N83 10519

# Technology Sessions

## FLAT-PLATE COLLECTOR RESEARCH AREA

### Silicon Material Task

R. Lutwack, Chairman

Reports of progress in research on processes for making silicon (Si) and in supporting studies were presented by three contractors and JPL.

Union Carbide Corp. reviewed its research on silane decomposition in a fluidized-bed reactor (FBR) process development unit (PDU) to make semiconductor-grade Si. The PDU, reactivated in late 1981 after having been shut down in May 1981 because of funding recisions, was modified by installation of a new heating system to provide the required temperature profile and better control, and testing was resumed. In one test, at 6.3% silane concentration, 100% conversion to Si was achieved.

Solarelectronics, Inc., reported on its investigation of a process for making trichlorosilane by the hydrochlorination of metallurgical-grade Si and silicon tetrachloride. Fabrication and installation of the test system employing a new 2-in.-dia reactor was completed, and tests were conducted to compare reactor performance with that of the earlier 1-in.-dia reactor. Good agreement was obtained. A corrosion test was also carried out on various candidate materials of construction for the reactor. All samples tested showed a weight gain, attributed to formation of metal silicide films that prevent further corrosion.

Hemlock Semiconductor Corp. described progress in the program to develop a process that converts trichlorosilane to dichlorosilane (DCS), which is reduced by hydrogen to make Si by a chemical vapor deposition step in a Siemens-type reactor. Testing of the DCS PDU integrated with Si deposition reactors continued, and semiconductor-grade Si is being made. It was found that hydrogen chloride can be used after a deposition run to remove selectively the Si deposited on the inside surfaces of the reactor bell jar, thereby preventing deposit build-up and bell-jar breakage.

In the JPL in-house program on conversion of silane to Si in an FBR, experiments in a 2-in.-dia reactor to define the operating window and to investigate the Si deposition kinetics were completed. Even with silane concentration as high as 65% in hydrogen, excessive formation of Si fines as well as bed agglomeration can be prevented by proper choice of operating conditions.

ORIGINAL PAGE IS
OF POOR QUALITY

# SILANE-TO-SILICON PROCESS

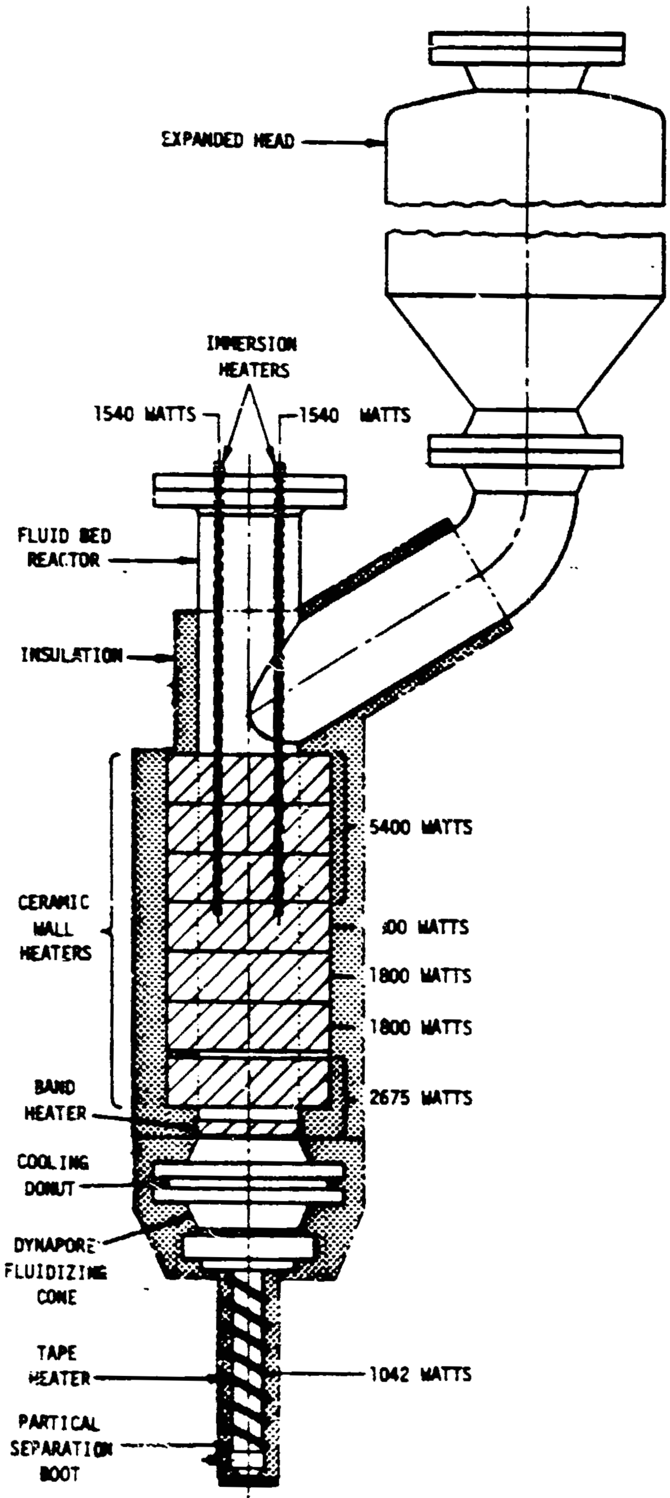
UNION CARBIDE CORP.

| TECHNOLOGY<br>POLYCRYSTALLINE SILICON R&D | REPORT DATE<br>APRIL 22, 1982 |
|---|---|
| APPROACH<br>SILANE DECOMPOSITION IN A FLUID BED REACTOR TO MAKE SEMICONDUCTOR-GRADE POLYSILICON<br><br>CONTRACTOR<br>UNION CARBIDE CORPORATION | STATUS<br>• FLUID BED REACTOR PDU WAS MODIFIED WITH A NEW HEATING SYSTEM<br>• PDU IS OPERATIONAL & EXPERIMENTS ARE IN PROGESS |
| GOALS<br>• INVESTIGATE PROCESS FEASIBILITY<br>• DETERMINE OPERATING WINDOW<br>• DEMONSTRATE STEADY STATE OPERATION<br>• DEMONSTRATE SILICON PURITY FOR PV APPLICATION | |

## Fluid-Bed Silane Decomposition R&D Summary

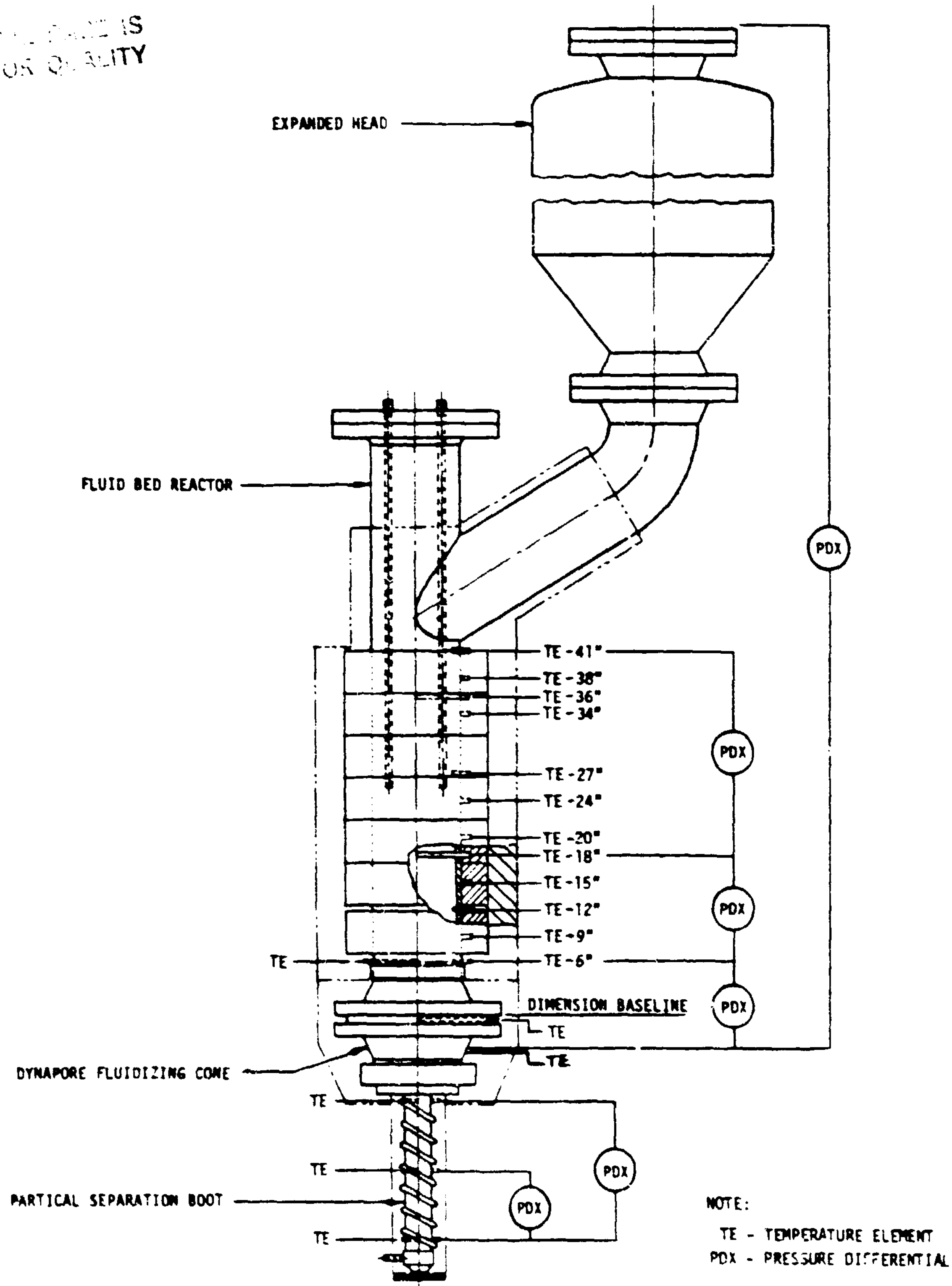
- 6-INCH DIAMETER FLUID BED PDU WAS ASSEMBLED & STARTED UP IN EARLY 1981 UNDER PREVIOUS CONTACT-PHASE
- R & D WORK WAS TEMPORARILY SUSPENDED & REACTIVATED IN $4^{th}$ Q 1981
- PDU MODIFICATIONS INVOLVING INSTALLATION OF A NEW HEATING SYSTEM WERE COMPLETED
- FLUIDIZATION & BED HEATING TESTS IN HYDROGEN ATMOSPHERE WERE CONDUCTED
- PDU WAS RESTARTED WITH SILANE & 3 EXPERIMENTAL RUNS HAVE BEEN CONDUCTED

## Fluid-Bed Reactor Heating System



## FBR Temperature & Pressure Tap Locations

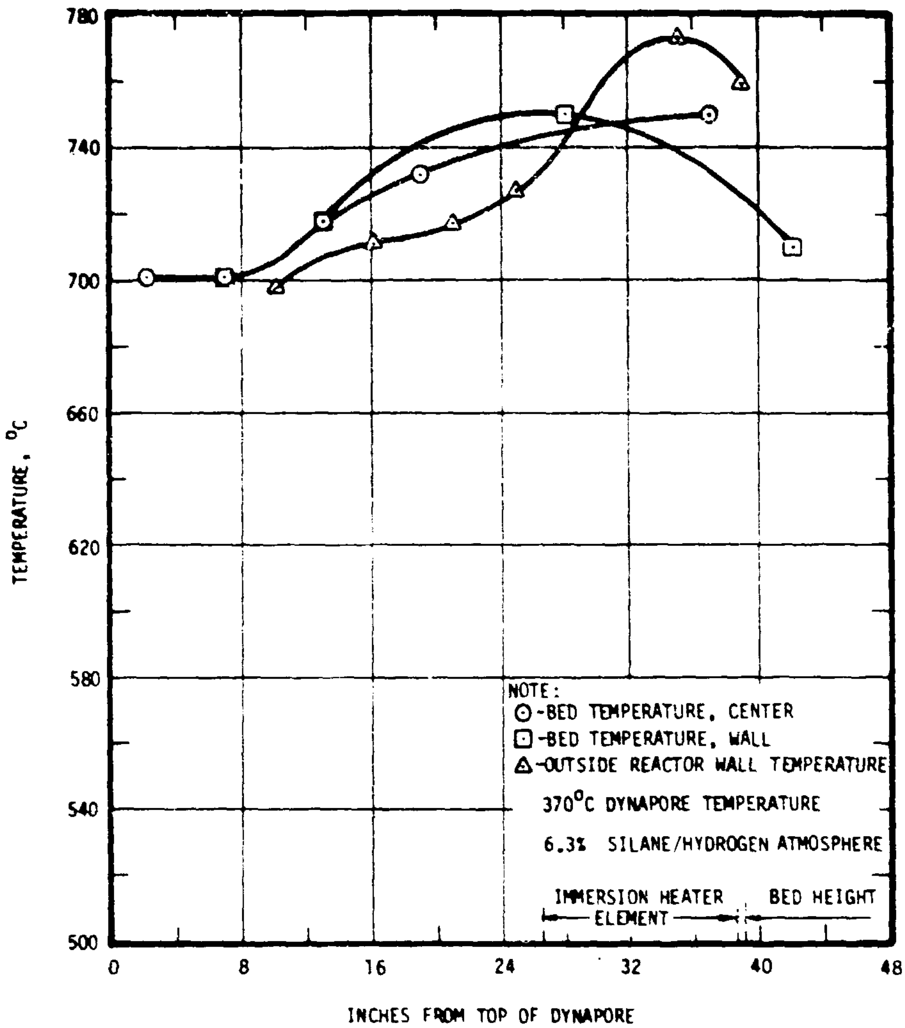




# FBR Run Summary

| RUN NO. | SILANE FEED DURATION HRS. | MAXIMUM SILANE IN FEED, % | SILANE CONVERSION, % | BED TEMP., °C | DISTRIBUTOR TEMP., °C | $U/U_{MF}$ | COMMENTS |
|---|---|---|---|---|---|---|---|
| 1 | 4.5 | 6.3 | 100 | 700-730 | 340-375 | 5.5 | • COMPLETE CONVERSION<br>• GRADUAL INCREASE OF $\Delta P$ ACROSS DISTRIBUTOR |
| 2 | 2.0 | 18.6 | 99.2 | 500-645 | 300-335 | 3.5 | • GOOD CONVERSION WITH HIGH SILANE FEED CONCENTRATION<br>• PARTIAL PLUGGING OF DISTRIBUTOR AT THE END OF RUNS 1 & 2 |
| 3 | 3.0 | 12.0 | <90 | 500-560 | 310-325 | 6 | • INCOMPLETE CONVERSION SINCE BED TEMP. WAS LOW.<br>• DISTRIBUTOR $\Delta P$ CONSTANT<br>• PRODUCT WITHDRAWAL & SEED INJECTION TESTED |

## FBR Test Temperature Profile




## Problems and Concerns

- **GAS DISTRIBUTOR OVER-HEATING & PLUGGING**
- **AGGLOMERATION OF SILICON PARTICLES IN FLUID BED REACTOR**
- **INSUFFICIENT BED HEIGHT IN THE CURRENT PDU**
- **POSSIBLE SILICON CONTAMINATION DUE TO IMPURE FEED/IMPROPER MATERIALS**

## Plans

- FINISH CURRENT EXPERIMENTS TO DETERMINE OPERATING WINDOW.
- CONDUCT LONG RUN TO INVESTIGATE STEADY STATE OPERATION.
- EVALUATE PARTICLE GROWTH RATE & MORPHOLOGY.
- PROVIDE SAMPLES TO JPL FOR ANALYSIS.

# HYDROCHLORINATION PROCESS

SOLARELECTRONICS, INC.

**TECHNOLOGY**

POLYCRYSTALLINE SILICON METAL

**REPORT DATE**

APRIL 22, 1982. 20TH PIM

**APPROACH**

HYDROCHLORINATION OF METALLURGICAL GRADE SILICON TOGETHER WITH SILICON TETRACHLORIDE AND HYDROGEN TO FORM TRICHLOROSILANE FOR PRODUCING SILICON METAL

**CONTRACTOR**

SOLARELECTRONICS, INC.

**GOALS**

TO CARRY OUT A BASIC RESEARCH PROGRAM ON THE HYDROCHLORINATION REACTION OF $SiCl_4$,

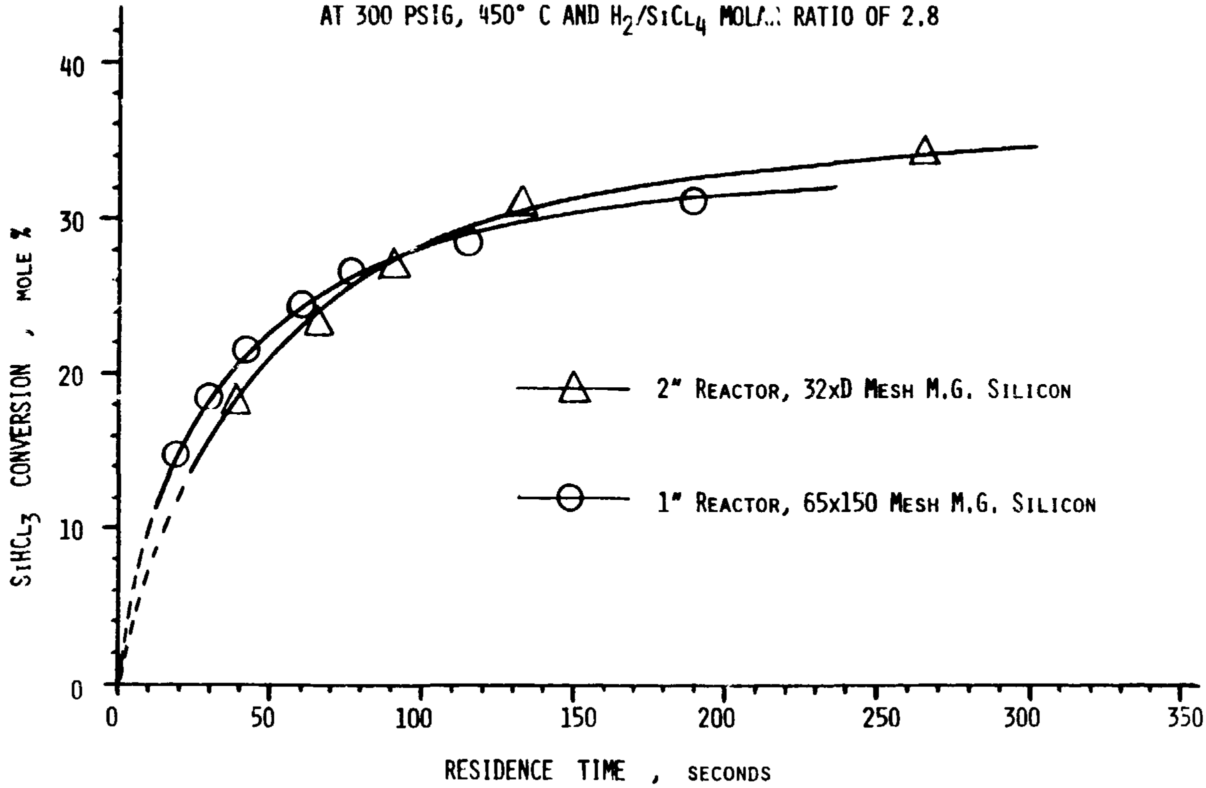
- REACTION KINETICS MEASUREMENTS: AS A FUNCTION OF T, P AND C
- EFFECT OF PRESSURE
- REACTION MECHANISM: STEP-WISE REACTION, INTERMEDIATE AND BY-PRODUCT
- CORROSION MECHANISM OF METALS AND ALLOYS IN THE HYDROCHLORINATION REACTION ENVIROMENT.

**STATUS** JPL CONTRACT NO. 956061 (JULY 9, 1981 - JULY 8, 1982.)

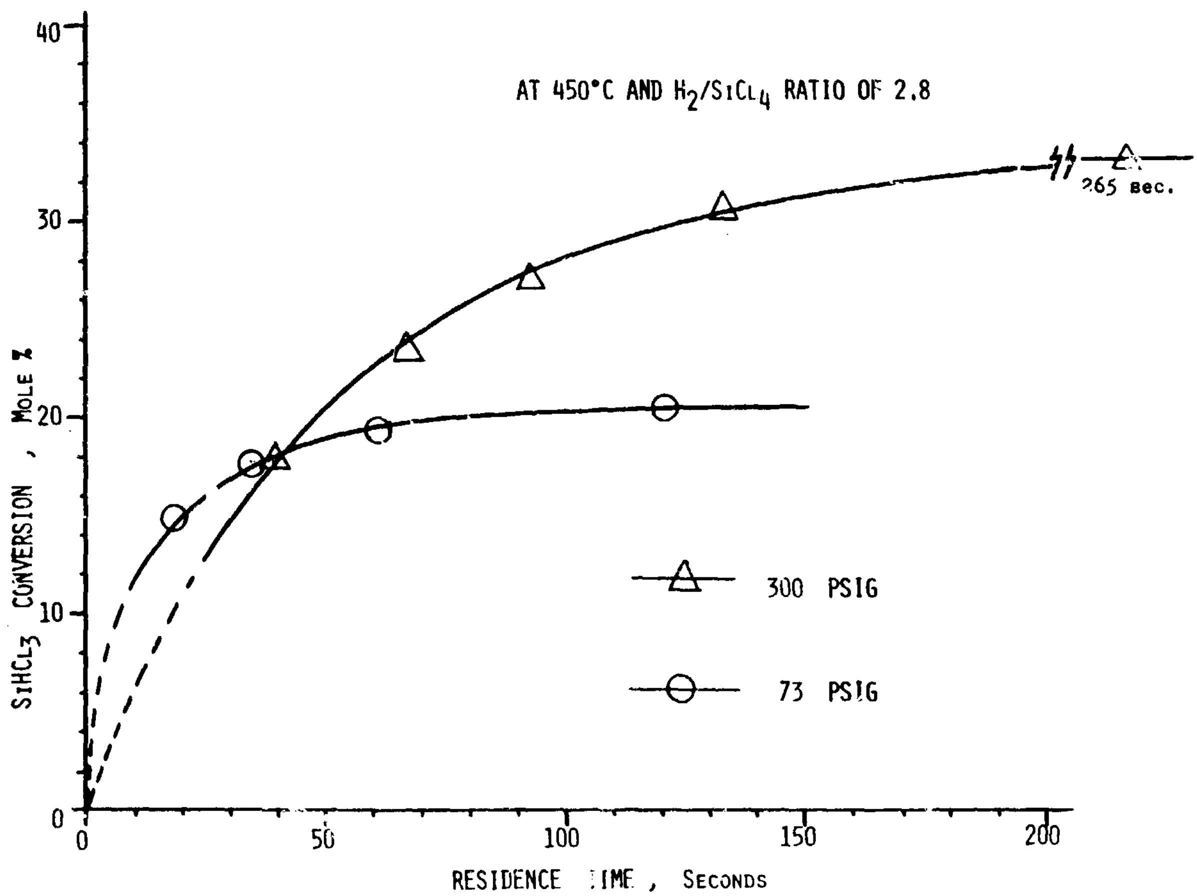
$$3\ SiCl_4 + 2\ H_2 + Si = 4\ SiHCl_3$$

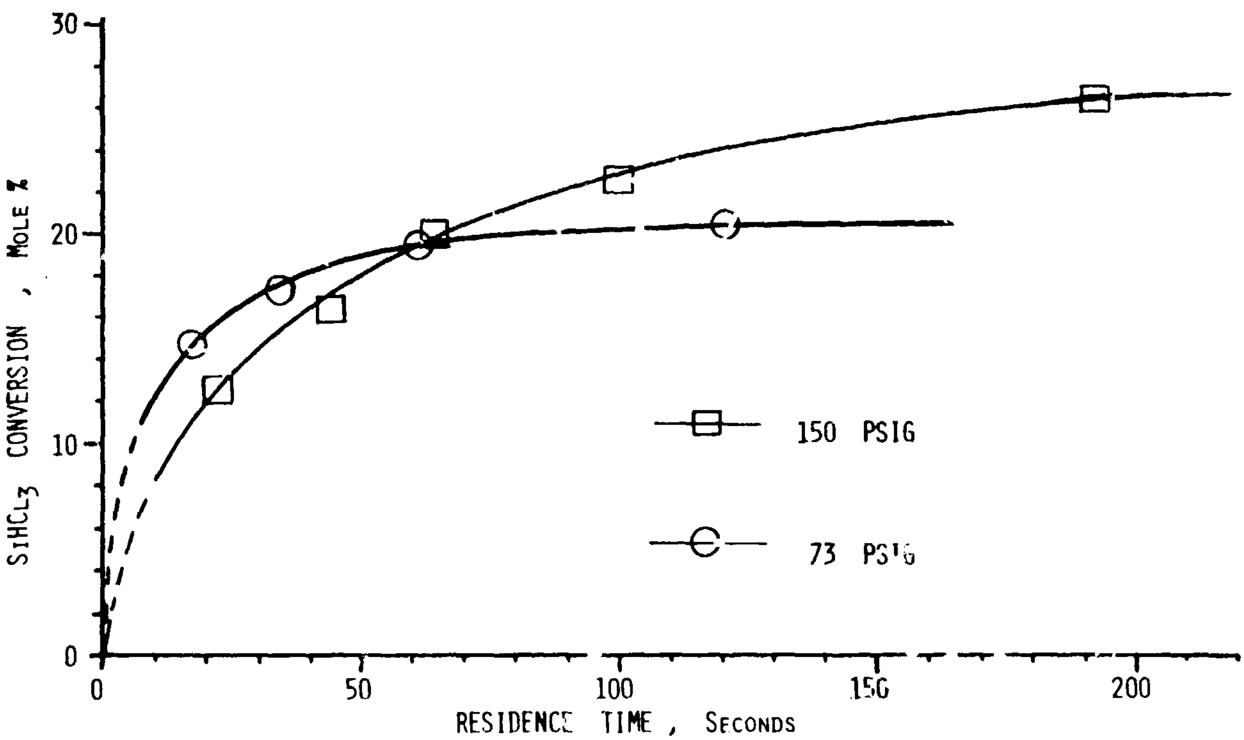
- NEW TWO INCH REACTOR OPERATIVE; RESULTS CHECKED OUT WITH PREVIOUS EXPERIMENTS
- EFFECT OF PRESSURE: HIGHER PRESSURE GIVES A HIGHER $SiHCl_3$ CONVERSION BUT AT A SLOWER REACTION RATE
- HCL ANALYSIS: 0.1 - 0.5% HCL PRESENT
- CORROSION TESTS: CARBON STEEL, NICKEL, COPPER, STAINLESS STEEL, INCOLOY 800H, HASTELLOY B-2
- CORROSION MECHANISM STUDY: THE NATURE OF THE SILICIDE PROTECTIVE FILM, ELEMENTAL ANALYSIS, SEM ANALYSIS
- MILESTONE CHECK POINT, PROGRAM REVIEW, REVISED PROGRAM PLAN.

## Hydrochlorination of $SiCl_4$ and mgSi to $SiHCl_3$



## Effect of Pressure on Hydrochlorination of $SiCl_4$ and mgSi



## HCl Analysis in the Hydrochlorination of $SiCl_4$

AT 500°C, 300 PSIG AND $H_2/SiCl_4$ RATIO OF 2.0

| SAMPLE NO. | RESIDENCE TIME SECOND | REACTION PRODUCT COMPOSITION, AREA% HCL | $SiH_2Cl_2$ | $SiHCl_3$ | $SiCl_4$ |
|---|---|---|---|---|---|
| A | 207 | 0.5970 | 0.7512 | 32.66 | 64.04 |
| B | 207 | 0.5875 | 0.7611 | 31.64 | 66.03 |
| 1 | 96 | 0.1235 | 0.4878 | 26.69 | 71.98 |
| 2 | 96 | 0.1388 | 0.5163 | 26.75 | 71.92 |
| 3 | 138 | 0.3326 | 0.7961 | 31.75 | 66.57 |
| 4 | 138 | 0.4343 | 0.3325 | 31.84 | 66.80 |
| 5 | 207 | 0.5962 | 0.7303 | 31.95 | 65.82 |
| 6 | 207 | 0.5735 | 0.8337 | 31.93 | 66.05 |

## Corrosion Tests on Metals and Alloys

( 87 HOURS @ 500°C, 300 PSIG, $H_2/SiCl_4$= 2.0)

| METALS, ALLOYS | APPROXIMATE COMPOSITION |
|---|---|
| CARBON STEEL | BASICALLY IRON, + 95% FE |
| NICKEL | PURE |
| COPPER | PURE |
| STAINLESS STEEL (TYPE 304) | 68% FE, 19% CR, 10% NI, 2% MN, 1% SI |
| ALLOY 400 (MONEL) | 2/3 NICKEL, 1/3 COPPER |
| INCOLOY 800H | 45% FE, 30% NI, 23% CR, 1% MN, 0.6% SI |
| HASTELLOY B-2 | 68% NI, 28% MO, 2% FE, 1% CR, 1% MN |

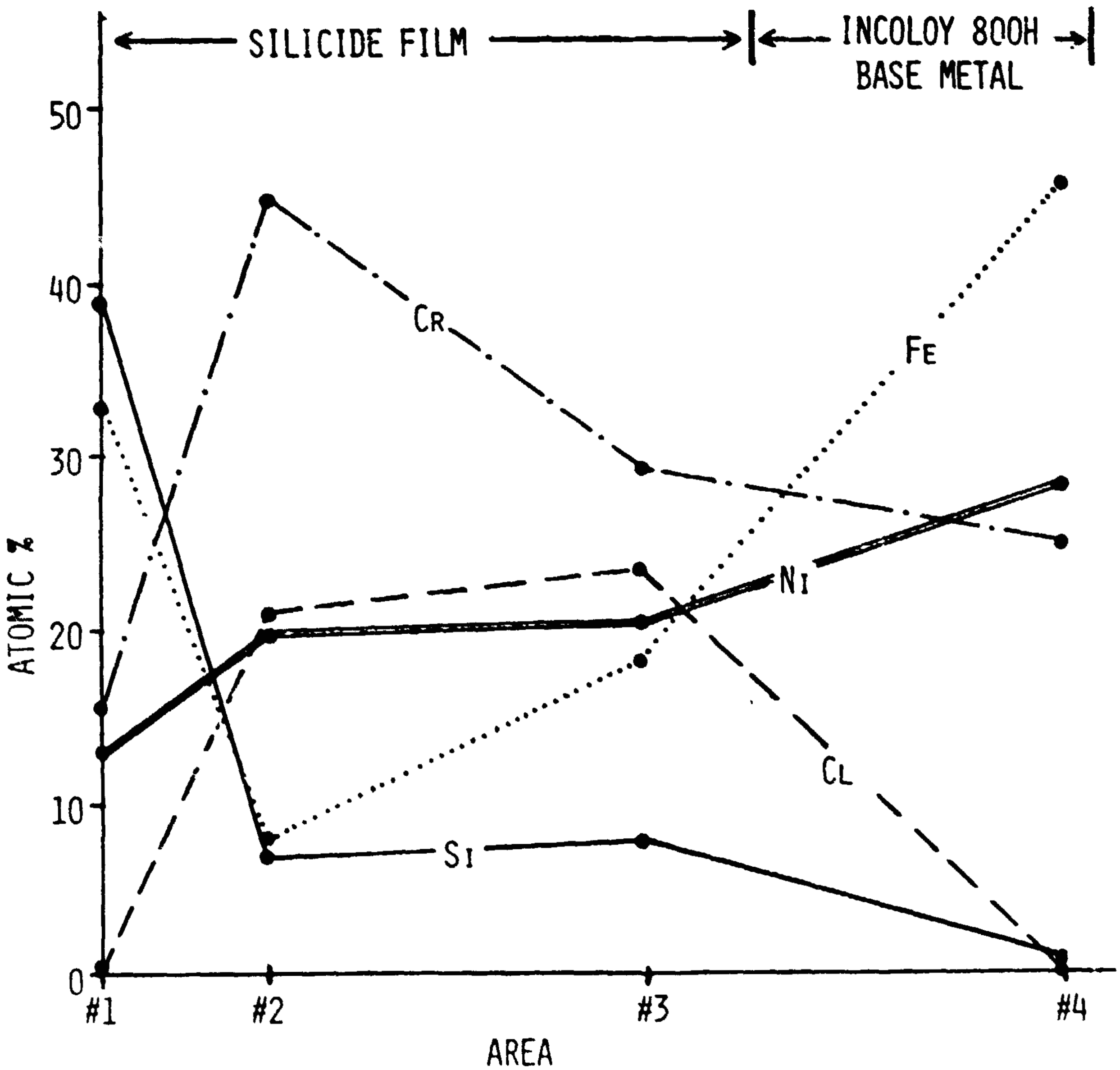
## Corrosion Test on Pure Nickel, 87 Hours

AT 500°C, 300 PSIG AND $H_2/SiCl_4$ OF 2.0

| SAMPLE NO. | DISTANCE* FROM GRID PLATE INCH | TOTAL SURFACE AREA $CM^2$ | WEIGH BEFORE REACTION G. | WEIGH AFTER REACTION G. | WEIGH GAIN M.G. | WEIGH GAIN PER UNIT AREA $M.G./CM^2$ |
|---|---|---|---|---|---|---|
| 1 | 31.3 | 20.4 | 4.8807 | 4.9017 | 21.2 | 1.04 |
| 2 | 28.0 | 20.7 | 4.9450 | 4.9676 | 22.6 | 1.09 |
| 3 | 23.2 | 19.7 | 4.7197 | 4.7576 | 37.9 | 1.92 |
| 4 | 20.0 | 20.8 | 4.9888 | 5.0921 | 103.3 | 4.97 |
| 5 | 16.2 | 20.3 | 4.8710 | 5.0639 | 192.9 | 9.50 |
| 6 | 13.0 | 20.4 | 4.8850 | 5.1481 | 261.3 | 12.8 |
| 7 | 9.5 | 20.8 | 4.9808 | 5.3098 | 329.0 | 15.8 |
| 8 | 6.2 | 20.3 | 4.8638 | 5.2190 | 355.2 | 17.5 |
| 9 | 2.5 | 20.8 | 4.9680 | 5.2502 | 282.2 | 13.6 |

* THE SILICON METAL BED IS ABOUT 18 INCHES HIGH.

NOTE: UNLIKE INCOLOY 800H AND STAINLESS STEEL, THE SILICIDE FILM ON PURE NICKEL IS NOT REACTIVE TOWARD AIR AND MOISTURE




Corrosion Test on Incoloy 800H: 238 h at 500°C, 300 psig, $H_2/SiCl_4 = 2$
SEM Analysis of Cross-Sectional Area

# Corrosion Mechanism Study

FORMATION OF THE SILICIDE PROTECTIVE FILM: CHEMICAL REACTIONS

(1) CHEMICAL VAPOR DEPOSITION OF SILICON

$$4\,SiHCl_3 = 3\,SiCl_4 + 2\,H_2 + Si \qquad K_p = \frac{(SiHCl_3)^3 (H_2)^2 (Si)^0}{(SiCl_4)^4}$$

(2) REACTION WITH HCL

$3\,HCl + Si = H_2 + SiHCl_3$

$6\,HCl + 2\,Fe = 3\,H_2 + 2\,FeCl_3$

$2\,HCl + Ni = H_2 + NiCl_2$

$6\,HCl + 2\,Cr = 3\,H_2 + CrCl_3$

VAPOR PRESSURE OF METAL CHLORIDE

(3) REACTION OF METAL CHLORIDE WITH SILICON

$FeCl_3 + Si = SiCl_4 + FeCl_2$

$FeCl_2 + Si = SiCl_4 + Fe/Si$

$NiCl_2 + Si = SiCl_4 + Ni/Si$

$CrCl_3 + Si = SiCl_4 + Cr/Si$

SILICIDE FILM

A STEADY STATE EQUILIBRIUM ?

QUESTION: INCOLOY 800H, 87 HOURS = 1.78 M.G./CM$^2$, 238 HOURS = 2.71 M.G./CM$^2$

FORMATION OF THE SILICIDE PROTECTIVE FILM: PHYSICAL PROCESS

(1) MELTING POINTS OF THE BASE METAL

Cu = 1083°, Mn = 1260°, Si = 1420°, Ni = 1455°, Fe = 1535°, Cr = 1890°, Mo = 2620°

(2) METAL-SILICON PHASES (SILICIDES)

$Cu_3Si$ 558°, 802°

$Ni_3Si_2$ 845°, 964°; $NiSi$ 992°

$Cu_3Ni$ 1185°

$Fe_5Si_3$ 825°, 1030°; $FeSi_2$ 1220°

$Mn_3Si$ 1075°; $MnSi$ 1275°

$CrSi_2$ 1550°; $CrSi$ 1600°

$MoSi_2$ 1870°; $Mo_3Si_2$ 2190°

VERSUS REACTION TEMPERATURE = 500°

Cr, Mo SILICIDES ARE FORMED AT HIGHER TEMPERATURES THAN THOSE OF Cu, Ni. THIS MAY EXPLAIN THE LARGE DIFFERENCES ON THE AMOUNT OF Si DEPOSITED ON THE TEST SAMPLES.

## Corrosion Test Results: Weight Gain by Test Samples

( 87 HOURS AT 500°C, 300 PSIG, $H_2/SiCl_4$ = 2.0 )

| METALS, ALLOYS | TOTAL SURFACE AREA $CM^2$ | WEIGH BEFORE REACTION G. | WEIGH AFTER REACTION G. | WEIGH GAIN M.G. | WEIGH GAIN PER UNIT AREA $M.G./CM^2$ |
|---|---|---|---|---|---|
| CARBON STEEL | 15.6 | 3.6100 | 3.7435 | 133.5 | 8.56 |
| PURE NICKEL | 20.8 | 4.9808 | 5.3098 | 329.0 | 15.8 |
| PURE COPPER | 23.1 | 8.2623 | 8.5986 | 336.3 | 14.6 |
| ALLOY 400 (MONEL) | 31.2 | 21.3429 | 21.4448 | 101.9 | 3.27 |
| S.S. (TYPE 304) | 20.0 | 12.2397 | 12.2972 | 57.5 | 2.88 |
| INCOLOY 800H | 28.7 | 13.4049 | 13.4561 | 51.2 | 1.78 |
| HASTELLOY B-2 | 32.2 | 23.1987 | 23.2417 | 43.0 | 1.34 |

QUESTION: WILL THE SILICIDE FILM CONTINUE TO GROW ?

## Corrosion Mechanism Study

IS THERE ANY CORROSION ?

(1) THE SILICIDE PROTECTIVE FILM

- ALL TEST SAMPLES SHOW A WEIGH GAIN
- A SILICIDE FILM IS FORMED ON THE SURFACE OF ALL TEST SAMPLES
- NO SIGNIFICANT CORROSION IS EXPECTED WITH A STABLE SILICIDE PROTECTIVE FILM

(2) SCALING: UNLIMITED GROWTH OF THE SILICIDE FILM

- A THICK SILICIDE SCALE CAN WEAKEN THE REACTOR WALL DUE TO THE POOR MECHANICAL PROPERTY OF SILICIDES
- A THICK SILICIDE SCALE CAN BE BROKEN OFF DUE TO MECHANICAL AND THERMAL STRESS - ERROSION BY SCALING

CASE I NICKEL: 110 MICRONS FILM AFTER 87 HOURS, CA. 50% NICKEL

$$\frac{0.5 \times 365 \times 24 \times 110 \times 10^{-4}}{87 \times 2.54 \times 10^{-3}} = 218 \text{ MILS/YEAR}$$

CASE II INCOLOY 800H: 238 HOURS, SILICIDE FILM WITH 8.6 MICRON BASE METAL

$$\frac{365 \times 24 \times 8.6 \times 10^{-4}}{238 \times 2.54 \times 10^{-3}} = 12 \text{ MILS/YEAR}$$

CONCLUSION:

(1) MECHANISM

- CHEMICAL VAPOR DEPOSITION OF Si
- INTERACTION OF Si WITH BASE METALS - CHEMICAL PROCESS
- INTERACTION OF Si WITH BASE METALS - PHYSICAL PROCESS
- DEVELOPMENT OF METAL-SILICON PHASES
- FORMATION OF THE SILICIDE FILM OF COMPLEX COMPOSITION
- ALLOYS WITH HIGH Ni, Cr, Mo CONTENTS DESIRABLE

(2) FURTHER EXPERIMENTAL STUDIES RECOMMENDED

- TIME DEPENDENCY: GROWTH OF THE SILICIDE FILM AS A FUNCTION OF TIME, LIMITED OR UNLIMITED GROWTH,
- GROWTH OF THE SILICIDE FILM AS A FUNCTION OF TEMPERATURE - ACCELERATED TEST AT HIGHER TEMPERATURES, UPPER TEMP. LIMIT,
- COMPOSITIONS OF THE BASE ALLOYS.

## Other Forms of Corrosion to Consider

CORROSION MECHANISM OF METAL ALLOYS IS BY FAR A ELECTRO-CHEMICAL PROCESS IN THE PRESENCE OF A ELECTROLYTE, SUCH AS, WATER. IN THIS OXYGENATED ENVIROMENT (ACID OR BASE), THE ALLOY RELIES ON A STABLE OXIDE FILM FOR PROTECTION. THE STABLE NICKEL AND CHROMIUM OXIDE FILM IS THE BASIS FOR THE CORROSION RESISTENCE OF MANY Ni, Cr BASED ALLOYS. STILL MORE STABLE OXIDE FILMS ARE THOSE OF TITANIUM, ZIRCONIUM AND TANTALUM. THE PROTECTIVE MECHANISM IS DIFFERENT FROM THAT OF THE SILICIDE PROTECTIVE FILM FORMED UNDER THE HYDROCHLORINATION REACTION ENVIROMENT, WHICH DOES NOT APPEAR TO INVOLVE THE PRESENCE OF AN OXIDE FILM.

## Potential Corrosions Other Than the Reaction Environment

(1) MANUFACTURING PROCESS: METALLURGICAL HISTORY OF THE METAL ALLOY

(2) FABRICATION: MECHANICAL AND THERMAL PROCESS (FORMING, WELDING, ETC.) "SENSITIZATION" OF AUSTENITIC STAINLESS STEEL: PRECIPITATION OF CHROMIUM CARBIDE ($Cr_{23}C_6$) AND DEPLETION OF CHROMIUM AT GRAIN BOUNDARY BY HEATING.

(3) TESTING, STORAGE, TRANSPORTATION: CONTAMINATION

(4) ATMOSPHERIC ENVIROMENT: PLANT ENVIROMENT IS CORROSIVE (HCl, CHLORIDE PRESENT) "CHLORIDE STRESS CORROSION CRACK" OF STAINLESS STEEL DUE TO INTERGRANULAR ATTACK BY CHLORIDE ACCELERATED BY INTERNAL STRESS (316L OR HIGH Ni)

(5) HIGH TEMPERATURE ENVIROMENT: OXIDATION, CHLORIDE, SULFUR (GAS-FIRED)

(6) SERVICE, REPAIR: SILICIDE PROTECTIVE FILM IS REACTIVE TOWARD MOISTURE.

## Optimum Material of Construction for the Reactor

(1) CARBON STEEL: POOR
- POTENTIAL PROBLEMS: HYDROGEN EMBRITTLEMENT, SCALING
- LOW CORROSION RESISTENCE IN GENERAL
- LOWER COST

(2) STAINLESS STEEL: GOOD
- SATISFACTORY UNDER THE HYDROCHLORINATION REACTION ENVIROMENT
- GOOD CORROSION RESISTENCE IN GENERAL, CHLORIDE STRESS CORROSION CRACK
- MEDIUM COST

(3) HIGH NICKEL, CHROMIUM, MOLYBDENUM ALLOYS: BETTER
- GOOD CORROSION RESISTENCE ALL-ROUND
- HIGH CREEP RESISTENCE (STRENGTH AT HIGH TEMPERATURES)
- HIGHER COST

| ALLOYS | APPROXIMATE COMPOSITION | | | | | | | PRICE | PRICE RATIO CORRECTED FOR |
|---|---|---|---|---|---|---|---|---|---|
| | Ni | Cr | Mo | Fe | Co | Mn | Si | $/LB. | DESIGN STRENGTH |
| S.ST.(TYPE 316L) | 12 | 17 | 2.5 | 66 | - | 1.5 | 0.5 | 2.09 | 1.00 |
| INCOLOY 800H | 31 | 22 | - | 45 | - | 1.0 | 0.6 | - | - |
| HASTELLOY G | 20 | 22 | 6 | 36 | 2.5 | 1.5 | 0.5 | 7.86 | 2.65 |
| HAYNES ALLOY 625 | 62 | 22 | 9 | 5 | 1.0 | 0.5 | 0.5 | 11.37 | 3.11 |
| HASTELLOY B-2 | 67 | 1 | 28 | 2 | 1.0 | 1.0 | - | 13.95 | 5.18 |

# DICHLOROSILANE CVD PROCESS

## HEMLOCK SEMICONDUCTOR CORP.

| | |
|---|---|
| **TECHNOLOGY**<br>POLYCRYSTALLINE SILICON | **REPORT DATE**<br>APRIL, 1982 |
| **APPROACH**<br>CHEMICAL VAPOR DEPOSITION OF POLY-SILICON FROM DICHLOROSILANE (DCS)<br><br>**CONTRACTOR**<br>HEMLOCK SEMICONDUCTOR CORPORATION | **STATUS**<br>• 5" Ø REDISTRIBUTION REACTOR EVALUATION COMPLETED<br>• CATALYST LIFE >90% ORIGINAL CAPACITY AFTER 2 MONTHS OPERATION AT CAPACITY<br>• QUARTZ TUBE DEPOSITION REACTOR CONSTRUCTION COMPLETED<br>• HCL ETCH LINES INSTALLED TO INTERMEDIATE REACTOR<br>• MODEL 11D REACTOR STARTED UP AND EVALUATION IN PROGRESS<br>• SILICON PURITY FROM REDISTRIBUTED TCS IS SEMICONDUCTOR GRADE QUALITY |
| **GOALS**<br>• ESTABLISH PROCESS FEASIBILITY THROUGH LABORATORY EXPERIMENTS AND COMPONENT TESTING<br>• INVESTIGATE CRITICAL ELEMENTS OF PROCESS VIA OPERATION OF PROCESS DEVELOPMENT UNIT<br>• POLYSILICON PRICE OF LESS THAN \$21/KG (1980 \$, 1000-MT/YR, 20% ROI) AND PURITY APPROACHING OR EQUALLING SEMICONDUCTOR-GRADE POLYSILICON | |

## Schedule of Effort by Phases

Oct. 1979 June 25, 1981

| Project / Time (Mo.) | 3 | 6 | 9 | 12 | 15 | 18 | 21 | 24 | 27 | 30 | 33 | 36 | 39 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Phase 1**<br>Feasibility/EPSDU<br>Preliminary Design | | | | | | | | | | | | | |
| **Phase 2**<br>Redistribution Rx and Decomposition Rx PDU Evaluation<br>EPSDU Design (Deleted) | | | | | | | | | | | | | |
| **Phase 3 (Curtailed)**<br>EPSDU Detailed Design and Construction | | | | | | | | | | | | | |

## Activities

- PDU START UP
- PDU CLEAN UP
- PDU START UP AND OPERATION
- EVALUATION OF 5" DIAMETER REDISTRIBUTION REACTOR
- QUARTZ TUBE DEPOSITION REACTOR CONSTRUCTED
- INTERMEDIATE REACTOR OPERATION USING DICHLOROSILANE FEED STOCK WITH POST HCL ETCH
- POLYCRYSTALLINE SILICON PURITY EVALUATION (BORON, DONOR, CARBON)
- START-UP OF MODEL 11D DICHLOROSILANE DECOMPOSITION REACTOR
- SAMPLES OF DCS POLYSILICON SENT TO JPL AND WESTINGHOUSE FOR EVALUATION

## December Shutdown

Scheduled shutdown
before Christmas

Flush system with TCS

Purge out with Nitrogen

Pressure with Nitrogen

## Startup Problems

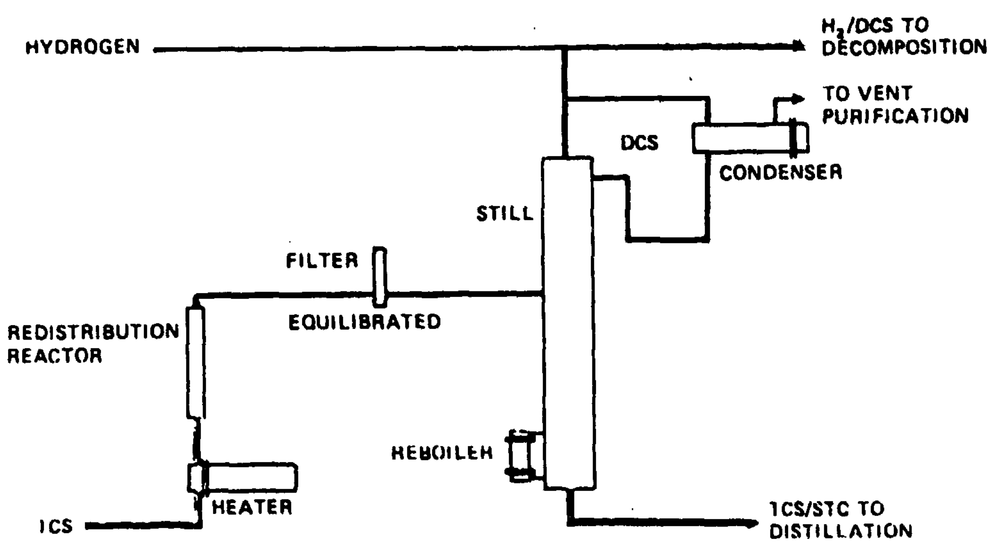
PROBLEM: Filter plugging

CAUSE: Catalyst support screen separated from plate

ACTION: Redesign support plates
Dual filter system

** MAJOR CLEAN UP EFFORT **

## DCS PDU Flow Diagram



## PDU Cleanup

PDU pressure checked &
purged out with nitrogen

Redistribution reactor repacked
with DOWEX

Steam tracing turned on cclumn &
purged out with nitrogen

Moisture check showed no sign of water

## PDU Startup Plan

Load in new DOWEX catalyst
3/9

Purge with hot nitrogen
3/9 to 3/10

Start-up
3/11

Safety review for Model 11 reactor
& updated SOP for PDU complete

## Si Production by DCS Decomposition

A. DICHLOROSILANE PRODUCTION

(CATALYZED REDISTRIBUTION OF TRICHLOROSILANE)

$$2\ SiHCl_3 \rightleftharpoons SiH_2Cl_2 + SiCl_4$$

B. SILICON PRODUCTION

(DICHLOROSILANE DECOMPOSITION)

$$SiH_2Cl_2 + H_2 \longrightarrow Si + H_2,\ HCl,\ SiH_2Cl_2,\ SiHCl_3,\ SiCl_4$$

C. TRICHLOROSILANE PRODUCTION

(HYDROGENATION OF SILICON TETRACHLORIDE)

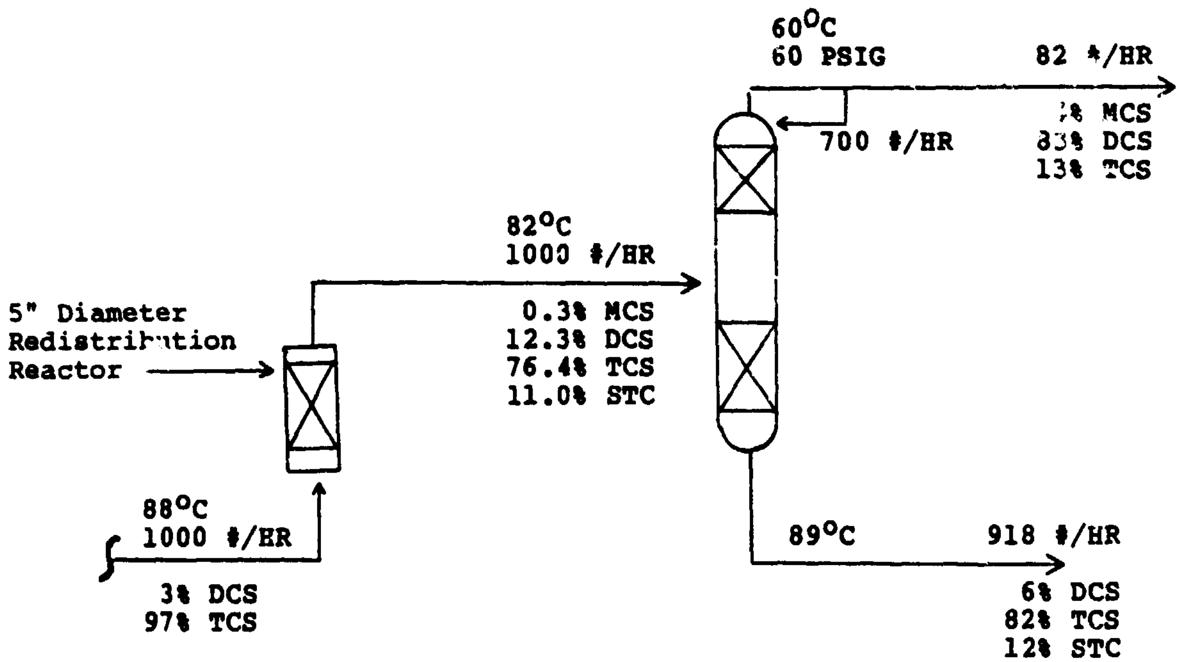
$$SiCl_4 + H_2 + Si_{MG} \rightleftharpoons SiHCl_3 + H_2 + SiCl_4$$

## PDU Objectives

- DCS PRODUCTION 70 LB/HR
- REDISTRIBUTION CONVERSION >10%; DETERMINE TEMPERATURE AND RESIDENCE TIME TO ACHIEVE THIS
- PRESSURE DROP *vs*. VELOCITY IN CATALYST BED
- CATALYST LIFE >90% ORIGINAL CAPACITY AFTER 2 MONTHS OPERATION AT CAPACITY
- DETERMINE IF CATALYST MIGRATION OCCURS

## 5-in.-Dia Redistribution Reactor

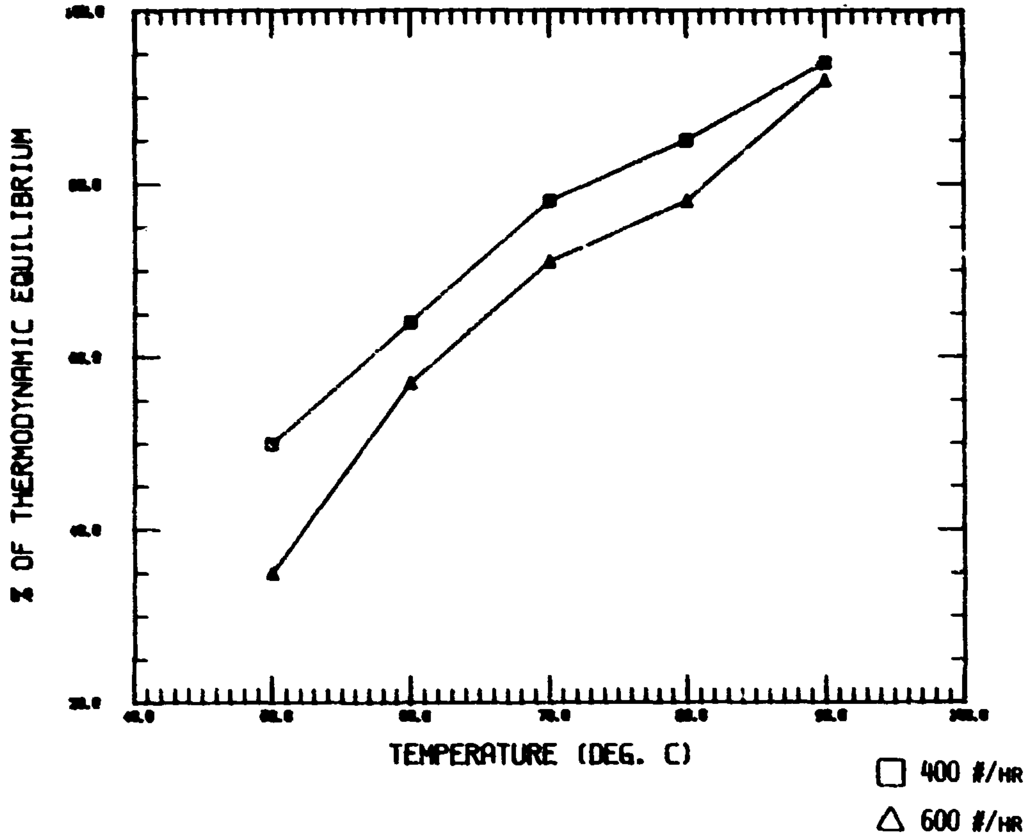
### PDU Conditions at Capacity



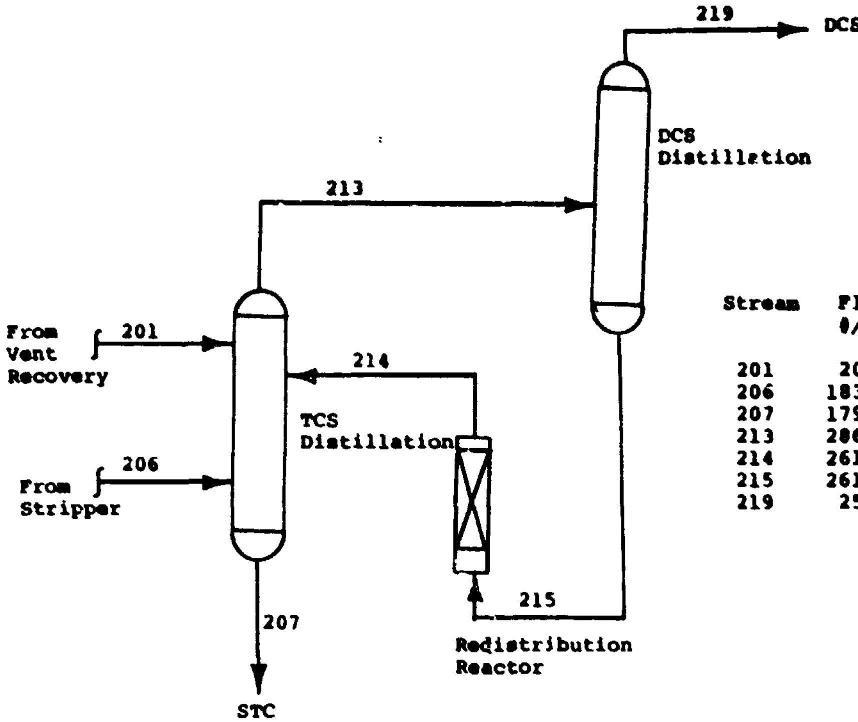
## DCS Production From PDU

| MONTH | # DCS PRODUCED | % ON-LINE TIME | REDISTRIBUTION REACTOR SIZE (O) |
|---|---|---|---|
| JUNE | 11.000 | 96 | 3" |
| JULY | 7,000 | 36 | 3" |
| AUGUST | 10,000 | 45 | 3" |
| SEPTEMBER | 6,200 | 27 | 5" |
| OCTOBER | 18,400 | 95 | 5" |
| NOVEMBER | 14,350 | 70 | 5" |
| DECEMBER | 16,000 | 66 | 5" |
| JANUARY | 0 | 0 | 5" |
| FEBRUARY | 0 | 0 | 5" |
| MARCH | 7,840 | 46 | 5" |

## Kinetic Evaluation for 5-in. Redistribution Reactor



# Redistribution Reactor at Bottom of DCS Column I




| Stream | Flow #/Hr. | Weight Percent MCS | DCS | TCS | STC |
|---|---|---|---|---|---|
| 201 | 2070 | | 12.1 | 55.2 | 32.7 |
| 206 | 18350 | | .6 | 25.1 | 74.3 |
| 207 | 17907 | | | | 100.0 |
| 213 | 28656 | | 8.7 | 91.3 | |
| 214 | 26153 | .2 | 7.5 | 78.5 | 13.8 |
| 215 | 26153 | | | 100.0 | |
| 219 | 2508 | 2.8 | 91.5 | 5.7 | |

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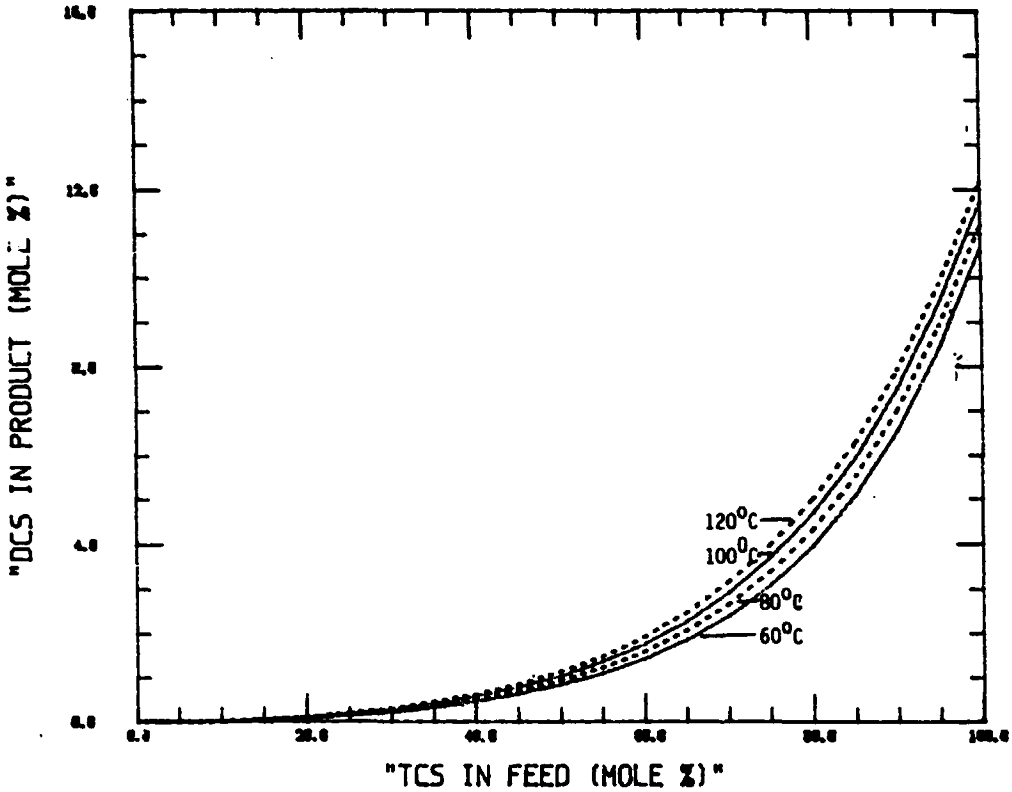
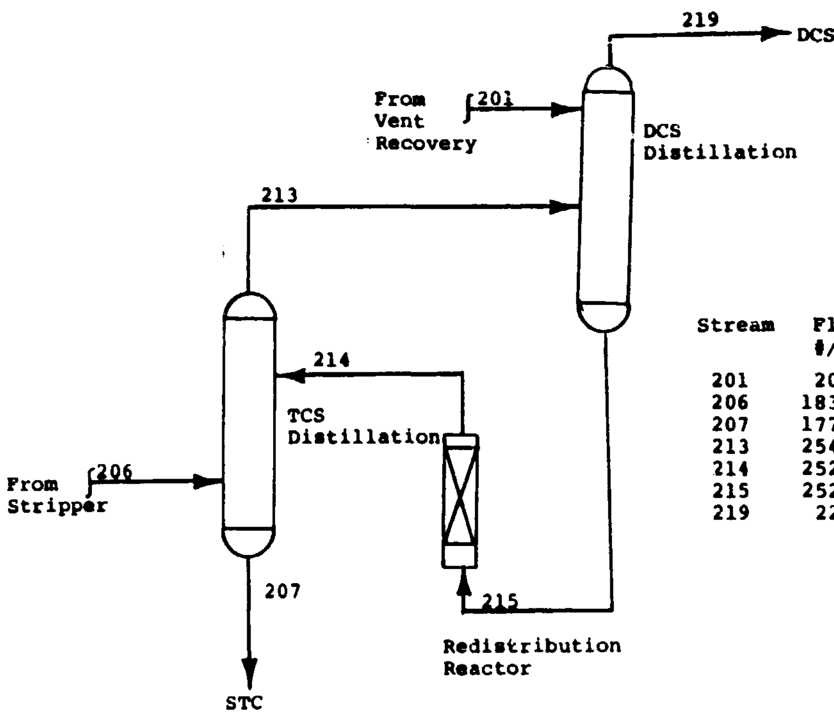
OF POOR QUALITY

## Thermodynamic Redistribution Data: TCS-STC Mixed-Feed System

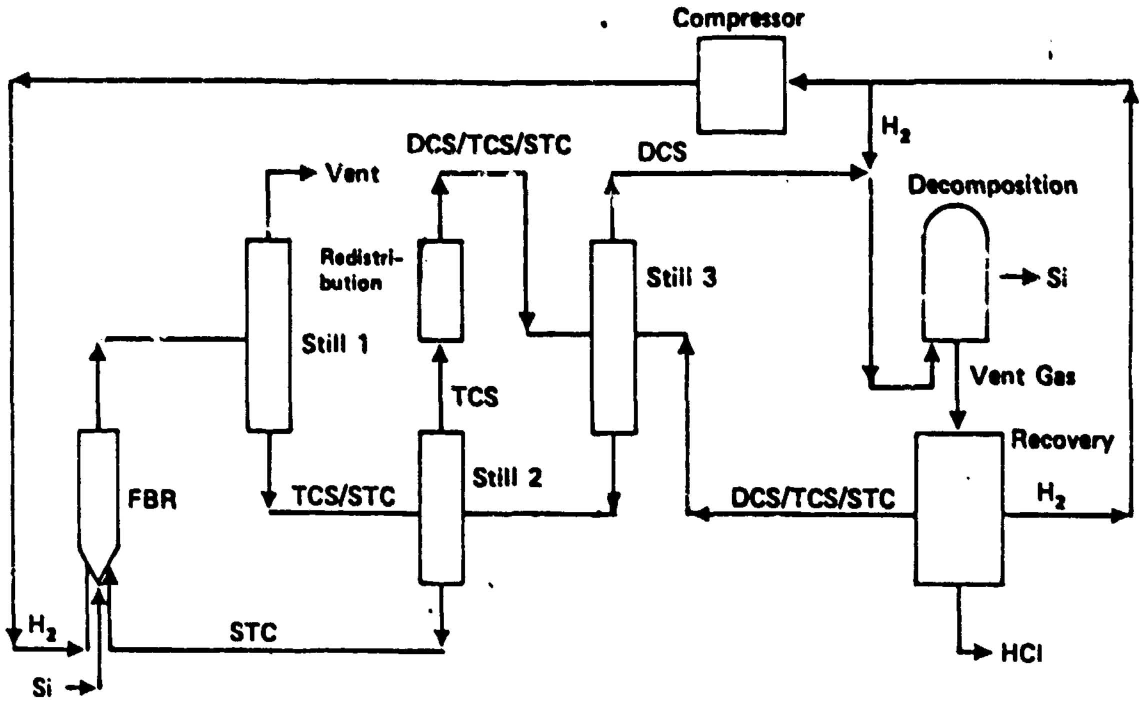



## Redistribution Reactor at Bottom of DCS Column II




| Stream | Flow #/Hr. | Weight Percent MCS | DCS | TCS | STC |
|---|---|---|---|---|---|
| 201 | 2070 | | 12.1 | 55.2 | 32.7 |
| 206 | 18350 | | .6 | 25.1 | 74.3 |
| 207 | 17713 | | | | 100.0 |
| 213 | 25482 | | 8.0 | 92.0 | |
| 214 | 25263 | | 8.0 | 76.0 | 16.0 |
| 215 | 25263 | | | 97.3 | 2.7 |
| 219 | 2271 | | 100.0 | | |

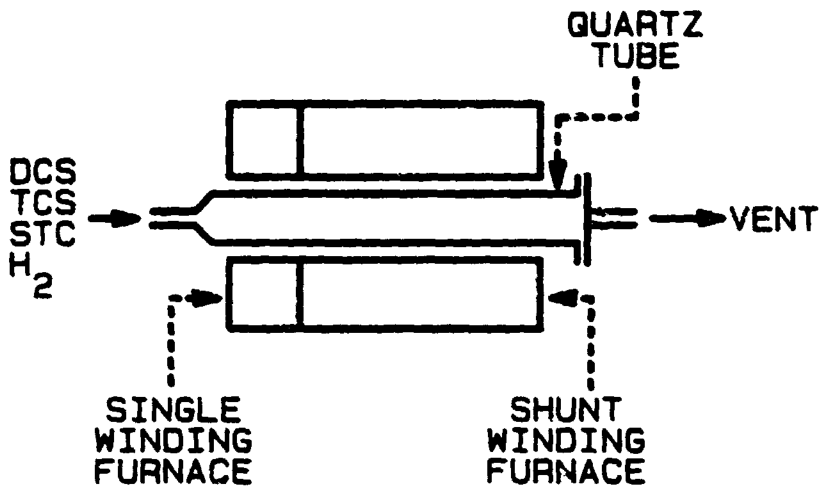
## Low-Cost Silicon Process




## Decomposition Goals

- DEPOSITION RATE 2.0 G/H/CM
- CONVERSION EFFICIENCY 40%+
- POWER CONSUMPTION <60 KWH/KG
- RUN TIME 100H+

## Quartz Tube Deposition Reactor Unit




## DCS Process Data: Intermediate Reactor

| Run No. | Feed Type | Run Time (hours) | Rod Diameter (mm) | Silicon Fed ($gh^{-1}cm^{-1}$) | Silicon Deposition ($gh^{-1}cm^{-1}$) | Conversion (Mole %) | Power Consumption (kWh/kg) |
|---|---|---|---|---|---|---|---|
| 324-481 | DCS | 40.7 | 48-51 | 2.9 | 1.07 | 36.3 | N.A. |
| 324-482 | *DCS | 75.1 | 60-65 | 2.9 | 0.96 | 32.5 | N.A. |
| 324-483 | *DCS | 67.5 | 63-68 | 2.9 | 1.12 | 38.1 | N.A. |
| 325-514 | *DCS | 78.0 | 84-89 | 3.9 | 1.63 | 41.9 | N.A. |
| 325-515 | DCS | 31.7 | 44-46 | 3.7 | 1.19 | 32.4 | N.A. |

* 5 Hour etch after run completed

## Intermediate Decomposition Reactor Summary

- DICHLOROSILANE REACTOR OPERATION SIMILAR TO TRICHLOROSILANE OPERATION
- NO VAPOR PHASE NUCLEATION IN THE REACTOR
- ROD SURFACE ACCEPTABLE
- PURITY IS SEMICONDUCTOR GRADE QUALITY
- POST HCL ETCH SELECTIVELY REMOVES SILICON FROM THE BELL JAR

QUESTION:

- CAN DECOMPOSITION GOALS BE ATTAINED USING MIXED FEED?
- CAN CONVERSION AND POWER CONSUMPTION GOALS BE ACHIEVED IN A LARGER DECOMPOSITION REACTOR ?

# Purity Data

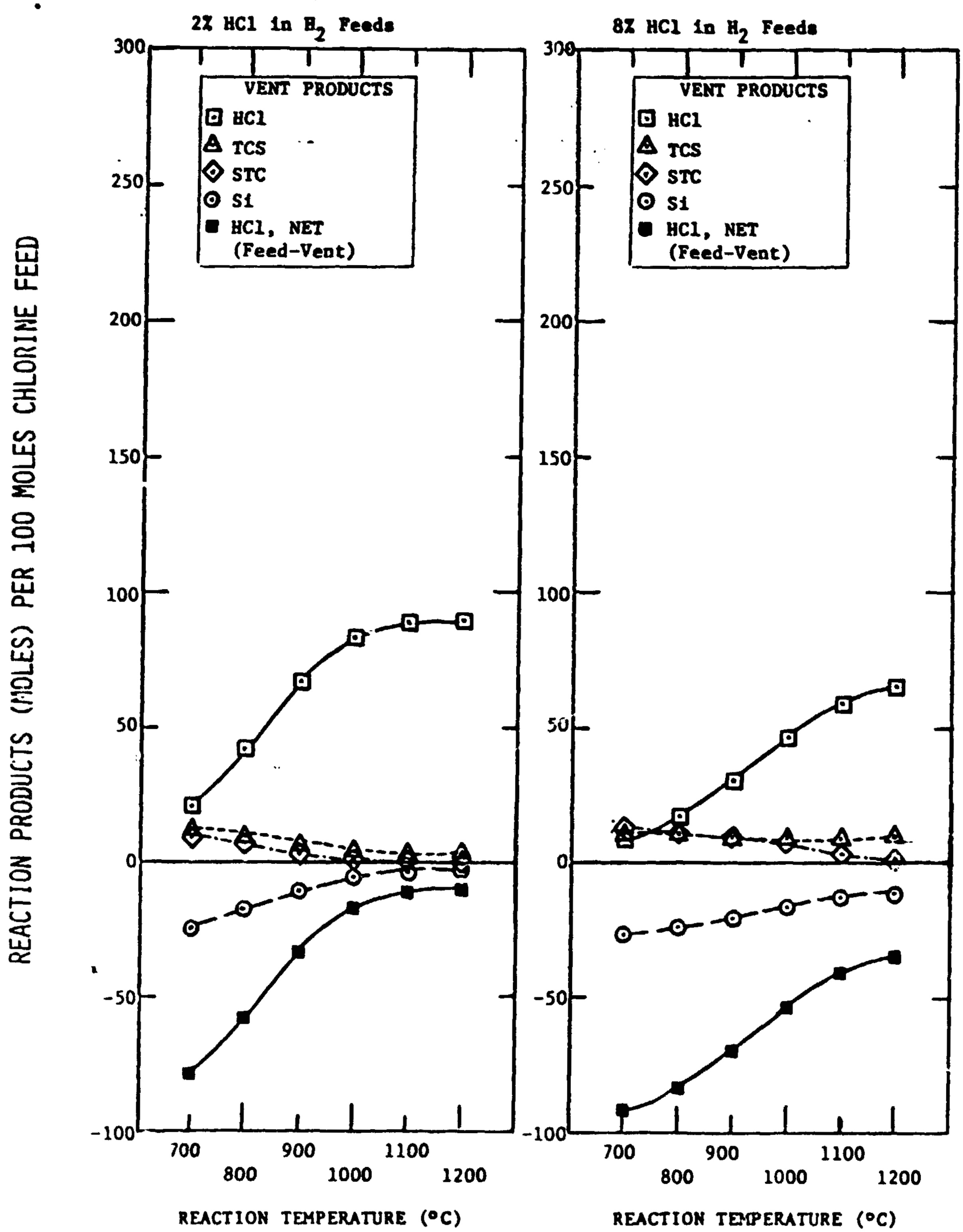
RXR 324 PURITY DATA FOR ELECTRICALLY ACTIVE ELEMENTS
(Boron, Phosphorus, Carbon)

| Run No. | Boron (ppba) | Donor (ppba) | Carbon (ppma) |
|---|---|---|---|
| 324-419 | .08 | 2.88 | 0.88 |
| 324-420 | .08 | 3.94 | 0.4 |
| 324-421 | .08 | 0.38 | |
| 324-422 | .06 | 0.37 | |
| 324-423 | .06 | 1.18 | |
| 324-424 | .06 | 1.19 | |
| 324-425 | .06 | 1.27 | |
| 324-426 | .06 | 0.73 | 0.1 |
| 324-427 | .06 | 0.99 | |
| 324-428 | .05 | 0.68 | 0.5 |
| 324-429 | .07 | 1.24 | |
| 324-430 | .07 | 1.02 | |
| 324-431 | .07 | 0.23 | 0.2 |
| 324-432 | .07 | 0.43 | 0.1 |
| 324-433 | .07 | 0.24 | |
| 324-434 | .04 | 0.4 | 0.2 |
| 324-435 | .04 | 0.29 | 0.3 |
| 324-436 | .30 | 0.85 | |
| 324-437 | .10 | 0.48 | |
| 324-438 | .10 | 1.20 | 0.2 |
| 324-439 | .08 | 0.42 | 0.1 |
| 324-440 | .04 | 0.20 | 0.1 |
| 324-441 | .04 | 0.30 | |
| 324-442 | .06 | 0.20 | 0.3 |
| 324-443 | .06 | 0.34 | 0.1 |
| 324-444 | .04 | 0.44 | 0.3 |
| 324-445 | .06 | 0.62 | |
| 324-446 | .06 | 1.11 | |
| 324-447 | .06 | 10.46 | |
| 324-450 | .05 | 0.21 | |
| 324-451 | .05 | 0.13 | 0.3 |
| 324-452 | .05 | 0.32 | 0.6 |
| 324-453 | .06 | 1.17 | |
| 324-454 | .06 | 0.35 | 0.2 |
| 324-455 | .06 | 5.79 | 0.6 |
| 324-456 | .07 | 0.32 | 0.5 |
| 324-457 | N.E. | N.E. | |
| 324-458 | .06 | 0.17 | 0.5 |
| 324-459 | .06 | 0.28 | 0.5 |
| 324-460 | N.E. | N.E. | |
| 324-461 | .10 | 0.33 | 0.7 |
| 324-462 | .10 | 0.27 | 0.3 |
| 324-463 | .10 | 0.56 | |
| 324-464 | .10 | 0.72 | |
| 324-465 | 8.0 | 7.41 | 0.5 |
| 324-466 | .10 | 0.18 | 0.3 |
| 324-467 | .10 | 0.20 | 0.6 |
| 324-468 | .10 | 0.23 | 0.5 |
| 324-469 | N.E. | N.E. | |
| 324-470 | 1.0 | 1.21 | 0.1 |
| 324-471 | .06 | 0.69 | 0.1 |
| 324-472 | .06 | 0.24 | 0.1 |
| 324-473 | .06 | 0.73 | 0.2 |
| 324-474 | .06 | 0.25 | 0.4 |
| 324-475 | .06 | 0.39 | 0.9 |
| 324-476 | .06 | 1.83 | |
| 324-477 | .06 | 0.87 | 0.1 |
| # Runs-49 | .09 (Avg.) | 0.58 (Avg.) | 0.3 (Avg.) |

RXR 325 PURITY DATA FOR ELECTRICALLY ACTIVE ELEMENTS
(Boron, Phosphorus, Carbon)

| Run No. | Boron (ppba) | Donor (ppba) | Carbon (ppma) |
|---|---|---|---|
| 325-449 | .6 | 30.0 | |
| 325-450 | .09 | 0.25 | 0.7 |
| 325-451 | .06 | 1.30 | |
| 325-452 | .06 | 0.26 | |
| 325-453 | | | |
| 325-454 | .07 | 0.40 | 0.6 |
| 325-455 | .10 | 4.70 | |
| 325-456 | .51 | 0.74 | |
| 325-457 | .19 | 0.37 | 0.1 |
| 325-458 | .19 | 0.63 | 0.2 |
| 325-459 | .08 | 0.49 | |
| 325-460 | .06 | 1.60 | 0.2 |
| 325-461 | .08 | 0.37 | 0.1 |
| 325-462 | .06 | 0.76 | |
| 325-463 | .06 | 0.77 | |
| 325-464 | .06 | 0.86 | |
| 325-465 | .06 | 1.05 | |
| 325-466 | .06 | 10.16 | |
| 325-467 | .06 | 0.56 | 0.3 |
| 325-468 | .05 | 0.26 | 0.6 |
| 325-469 | .06 | 0.38 | 0.2 |
| 325-470 | .06 | 3.79 | 0.1 |
| 325-471 | .04 | 0.48 | 0.2 |
| 325-472 | .06 | 0.53 | 0.2 |
| 325-473 | .06 | 0.77 | 0.4 |
| 325-476 | .04 | 0.24 | 0.4 |
| 325-477 | .04 | 0.22 | 0.1 |
| 325-478 | .04 | 0.30 | |
| 325-479 | .03 | 0.46 | 0.2 |
| 325-480 | .08 | 0.15 | 0.3 |
| 325-481 | .10 | 0.36 | 0.1 |
| 325-482 | .10 | 0.69 | 0.2 |
| 325-483 | .10 | 0.32 | 0.7 |
| 325-484 | .06 | 0.29 | 0.5 |
| 325-485 | .06 | 0.18 | 0.1 |
| 325-486 | .10 | 3.24 | |
| 325-487 | .10 | 0.77 | 0.6 |
| 325-488 | .10 | 0.48 | 0.3 |
| 325-489 | .10 | 0.27 | 0.1 |
| 325-490 | N.E. | N.E. | |
| 325-491 | N.E. | N.E. | |
| 325-492 | .06 | 0.24 | 0.4 |
| 325-493 | .10 | 2.6 | 0.1 |
| 325-494 | .10 | 0.51 | 0.1 |
| 325-495 | .10 | 0.48 | 0.1 |
| 325-496 | N.E. | N.E. | |
| 325-497 | N.E. | N.E. | |
| 325-498 | N.E. | N.E. | |
| 325-499 | 1.0 | 0.07 | 0.1 |
| 325-500 | .10 | 0.29 | 0.3 |
| 325-501 | .06 | 0.43 | 0.1 |
| 325-502 | .06 | 0.28 | 0.8 |
| 325-503 | .06 | 0.39 | 0.1 |
| 325-504 | .06 | 0.51 | 0.1 |
| 325-505 | | | |
| 325-506 | | | |
| 325-507 | .06 | 0.34 | |
| 325-508 | .06 | 0.20 | 0.2 |
| # Runs-44 | .10 (Avg.) | 0.48 (Avg.) | 0.3 (Avg.) |

## Reaction Products vs Reaction Temperature



## DCS Purity Summary

| # OF RUNS | DONOR PPBA | BORON PPBA | CARBON PPMA |
|---|---|---|---|
| **REACTOR 324** | | | |
| 49 | 0.58 | 0.09 | 0.3 |
| 3 | 0.67 | 0.04 | N.A. |
| **REACTOR 325** | | | |
| 44 | 0.48 | 0.10 | 0.3 |
| 2 | 0.38 | 0.03 | N.A. |
| **REACTOR 382** | | | |
| 2 | 1.41 | 0.07 | N.A. |

## Intermediate Decomposition Reactor Results

| PARAMETER | OBJECTIVE | ACHIEVED |
|---|---|---|
| • DEPOSITION RATE ($GH^{-1}CM^{-1}$) | 2.0 | 1.6 - 2.1 |
| • CONVERSION EFFICIENCY (MOLE %) | >40 | 43.6 |
| • POWER CONSUMPTION (KWH/KG) | <60 | 80 - 100 |
| • RUN TIME | 100 | 87 |
| • ROD SURFACE | CZ QUALITY | CZ QUALITY |
| • PURITY | SEMICONDUCTOR QUALITY | SEMICONDUCTOR QUALITY |
| • VAPOR PHASE NUCLEATION | N.A. | NONE OBSERVED |

## Problems and Concerns

- ACHIEVING 40 PERCENT CONVERSION EFFICIENCY
- ACHIEVING A POWER CONSUMPTION AT THE REACTOR OF 60 KWH/KG
- QUARTZ BELL JAR INTEGRITY
- ECONOMICS AND FEASIBILITY OF HYDROGENATION PROCESS

# IN-HOUSE MATERIAL RESEARCH PROGRAM
# Si Deposition in FBR System

JET PROPULSION LABORATORY

G.C. Hsu

## 2-in. FBR Program

VARIABLES

- TEMPERATURE - 650°C, 700°C, 750°C
- SILANE CONC. - 20%, 50%, 65%
  ($SiH_4$, $H_2$)
- $U/U_{mf}$ - 1 TO 6

FIXED PARAMETERS:

- SEED PARTICLE SIZE - 335 μm (AVERAGE)
  (POLY SI - SEMICONDUCTOR GRADE)
- INITIAL BED WEIGHT ~400 gm
  ~6 inch HEIGHT
- DISTRIBUTOR: 200 MESH SCREEN ON S.S. SUPPORT WITH 1/16" HOLES
- DURATION OF RUN: UP TO 1 hour (16 RUNS)

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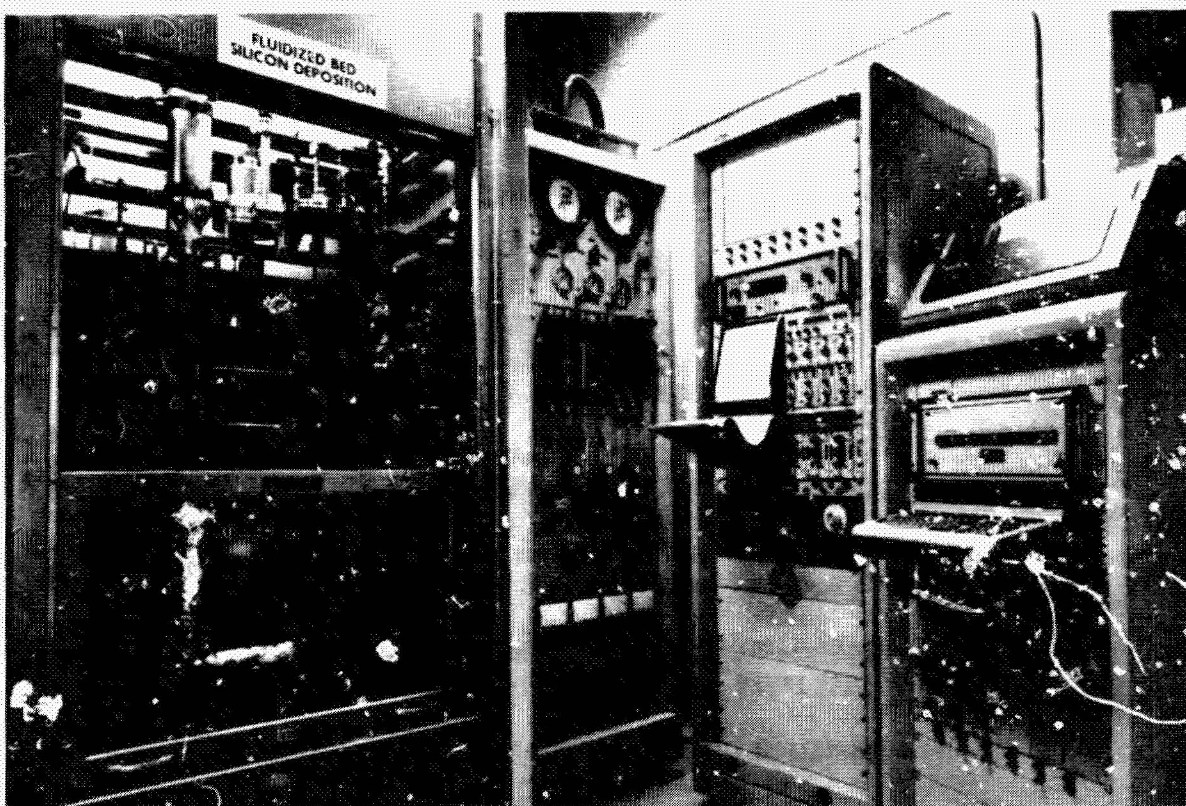
BLACK AND WHITE PHOTOGRAPH



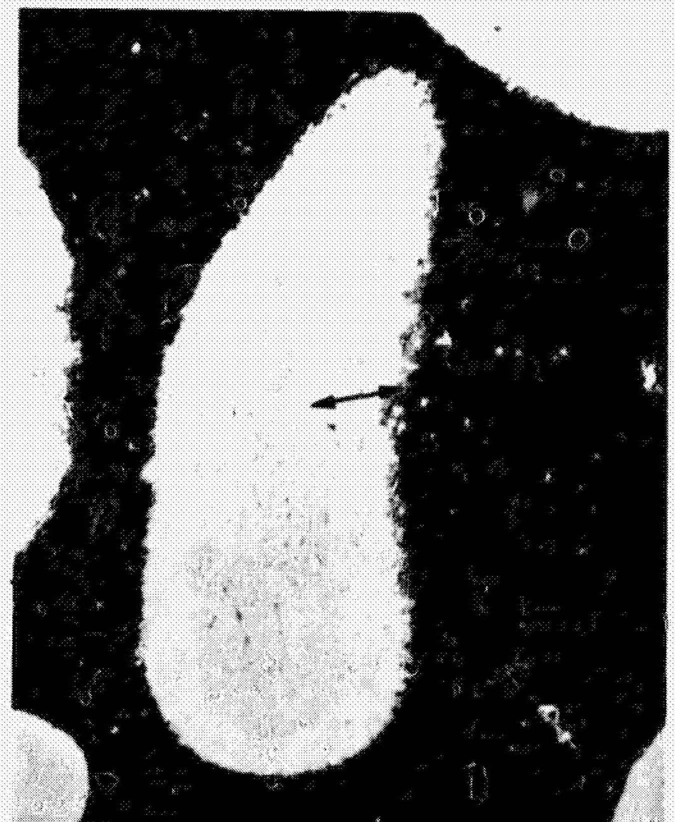
## FBR Si Deposition

**SEED**

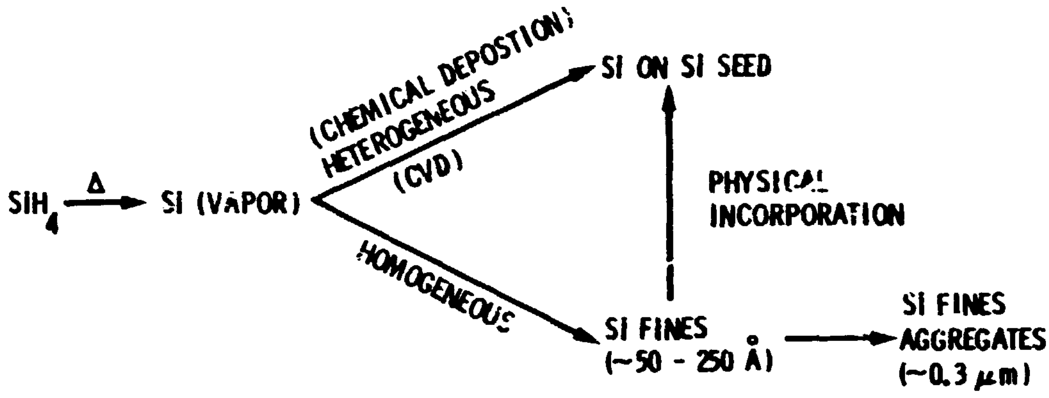
POLY Si PARTICLE (~335$\mu$m)

**PRODUCT**

CONDITONS: 2" FBR
650°C, 50% $SiH_4/H_2$, $U/U_{mf}$ = 6
DEPOSITION RATE: >1$\mu$m/min
DENSE FREE-FLOWING PARTICLES



## Growth Mechanism




## Color of Si Fines

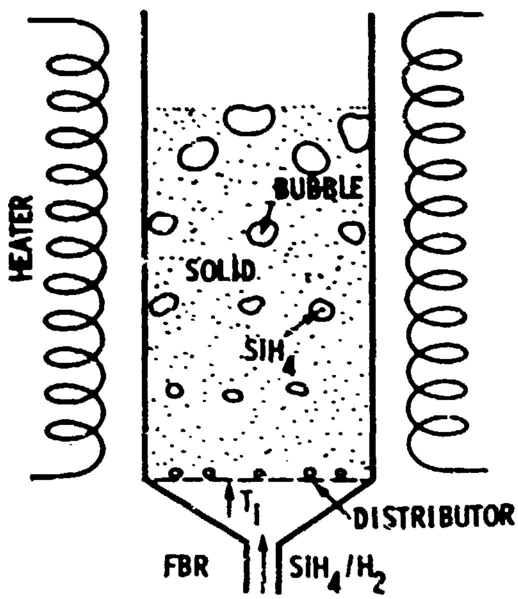
- Si FINES - SUBMICRON POWDERS SAME AS CFP Si FINES
- COLOR CHANGES AT DIFFERENT TEMPERATURES
  - SIZE AT DIFFERENT TEMPERATURES
  - HYDROGEN CONTENT $SiH_x$
    (E.G., $SiH_{0.2}$ STABLE AT ROOM TEMPERATURE AND DOES NOT DECOMPOSE IN AIR)

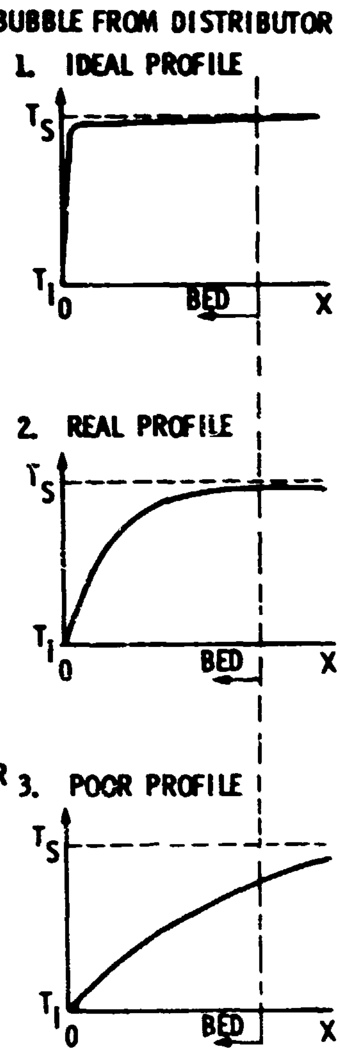
    E.G.,

    AT 650°C - DULL BLACK (FINELY-DISPERSED Si)

    AT 750°C - DARK BROWN
- COLOR IS REFLECTED IN FBR PRODUCT PARTICLES (FBR COATING IS COHERENT AND WITH SMALL POROSITY)

## Heat and Mass Transfer in Fluidized Bed




LOW GAS BUBBLE TEMPERATURE

- LARGE GAS BUBBLES FROM DISTRIBUTOR (CONVECTIVE HEAT TRANSFER)
- INSUFFICIENT HEATING FOR THE BOTTOM HEATER ZONE
- HIGH GAS VELOCITY
- INSUFFICIENT BED HEIGHT
- LOW SOLID TEMPERATURE
- POOR GAS PREHEATING

## Dust Formation

- LESS THAN 10% DUST, FOR PROPER FLUIDIZED BED OPERATION
- TO MAINTAIN THE SAME $U/U_{mf}$ FOR SIMILAR FLUIDIZATION QUALITY,

  $U_{650°C} > U_{750°C}$

  AT 650°C, REACTION ABOVE THE BED LED TO HIGHER DUST FORMATION (e.g. UP TO 17%)
- DUST LEVEL INCREASES MODERATELY WITH SILANE CONCENTRATION
- DUST COLLECTION INCREASES MODERATELY WITH $U/U_{mf}$

## Bed Agglomeration

- NO BED AGGLOMERATION FOR PROPER FLUIDIZATION
  - DISTRIBUTOR: FLOW PATTERN AND GAS BUBBLE SIZE
  - VIGOROUS AGITATION: $U/U_{mf} \geq 3$
  - FAST HEATING AT THE REACTOR BOTTOM (REACTION) REGION

## Key FBR Design Parameters

- DISTRIBUTOR
- HEATER

## Key Operating Parameters

- TEMPERATURE ($650°C \leq T \leq 750°C$)
- FLUIDIZATION QUALITY (e.g. $3 \leq U/U_{mf} \leq 6$)
- BED HEIGHT ($L/D \geq 3$ FOR 2" FBR)

## Kinetic Model

### Overall Growth, Chemical and Physical

$$SiH_4 \longrightarrow Si\,(S) + 2H_2$$

$$A \longrightarrow B$$

$$\frac{dC_B}{dt} = k\,S\,C_{A0}$$

ASSUME N (No. OF SEED PARTICLES/UNIT BED VOLUME) IS CONSTANT

$$S = 4\pi r^2 N$$

$$C_B = \frac{4}{3}\pi r^3 N\rho/M_B, \qquad W_B = \frac{4}{3}\pi r^3 N\rho$$

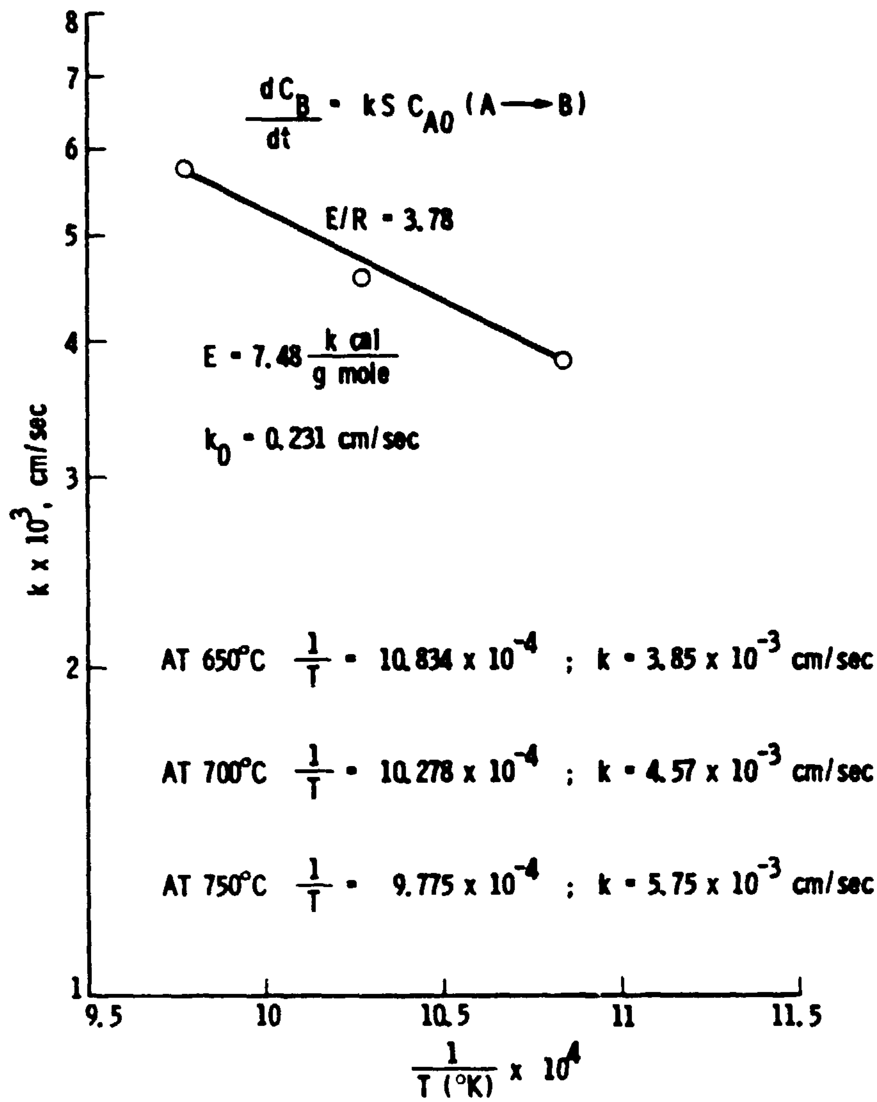
$$S = \left[4\pi N\left(3M_B/\rho\right)^2\right]^{1/3} C_B^{2/3} = bC_B^{2/3}$$

$$\therefore \frac{1}{b\,C_{A0}} \int_{C_{B0}}^{C_B} \frac{dC_B}{C_B^{2/3}} = kt\ , \qquad k = 3\left(C_B^{1/3} - C_{B0}^{1/3}\right)\Big/ bC_{A0}t$$

EQUIVALENT GROWTH MODEL (SEM)

$$\frac{dr}{dt} = k\,C_{A0}\,M_B/\rho\ , \quad k = \left(r - r_0\right)\rho/C_{A0}\,M_B t$$

## First-Order Heterogeneous Deposition Model



FBR Si Particle Growth by SEM
(2-in. FBR, 650°C, 50% $SiH_4/H_2$, $U/U_{mf}$ = 6)

t = 30 min
x = 200/1000

t = 45 min
x = 200

t = 60 min
x = 200

## Particle Growth

I VIA KINETIC MODEL:

MODEL ASSUMES
- UNIFORM DEPOSITION ACROSS THE BED
- NO COATING POROSITY
- CONSTANT VOLUME IN BED

ILLUSTRATION: (TYPICAL VALUES IN THE OPERATING RANGE)

$S = 94\ cm^2/cm^3,\ C_{A0} = 0.01\ gmole/\ell$

$\frac{dC_B}{dt} = 0.5\ C_{A0} = 0.005,\ \frac{dr}{dt} = 0.3\ \mu m/min$

- DEPOSITION RADIUS FROM KINETIC MODEL IS THE LOWER LIMIT FOR ACTUAL GROWTH

II VIA SEM MEASUREMENTS

$$\left.\begin{array}{l} \text{AT } 650^{\circ}C\ \ \Delta r_{60\ min} = 62\ \mu m \\ \text{AT } 700^{\circ}C\ \ \Delta r_{50\ min} = 70\ \mu m \\ \text{AT } 750^{\circ}C\ \ \Delta r_{60\ min} = 100\ \mu m \end{array}\right\} \left(\frac{\Delta r}{\Delta t}_{750^{\circ}C}\right) = 1.67\ \mu m/min$$

- DEPOSITION RADIUS FROM SEM MEASUREMENTS IS THE UPPER LIMIT FOR ACTUAL GROWTH

## Mass Balance Deposition Rate

(2" FBR, $T = 700^{\circ}C$, CONC. = 50% SILANE, $U/U_{mf} = 6$)

DEPOSITION RATE $\cong$ 8.24 gm/min

$\cong$ 0.5 kg/hr

PROJECTION:

FOR 6" FBR AT 50% SILANE, RATE ~ 4.5 kg/hr

FOR 6" FBR AT 100% SILANE, RATE ~ 9 kg/hr

(ASSUME 70% ON STREAM TIME, DEPOSITION RATE ~ 55 MT/yr)

~ TWO 6" FBR's FOR 100 MT/yr

COMPARISON:

SIEMENS TYPE OF REACTOR ($HSiCl_3$)

0.3 - 0.5 kg/hr (LENGTH 40 in., dia 3 in., 6 rods)

DEPENDING ON ROD DEPOSITION QUALITY
(e.g. 20 in. DIAMETER x 4 ft BELL JAR REACTOR)

## Status of FBR Program

- IN 2 Inch FBR SYSTEM:
  - ESTABLISHED CHEMISTRY AND PRODUCT MORPHOLOGY
  - IDENTIFIED OPERATING WINDOW AND DESIGN PARAMETERS
  - DETERMINED DEPOSITION KINETICS
- IN 6 Inch FBR SYSTEM:
  - STUDY DISTRIBUTOR FOR PRACTICAL FLOW (MINIMIZING WALL EFFECT)
  - STUDY BED DEPTH FOR INCREASING FINE COLLECTION EFFICIENCY
  - STUDY THE LIMIT OF SILANE CONCENTRATION
  - STUDY SEED PARTICLE SIZE EFFECT
  - STUDY PRODUCT PURITY

# Basic JPL 6-in. FBR Experimental Plan
## Current Phase (From May 1982)

COMMON CONDITIONS: 700°C, $U/U_{mf}$ = 5, PARTICLE SIZE RANGE: 150-250 μ (MESH No. 60-100)

| BASIC RUN No. | PURPOSE | TYPE (OR REMARK) | SILANE CONC. | BED DEPTH | DURATION |
|---|---|---|---|---|---|
| 1 | ESTABLISH BASELINE CONDITION | POROUS CARBON DISTRIBUTOR | 20% | 6 in | 1 hr. |
| 2 | BED DEPTH TEST | POROUS CARBON DISTRIBUTOR | 20% | 12 in. | 1 hr. |
| 3 | DISTRIBUTOR TESTS | SINTERED METAL (1" THICK) | 20% | 6 in. | 1 hr. |
| 4 | DISTRIBUTOR TESTS | CHOSEN FROM COLD FLOW TEST, METAL PLATE POROUS SCREEN | 20% | 6 in. | 1 hr. |
| 5 | DISTRIBUTOR TESTS | NOZZLE-CONE DISTRIBUTOR | 20% | 6 in. | 1 hr. |
| 6 | REPRODUCIBILITY AND LONG DURATION RUN | DISTRIBUTOR SELECTED FROM DISTRIBUTOR TESTS (FROM RUN No. 6 ON) | 20% | 6 in. | 3 hr. (SAMPLES: 0, 30 min., 1 hr., 2 hr., 3 hr.*) |
| 7 | CONCENTRATION LIMIT TEST | DISTRIBUTOR SELECTED FROM DISTRIBUTOR TESTS (FROM RUN No. 6 ON) | 0-100% (10% INCRE) | 6 in. | TIME AS REQUIRED |
| 8 | DEPOSITION KINETICS | CONTROL RUN FOR PURITY ANALYSIS (ALSO WITH PARTICLE DISTRIBUTION ANALYSIS*) | 50% | 6 in. | 1 hr. (0, 15, 30, 45, 60 min.*) |
| 9 | PARTICLE DEPOSITION DISTRIBUTION | SIMULATED SEED SIZE ADDED (**10% SEED 44-74 μ, MESH No. 325-200) | 50% | 6 in. | 1 hr. (0, 15, 30, 45, 60 min*) |
| 10 | HIGH CONCENTRATION TEST | (COULD USE 12" BED DEPTH, IF NEEDED) | 100% | 6 in. | 30 min. |
| 11 | PURITY EVALUATION | USE QUARTZ LINER (NEUTRON ACTIVATION) | 50% | 6 in. | 1 hr. |