount of heat released by the reaction is roughly of the same magnitude as that entering the reactor by radiation and convection from the heated reactor wall. To account for the effects of the chemical reaction, the species conservation equation for silane has been added to the computer model for the free-space reactor. Through solution of this equation, and the addition of a heat-of-reaction source term to the energy equation, the heat release due to the chemical reaction can be modeled. In addition, the concentration profiles of silane in the reactor can be calculated to determine the degree of conversion in the reactor. At present, the chemical kinetics are modeled by an Arrhenius expression of the type $r = A \exp(-\Delta E/RT) C_{\text{Si}}$. Effects of turbulent fluctuations of the temperature and silane concentration on the reaction rate can be modeled by adjusting the constants in the Arrhenius equation to agree with experimental results or by using simple fluctuation models. Adding more equations to solve for the temperature and concentration

fluctuations seems unjustified due to the questionable quality of the experimental data and the considerable increase in program run-time that would result.

The runs made to date have contributed to our physical understanding of the processes in the free-space reactor. The following are several important observations.

a. The Galloway model does not appear to accurately represent the fluid recirculation. "FREESPACE" integrates the axial velocity profile to determine the ratio of fluid recirculation to mass flow rate at any point in the recirculation region. Results show the maximum volume flow of recirculating fluid occurs considerably before the reattachment point, where it falls to zero. The Galloway model chooses the reaction distance to be at the point where the volume flow of recirculating fluid equals the volume flow of fluid moving forward. The distances calculated, however, are very near the reattachment points observed experimentally and predicted by "FREESPACE".

b. The standard pipe flow turbulence criterion that a flow is turbulent if $Re > 2300$ and laminar if $Re < 2300$ does not apply in the vicinity of a sudden enlargement. A local criterion for turbulence based on the local properties of the flow such as the turbulent Reynold's number $R_t = \frac{\rho K^2}{\epsilon \mu}$ as defined by Jones and Launder (where K is the turbulent kinetic energy and ϵ is the rate of turbulent energy dissipation) is more appropriate. In the free-space reactor, the jet Reynold's number is > 2300 , while the reactor cylinder Reynold's number is < 2300 . However, the flow in the cylinder is not completely laminar. Where the jet issues into the reactor, significant turbulent mixing will occur due to the shearing of fluid streams moving at different velocities. Once the jet has reattached, fully developed laminar flow will develop if the reactor is long enough. Ideally, a turbulence model capable of spanning the transition from turbulent to laminar flow is needed. However, the results obtained from the high Reynold's number $K-\epsilon$ turbulence model presently being used appear acceptable.

c. The time averaged energy equation can be written in the following form:

$$\rho \frac{DT}{Dt} = \nabla \cdot \left(\frac{K}{C_p} + \frac{\mu_T}{Pr_T} \right) \nabla T$$

The term on the right represents the effects of heat conduction. The coefficient $\frac{\mu_T}{Pr_T}$ represents the enhancement of conduction by the turbulence of the fluid flow. In most turbulent flow, the turbulent Prandtl number $Pr_T \sim 1$, and the turbulent viscosity $\mu_T \gg \frac{K}{C_p}$, so that heat conduction is almost solely a function of the turbulent intensity, and not of the fluid properties. Test runs indicate that heat transfer by such turbulence enhanced conduction is important in raising the centerline temperature.

1.6.2 Melting/Consolidation

Melter Subcontract: In response to the RFP for melter subcontract work, proposals were received from four candidate subcontractors with two vendors declining to bid. These technical proposals were reviewed in detail by an evaluation team, whose members included an outside consultant. The proposals were rated for seven criteria, specifically; technical success, reliability and scale-up, product purity, 1986 cost goals, product form, fabrication facilities, and schedule. The evaluation⁽¹⁾ resulted in ranking the proposals.

Negotiations with the winning subcontractor have begun, and a tentative agreement was reached on manpower requirements and work schedule⁽²⁾. The work is expected to start on March 1, 1980 and extend through August 1981. Task 1 - Melter Development will be completed in 16 months and Task 2 - Equipment Functional Design will take an additional 2 months. The subcontractor will provide preliminary equipment specifications for the EPSDU melter by the end of February 1981. The melter PDU assembled for development work will be suitable for transfer to EPSDU where it can be installed and used for silicon consolidation.

Fact finding v correspondence was initiated with the second vendor as a possible backup.

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- (1) S. K. Iya, "Silicon Melter System Proposal Evaluation", Memo to H. Morihara, November 6, 1979.
- (2) S. K. Iya, "Melter Subcontract Negotiations", Memo to H. Morihara, November 30, 1979.

Silicon Powder Transport: A simple test rig was assembled to study pneumatic transport characteristics of free-space pyrolysis powder. Initial tests have demonstrated the viability of this technique. The powder, when dry, can be conveyed vertically and horizontally through a 3/4-inch diameter plastic line with a hopper pressurized at 5 psi. Rat-holing of the powder was observed at times, and can be eliminated, presumably, by either fluidizing from the bottom or by vibration.

1.6.3 Fluid-Bed Development

1.6.3.1 Fixed-Bed Deposition

Work on the determination of the critical silane decomposition curve has been completed. Data has been collected in the range of 600° to 900°C and 1 to 20% silane. These tests involve running the experiments at a specific silane concentration in the feed at various bed temperatures. At the end of each run, the bed particles are examined to see if fine powder formation resulted. The experiments are then repeated at other silane feed concentrations. The test apparatus is shown in Figure 11.

Initially, a series of runs at 1% silane-in-hydrogen was conducted at 100°C intervals in the temperature range of 600° to 900°C. Powder was seen only at 900°C. At 10% silane, powder was formed at 750°C but not at 700°C. One run with 15% silane indicated no powder formation at 700°C. These and other data points are shown in Figure 12, along with literature data from Everstein⁽³⁾ and Murthy⁽⁴⁾. This curve is a function of the silane concentration in the feed and the temperature at which the silane decomposition occurs. If the temperature or the silane concentration is too high, unfavorable homogeneous decomposition occurs. It is clear that the present data below 900°C fits well with the results of Murthy obtained at or above 900°C.

(3) F. C. Everstein, Philips Res. Repts., 26, 134 (1971)

(4) T. U. M. S. Murthy, et al, J. Crystal Growth, 33, 1 (1976)

Figure 11 Fixed-Bed Test Apparatus

Figure 12 TEMPERATURE DEPENDENCE OF CRITICAL SILANE CONCENTRATION

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Scanning electron micrographs of the bed of silicon particles, after silane decomposition, shows that fiber growth is seen in all cases of heterogeneous decomposition. Fiber growth was also seen in previous work at Parma⁽⁵⁾ and by other investigators. Some nodular growth was also observed and is the preferred type of morphology; however, the most dominant type of growth is by fibers.

To test the hypothesis that the fiber growth on the silicon particles is due to impurities or surface residue, some silicon particles were etched by washing first in HCl to remove the iron added by grinding, and then washed with a mixture of acetic acid, nitric acid, and HF (1:1.5:1 respectively). The particles were then washed with de-ionized water, followed by a washing with methanol, and then tested in the fixed bed. Fiber growth was found to be substantially reduced but not eliminated. All future tests will be conducted with cleaned silicon particles.

The fixed-bed reactor has been modified to permit the collection of deposition rate data. These experiments have started and will continue next quarter.

1.6.3.2 Inert-Gas Fluidization

Capacitive Heating Tests: Heating of a silicon fluidized bed to high temperatures was tried this quarter. The apparatus used is shown in Figure 13. Test No. 19 was run at 850°C and Test No. 20 was conducted at 750°C bed temperature. There were problems maintaining a stable electric field with Test No. 19 conducted at a U/U_{mf} of 6. Test No. 20 was conducted at a U/U_{mf} of 7.5 and was electrically very stable in performance. Sintering rates were 3.8% per day and 1.1% per day, respectively. Since both tests were run at the same power (about 200 watts) and Test No. 20 was at higher impedance, the increased sintering at 800°C can be attributed to either the higher temperature or the lower U/U_{mf} .

(5) Silane Pyrolysis in a Fluid Bed Reactor, P. J. Timmel and J. Rexer, Report No. PR-79-230, April 26, 1979

Figure 13 Capacitive Heating Test Apparatus

The second possibility seems more probable, considering the erratic electrical behavior experienced.

Test No. 20 was operated for a total of 44.7 hours, including 29 hours of continuous operation. At the termination of the test, performance was still very stable and trouble-free.

Besides establishing acceptable sintering rates at high temperatures, the second objective of these tests was to demonstrate a reactor wall temperature significantly lower than the silicon bed temperature during steady-state conditions. This was accomplished as shown in Figures 14 and 15. For Test No. 20, the bed height was increased to 8.0 inches so that the electric field could be established above the TCs in the bed to eliminate interference. The lower operating temperature is the result of lowering the guard temperature. The capacitive power was 200 watts in both cases.

Test No. 21 was conducted to determine if by resistively heating the top of a fluidized silicon bed, a favorable ΔT could be established at the bottom of the bed with respect to the reactor wall. The upper guard heater was used as the heat source. It was found that the bed and wall were at about the same temperature throughout the bed height. Possibly, using an immersion heater at the top of the bed will result in the desired ΔT being achieved at the bottom. No future work is planned to pursue this concept at the present time.

The next series of tests will have the following features:

- a. High temperature test at Bed/Wall ΔT of 50°C or greater.
- b. Evaluation of high purity silicon electrode.
- c. Low frequency test at 8000 Hz.

Tests having a high Bed/Wall ΔT are accomplished by reducing the guard heater temperatures and increasing the capacitive heating power input to about 450 watts. This power input is more than double the power setting used for Test No. 20. A consequence of the higher density has been the need to increase the superficial velocity to prevent bed sintering.

Figure 14 TEMPERATURE PROFILE IN FLUIDIZED BED, $U/U_{mf} = 6.0$

Figure 15 TEMPERATURE PROFILE IN FLUIDIZED BED, $W/U_{mf} = 7.5$

An electrode assembly was designed for use in the 6-inch diameter pyrex column and is presently being fabricated. It consists of two pairs of electrodes attached to dielectric crossarms which can be rotated with respect to each other to adjust electrode spacing. The entire assembly can be adjusted vertically to vary electrode penetration into the silicon bed.

Particle Separation Tests: Two tests were conducted using the 2.0-inch boot and beds of wide particle size distribution, as shown in Figure 16. For Test No. 9, the initial diameter ratio and coarse fraction were respectively 2.48 and 0.51, while for Test No. 10, they were 2.29 and 0.55, respectively.

With Test No. 9, maximum boot coarse fraction was achieved in 0.6 hr. It was found that by reducing the superficial velocity at the bottom of the 6.0-inch column, 37% of the separation process could be repeated. The reduction in boot velocity was 14%.

With Test No. 10, maximum boot coarse fraction was achieved in 1.0 hr. For this test, the pressure at the bottom of the 6.0-inch column was held constant, whereas with all previous tests, the pressure above the bed was maintained constant at 1/6 atm. Test was conducted at a U/U_{mf} of 1.25 at the bottom of the column. As the bed mean size decreased with selective removal of coarse particles, the velocity was reduced to maintain the 1.25 U/U_{mf} .

The initial velocity at the top of the boot was 0.94 ft/sec, which is a U/U_{mf} of 2.19 for the coarse fraction and a U/U_{mf} of 9.4 for the bed mean size. By running the test at these conditions, it has been possible to achieve three boot coarse fraction peaks in a 4.5 hour period. However, the magnitude of the peaks has been decreasing from 0.98 to 0.81 to 0.68 coarse fraction. The test was continued until separation became marginal. An interesting point to note is that significant separation was achieved for a small diameter ratio (2.29), which was not possible with the binary system tests. It should also be noted that the wide particle size distribution systems have coarse fractions (i.e. fractions above the mean particle diameter) greater than 50% and that may be the reason separation is large, even for a smaller diameter ratio.

Figure 16. Particle Separation Test Unit

From the particle separation tests conducted to date, it appears that segregation of coarse fraction into the boot can be maintained for long intervals only if U/U_{mf} at the base of the column is not very high.

In order to facilitate particle segregation at a wide range of superficial velocities in the column, we investigated the performance of a terminal velocity orifice separator at the top of the boot. Test No. 11, using the 7/16-inch diameter orifice separator, has been completed. A bed of wide particle size distribution was used. Orifice superficial velocity was varied from 4.6 ft/sec to 37 ft/sec. Only marginal separation was obtained with the maximum coarse fraction collected being 0.68 vs. 0.55 for the bed.

Test No. 12, using a 1/4-inch diameter orifice, exhibited results similar to Test No. 11. With the 1/4-inch orifice, it has been possible to throttle or completely shut off the silicon passing through the hole. However, the silicon composition does not seem to vary with this rate.

It is clear from these tests that particle segregation, using a terminal orifice separator, is very difficult to attain; and that a boot concept is the most promising. Since the separation efficiency appears improving as the boot diameter is decreased, a 1½-inch diameter clear plastic boot section and a 6-inch diameter vacuum hopper have been designed and are in the fabrication process. It is expected that these will be completed and installed on the column in January.

1.6.4 Quality Control

The preliminary analytical plan for EPSDU was reviewed to improve the definition of the mechanical equipment required. Consultation with other groups within Union Carbide specializing in on-line sample analysis and especially with chlorosilanes was beneficial in identifying standard commercial hardware which could be used. Arrangements were also made to utilize the analytical methods currently used by UCC for analysis of metals in chlorosilanes by atomic adsorption. The basis for sample frequency, time required for analysis and the particular analysis scheme were also reviewed and updated from the preliminary analytical plan in preparation for more detailed system design.

1.6.4.1 Silane Analysis - Slim-Rod Deposition

The assembly of the slim-rod reactor unit for pyrolysis of silane into a rod-form suitable for float zoning and electrical characterization is about 80% complete. The reactor itself has been completed and is being prepared for leak testing. Installation of the control panel has been delayed awaiting delivery of the cabinet, which is expected in January. Utility installation for the reactor will begin once the control panel is completed.

1.6.4.2 Silane Analysis - Direct Measurement

Candidate chromatographic columns were prepared for the diborane-silane separation. These liquid/solid component systems were discovered through a literature search to have the highest probability for successful separation of the two components. Initially, a thermal conductivity detector (TCD) was used, whose sensitivity is on the order of 100 nano grams of material. This is not sensitive enough for the parts-per-billion sensitivity ultimately desired. However, it should be adequate to screen candidate columns and to be useful in measuring the B_2H_6 - SiH_4 vapor-liquid equilibrium (VLE) at the 1% concentration level. More sensitive TCD systems have been identified for parts-per-billion level analysis.

A dioctyl phthalate on Chromasorb P column elutes diborane after a desirable retention time. However, elution volume was not reproducible between injections. Peak areas increase with repeated injections and return to about their initial value when a modest time elapses between a series of injections. No foreign peaks are noted and potential contamination from moisture has been largely eliminated through elaborate means to dry all parts of the system including the carrier gas and sample handling plumbing. Problems with the chromatograph sensitivity to diborane were resolved by substituting a thermistor detector for the original thermal conductivity type. The thermistor detector showed higher stability and sensitivity. During the next period, the actual chromatographic separation of silane and diborane will be attempted preceding the vapor-liquid equilibrium studies.

A vapor liquid equilibrium (VLE) cell, Figure 17, is being modified for the B_2H_6 - SiH_4 system. Modifications include a method for assuring that a 100% liquid sample is obtained and that minimum sample quantities are used without disturbing cell composition. An estimate of expected vapor and liquid composition and quantity of material to be used in the equilibrium cell was made using published values of silane and diborane properties.

At the planned level of 1% B_2H_6 in SiH_4 , predicted vapor and liquid compositions of 0.4 and 1.0% respectively were obtained at $-25^{\circ}C$, 300 psig. If these predicted values are confirmed, the use of distillation to purify silane from silane-diborane mixture will be assured.

An evaluation of the use of infrared spectroscopy as a direct method of analysis of silane or silicon for low levels of boron, phosphorus, and other electrically active species is in progress. A Fourier Transform Infrared (FTIR) spectrophotometer using a 120 meter path length cell was roughly estimated to achieve a 10 part-per-billion detection sensitivity for diborane in silane based on a recent single test. Analysis of poly- and single-crystal silicon by FTIR for carbon, oxygen, boron, and phosphorus has been reported in the literature and is now a relatively common commercial practice.

A literature search was undertaken to consider the possibility of using a flame photometric detector on a gas chromatograph for analysis of diborane in silane or chlorosilanes. The literature contains little information

All valves to have extended operating shafts
valve bodies submerged in R-12 refrigerant bath

Figure 17 VAPOR LIQUID EQUILIBRIUM (VLE) CELL

on the sensitivity of the detector to boron compounds. The interferometric infrared technique, at this time, is still considered to be the best and least troublesome. An evaluation of several columns for the gas chromatographic separation of diborane and silane, at sub-ambient conditions, is being undertaken.

1.6.4.3 Silane Analysis - Epitaxy

Long-lead items, flow controllers, and a temperature controller have been ordered for the 2-wafer epitaxy reactor designed for quality control analysis of silane. Additional detailed design of the reactor components is in progress. Design ideology dictates identical items will be used, as in the slim-rod reactor, to reduce maintaining duplicate spare parts at EPSDU.

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SECTION III CONCLUSIONS

The significant highlights and conclusions are as follows:

- The current list of process equipment is updated regularly for the purposes of cost effectiveness, proper vendor selection, and realistic layout.
- Equipment specifications, vendor selection, and quotation evaluation represent a significant effort which is progressing well, to permit start of EPSDU equipment ordering during the first quarter, 1980.
- Periodic review of preliminary and intermediate engineering designs (including: facility and equipment layout drawings; process and instrumentation (P&I) diagrams; electrical diagrams; and equipment specifications) by both R&D and Engineering Team personnel is assuring consistent application of the process technology developed under the Phase I, Phase II, and on-going Phase III R&D programs.
- Initial mini-burner PDU tests show that the unit will operate to provide the necessary waste treatment data for EPSDU design. A thorough design review is underway to achieve a cost-effective and operable design.
- The free-space reactor PDU was assembled and the checkout was initiated.
- Modeling of the free-space reactor has shown that a two-dimensional model capable of portraying recirculation is required. Such a model has been developed and test cases are being studied.

- Fixed-bed experiments have identified operating regions in terms of silane feed concentration and bed temperature for heterogeneous deposition of silicon. Etching and washing the silicon particle surface has shown to minimize fiber growth.
- Capacitive heating at high temperature with a favorable bed/wall temperature gradient has been demonstrated. Improvement of particle separation efficiency has been noted as the boot diameter is decreased.
- The analysis of silane for low levels of diborane using chromatography faces the dual problems of separability and detection. Thermal conductivity, hot wire type, and detectors give erratic response; and photoionization types are not sensitive enough. No resolution to these problems has been achieved to date.

SECTION IV. PROJECTED QUARTERLY ACTIVITIES

1.1 EPSDU-DESIGN/PROCUREMENT

1.1.1 Process Design

- Complete waste burner tests.
- Complete waste treatment design.
- Complete silane column functional design.
- Complete vendor equipment specification review.

1.1.2 Facility Design

- Start equipment layout.
- Complete soil analysis.
- Continue permit applications.

1.1.3 Equipment Design

- Start preliminary schematic wiring diagram.
- Complete instrumentation lists.
- Complete instrumentation RFQs.
- Complete final electrical one-line.
- Complete electrical equipment RFRs.
- Complete distillation column support specifications.
- Complete process equipment RFRs.
- Complete equipment cleaning specifications.

1.1.4 Installation Design

- Start site preparation design package.

1.1.5 Cost Estimating

- Issue detailed cost estimate.
- Start cost improvement study.

1.2 EPSDU-EQUIPMENT FABRICATION/DELIVERY

1.2.2 Electrical Power

- Start/complete equipment orders.

1.2.4 Vendor Designed Process Equipment

- Start/complete compressor, heat exchanger and tank orders.
- Start compressor, heat exchanger and tank drawing approvals.

1.2.5 Auxiliary Equipment

- Start/complete therminol heater, refrigeration unit, cooling tower, instrument air unit and cryo tank orders.
- Start therminol heater, refrigeration unit, cooling tower, instrument air unit and cryo tank drawing approvals.

1.2.8 Procurement Support

- Complete procurement planning.
- Start equipment orders.
- Start vendor print control.

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Development

- Start-up and performance testing are planned.

1.6.2 Melting/Consolidation

- A subcontract will be signed with the winning vendor, who will start melter development work from March 1, 1980.
- Theoretical modeling work in support of the subcontract will be conducted.

1.6.3 Fluid-Bed Development

- Fixed Bed Deposition: Deposition rate experiments will be conducted. The results of all fixed bed experiments will be documented.
- Inert-Gas Fluidization: Capacitive heating tests and particle separation experiments with the 1½-inch diameter boot will be completed. Results will be documented.
- Fluid-Bed Pyrolysis: A preliminary flow sheet and P&I diagram will be prepared.

1.6.4 Quality Control

- Installation of the epitaxy and slim-rod reactors will be initiated in the next quarter. Both installations should be near completion and ready for operation by the end of the next reporting period.

1.7 MANAGEMENT AND DELIVERABLES

- The Baseline Performance Measurement System (PMS) package will be issued.
- Monthly Financial and Management Reports based on the PMS will be issued, starting with the January activity.
- A report, documenting further work recommended for the free-space reactor R&D, will be issued.
- A report will be prepared for presentation at the 15th PIM.