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SILICON PRODUCTION-PROCESS EVALUATIONS

QUARTERLY TECHNICAL PROGRESS REPORT (IV)

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HGW

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ABSTRACT

For the HSC process (Hemlock Semiconductor Corporation), chemical engineering analysis of the process for producing silicon from dichlorosilane in a 1,000 MT/yr plant is nearing completion. Progress and status for the major process engineering activities involved in the analysis are reported: base case conditions (100%), reaction chemistry (100%), process flow diagram (100%), material balance (100%) energy balance (100%), property data (100%), equipment design (90%), major equipment list (90%) and labor (90%).

Engineering design of the third distillation column (D-03, DCS column) in the process was accomplished. The initial design is based on a 94.35% recovery of the light key (DCS, dichlorosilane) in the distillate and a 99.9% recovery of the heavy key (TCS, trichlorosilane) in the bottoms. The specified separation of DCS and TCS is achieved at a reflux ratio of 15 with 20 trays (equilibrium stages). Additional specifications and results are reported including equipment size, temperatures and pressure.

Specific raw material requirements necessary to produce the silicon in the process are presented. The primary raw materials include metallurgical grade silicon, silicon tetrachloride, hydrogen, copper (catalyst) and lime (waste treatment). Hydrogen chloride is produced as a by-product in the silicon deposition.

Cost analysis of the HSC process (Hemlock Semiconductor Corporation) was initiated during this reporting period. The initial cost engineering activities are about 30% complete. The costs for raw materials and utilities necessary to produce silicon in HSC process are reported. Raw material costs are \$2.66 (1980 dollars) and \$3.07 (1982 dollars) per kg of silicon. Utility costs are \$4.75 (1980 dollars) and \$5.69 (1982 dollars) per kg of silicon.

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I. CHEMICAL ENGINEERING ANALYSIS

During this reporting period, primary efforts were continued on the chemical engineering analysis of the HSC process (Hemlock Semiconductor Corporation).

Progress and status for the chemical engineering analysis are summarized below for the major engineering activities:

		Prior	Current
1.	Base Case Conditions	96%	100%
2.	Reaction Chemistry	96%	100%
3.	Process Flow Diagram	85%	100%
4.	Material Balance	85%	100%
5.	Energy Balance	60%	100%
6.	Property Data	60%	100%
7.	Equipment Design	40%	90%
8.	Major Equipment List	30%	90%
9.	Labor	10%	90%

Initial engineering design of the third distillation column (D-03, DCS column) in the process was accomplished. The function of the distillation column is to separate DCS (dichlorosilane) and TCS (trichlorosilane). The distillation column has a single feed which is the distillate from D-02 column.

For the initial process engineering design of D-03 distillation column, the specifications for the separation include a 94.35% recovery of the light key (DCS, dichlorosilane) in the distillate and a 99.9% recovery of the heavy key (TCS, trichlorosilane) in the bottoms. A partial condenser is used for safety reasons in the distillation which is conducted at 90 psia. Additional specifications used in performing the design are given in Appendix A1.

The results for the process engineering design of D-03 indicate the specified separation of DCS and TCS is achieved at a reflux ratio of 15 with 20 trays (equilibrium stages). Additional detailed results including feed tray location and equipment size are given in Appendix A2.

The design curve for D-03 distillation column is shown in Figure I-1. The design curve discloses the results for number of trays (equilibrium stages) required for the separation versus reflux ratio for the distillation.

Status details for the chemical engineering analysis are given in Table I-1. The preliminary process engineering design is based on a 1,000 MT/yr plant for producing silicon from dichlorosilane.

Raw material requirements necessary to produce the product are presented in Table I-2. In the process, the primary raw materials required to produce the silicon include metallurgical grade silicon, silicon tetrachloride, hydrogen, copper (catalyst) and lime (waste treatment). Hydrogen chloride is produced as a by-product in the silicon deposition. The detailed raw material requirements are summarized in the tabulation including hourly flow rate and mass required per kg of silicon produced.

The utility requirements for the process are given in Table I-3. The major utilities include electricity, steam, cooling water, refrigeration, process water and fuel. Among these utilities, the primary utility involving the greatest energy usage is the electricity for the deposition reaction in which silicon is produced by deposition on an electrically heated polycrystalline rod. The surface of the polycrystalline silicon rod is usually in the temperature range of 1000-1100C for the silicon deposition.

Preparation of the list of major process equipment and production labor required to operate the equipment is in progress and nearing completion. The major process equipment and labor requirements are about 90% complete.

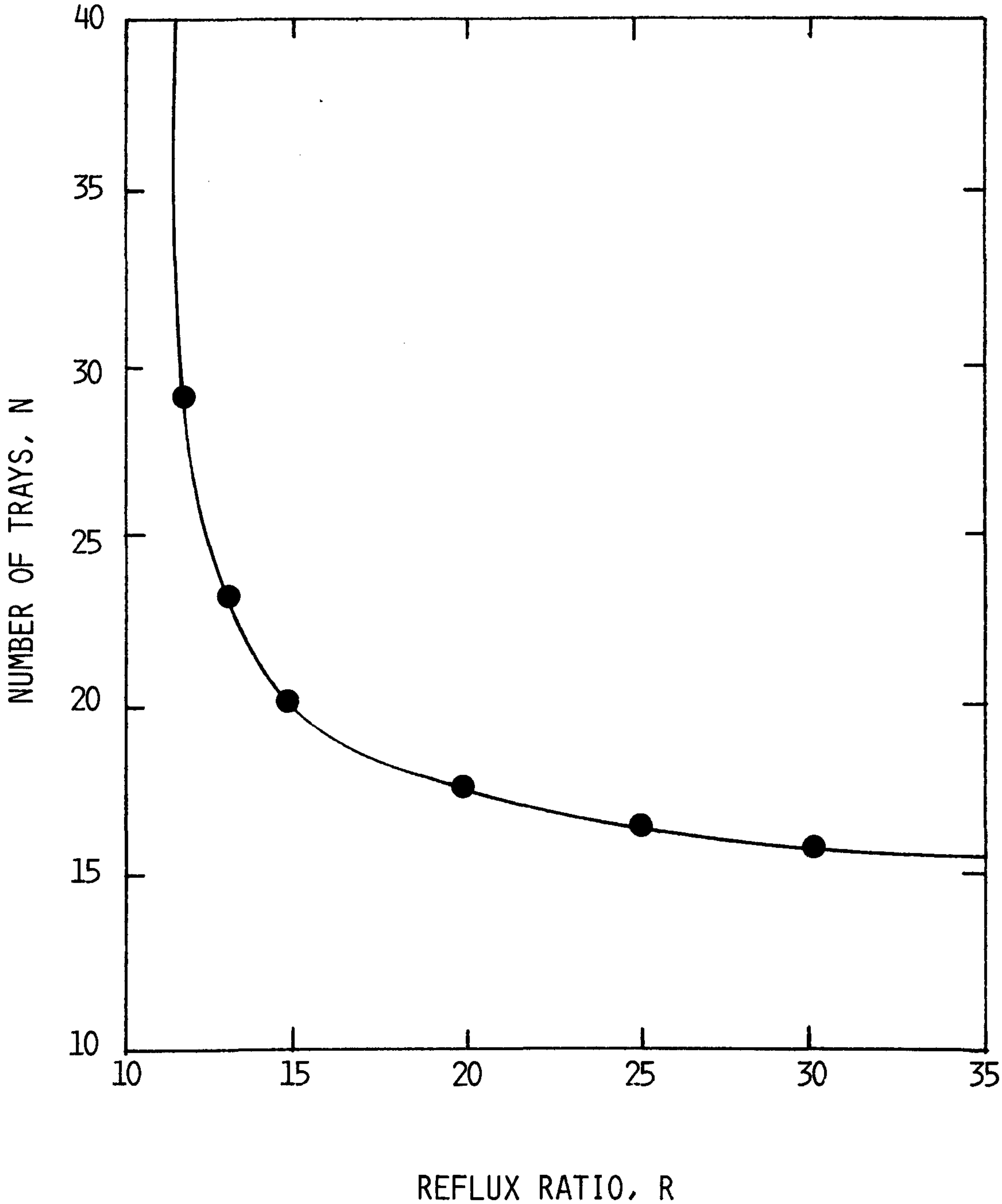

Figure 1-1 Design Curve for Distillation, D-03

Table I-1

CHEMICAL ENGINEERING ANALYSIS:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR HSC PROCESS

Prel. Process Design Activity	Status
1. Specify Base Case Conditions	●
1. Plant Size	●
2. Product Specifics	●
3. Additional Conditions	●
2. Define Reaction Chemistry	●
1. Reactants, Products	●
2. Equilibrium	●
3. Process Flow Diagram	●
1. Flow Sequence, Unit Operations	●
2. Process Conditions (T, P, etc.)	●
3. Environmental	●
4. Company Interaction (Technology Exchange)	●
4. Material Balance Calculations	●
1. Raw Materials	●
2. Products	●
3. By-Products	●
5. Energy Balance Calculations	●
1. Heating	●
2. Cooling	●
3. Additional	●
6. Property Data	●
1. Physical	●
2. Thermodynamic	●
3. Additional	●
7. Equipment Design Calculations	◐
1. Storage Vessels	◐
2. Unit Operations Equipment	◐
3. Process Data (P, T. rate, etc.)	◐
4. Additional	◐
8. List of Major Process Equipment	◐
1. Size	◐
2. Type	◐
3. Materials of Construction	◐
9. Production Labor Requirements	◐
1. Process Technology	◐
2. Production Volume	◐
10. Forward for Economic Analysis	O

O Plan
◐ In Progress
● Complete

TABLE I-2

RAW MATERIAL REQUIREMENTS FOR HSC PROCESS

RAW MATERIALS	REQUIREMENTS	
	lb/hr for 1000 MT/yr Silicon	lb/kg of Silicon
1. M. G. Silicon	270.11	2.014
2. Silicon Tetrachloride ($SiCl_4$, make-up)	535.73	3.67
3. Liquid Hydrogen (H_2, make-up)	45.82	0.342
4. Copper Catalyst	3.44	0.026
5. Hydrate Lime ($Ca(OH)_2$)	259.9	1.937
6. Hydrogen Chloride (HCl, by-product)	129.96	0.969

TABLE I-3

UTILITY REQUIREMENTS FOR HSC PROCESS

UTILITIES	TOTAL REQUIREMENTS FOR PLANT	REQUIREMENTS PER KG OF SILICON
1. Electricity		
1) For Deposition Reaction	12,000 kw	90.0 kw-hr
2) For Gas Compression	260 kw	1.94 kw-hr
3) For Pumping Liquid	55 kw	0.41 kw
	12,315 kw	92.35 kw
2. Steam		
1) Superheated, 100 psia	5 k lb/hr	37.3 lb
2) Saturated, 100 psia	17 k lb/hr	126.7 lb
	22 k lb/hr	164.0 lb
3. Cooling water		
1) Cooling and Condensing	96 k gal/hr	715.6 gal
4. Refrigerant	0.90 M BTU/hr	.007 M Btu
1) Refrigeration		
5. Process Water		
1) Waste Treatment	215 gal	3.39 gal
6. Fuel		
1) Direct-Fired Heater	4 M BTU/hr	.03 M BTU
2) Incineration	1.5 M BTU/hr	.011 M BTU
	5.5 M BTU/hr	.041 M BTU

NOTE:

k = kilo = 10^3

M = mega = 10^6

II. COST ANALYSIS

The cost analysis activity involves an economic analysis of the process under consideration for the production of silicon. The cost analysis for the particular technology is based on process design results, such as requirements for raw materials and major process equipment necessary to produce the product, from the chemical engineering analysis activity. Primary results issuing from the cost analysis include plant capital investment and product cost which are useful in identification of those processes showing promise for meeting project cost goals.

During this reporting period, cost analysis of the HSC process (Hemlock Semiconductor Corporation) was initiated for a 1,000 MT/yr silicon plant. Progress and status for the major cost engineering activities involved in the analysis are summarized below:

		Prior	Current
1.	Process Design Inputs	10%	50%
2.	Base Case Conditions	10%	50%
3.	Raw Material Costs	0%	50%
4.	Utility Costs	0%	50%
5.	Major Process Equipment Costs	0%	20%
6.	Production Labor Costs	0%	10%
7.	Plant Investment Cost	0%	Plan
8.	Total Product Cost	0%	Plan

Status details for the cost analysis are shown in Table II-1.

The cost of raw materials necessary to produce the product are presented in Table II-2. The tabulation indicates that raw material costs are $2.66 (1980 dollars) and $3.07 (1982 dollars) per kg of silicon.

The cost of utilities required for the production of the product are given in Table II-3. The utility costs are $4.75 (1980 dollars) and $5.69 (1982 dollars) per kg of silicon.

Cost estimation for the major process equipment necessary to produce the silicon product is in progress. Determination of the major process equipment costs is about 30% complete.

TABLE II-1

COST ANALYSIS:
PRELIMINARY ECONOMIC ANALYSIS ACTIVITIES FOR HSC PROCESS

Prel. Process Economic Activity	Status
1. Process Design Inputs	◒
1. Raw Material Requirements	◒
2. Utility Requirements	0
3. Equipment List	0
4. Labor Requirements	◒
2. Specify Base Case Conditions	◒
1. Base Year for Costs	◒
2. Appropriate Indices for Costs	◒
3. Additional	◒
3. Raw Material Costs	◒
1. Base Cost/Lb of Material	◒
2. Material Cost/Kg of Silicon	◒
3. Total Cost/Kg of Silicon	◒
4. Utility Costs	◒
1. Base Cost for Each Utility	◒
2. Utility Cost/Kg of Silicon	◒
3. Total Cost/Kg of Silicon	◒
5. Major Process Equipment Costs	◒
1. Individual Equipment Cost	◒
2. Cost Index Adjustment	◒

Prel. Process Economic Activity	Status
6. Production Labor Costs	0
1. Base Cost Per Man Hour	0
2. Cost/Kg Silicon Per Area	0
3. Total Cost/Kg Silicon	0
7. Estimation of Plant Investment	0
1. Battery Limits Direct Costs	0
2. Other Direct Costs	0
3. Indirect Costs	0
4. Contingency	0
5. Total Plant Investment (Fixed Capital)	0
8. Estimation of Total Product Cost	0
1. Direct Manufacturing Cost	0
2. Indirect Manufacturing Cost	0
3. Plant Overhead	0
4. By-Product Credit	0
5. General Expenses	0
6. Total Cost of Product	0

0 Plan
◒ In Progress
● Complete

TABLE II-2

RAW MATERIAL COST FOR HSC PROCESS

	Raw Material	Raw Material Requirement, lb/kg of Si	Raw Material Cost, $/lb of Material: 1980 dollars	Raw Material Cost, $/lb of Material: 1982 dollars	Cost, $/kg of Si: 1980 dollars	Cost, $/kg of Si: 1982 dollars
1.	M. G. Silicon	2.014	0.63	0.62	1.27	1.25
2.	Silicon Tetrachloride ($SiCl_4$, make-up)	3.67	0.26	0.36	0.95	1.32
3.	Liquid Hydrogen (H_2, make-up)	0.342	1.485	1.679	0.51	0.57
4.	Copper Catalyst	0.026	0.75	0.75	0.02	0.02
5.	Hydrate Lime ($CA(OH)_2$)	1.937	0.017 (33.5 $/ton)	0.017	0.03	0.03
6.	Hydrogen Chloride (HCl, by-product)	0.969	0.12	0.12	(0.12)	(0.12)
					2.66	3.07

TABLE II-3

UTILITY COST FOR HSC PROCESS

	Utility	Utility Requirement Unit/kg of Si	Utility Cost, $/unit		Cost, $/kg of Si	
			1980 dollars	1982 dollars	1980 dollars	1982 dollars
1.	Electricity	92.35 kw-hr	.045 $/kw-hr	.054 $/kw-hr	4.16	4.99
2.	Steam	164 lb	1.89 $/k lb	2.268 $/k lb	0.310	0.372
3.	Cooling Water	715.6 gal	.126 $/k gal	0.151 $/k gal	0.090	0.108
4.	Refrigeration	.007 M BTU	14.7 $/M BTU	17.64 $/M BTU	0.103	0.124
5.	Process Water	3.39 gal	.567 $/k gal	0.680 $/k gal	0.002	0.002
6.	Fuel	.041 M BTU	1.96 $/M BTU	2.352 $/M BTU	0.080	0.096
					4.745	5.694

Note:

k = kilo = 10^3
M = mega = 10^6

III. SUMMARY - CONCLUSIONS

As a result of accomplishments during this reporting period, the following summary-conclusions are made:

1. For the HSC process (Hemlock Semiconductor Corporation), chemical engineering analysis of the process for producing silicon from dichlorosilane in a 1,000 MT/yr plant is nearing completion. Progress and status for the major process engineering activities involved in the analysis are reported: base case conditions (100%), reaction chemistry (100%), process flow diagram (100%), material balance (100%), energy balance (100%), property data (100%), equipment design (90%), major equipment list (90%) and labor (90%).

2. Initial engineering design of the third distillation column (D-03, DCS column) in the process was accomplished. The initial design is based on a 94.35% recovery of the light key (DCS, dichlorosilane) in the distillate and a 99.9% recovery of the heavy key (TCS, trichlorosilane) in the bottoms. The specified separation of DCS and TCS is achieved at a reflux ratio of 15 with 20 trays (equilibrium stages). Additional specifications and results are reported including equipment size, temperatures and pressure.

3, Cost analysis of the HSC process (Hemlock Semiconductor Corporation) was initiated during this reporting period. The initial cost engineering activities are about 30% complete. The costs for raw materials and utilities necessary to produce silicon in HSC process are reported. Raw material costs are $2.66 (1980 dollars) and $3.07 (1982 dollars) per kg of silicon. Utility costs are $4.75 (1980 dollars) and $5.69 (1982 dollars) per kg of silicon.

IV. PLANS

Plans for the next reporting period are summarized below:

1. Continue chemical engineering analysis of the HSC process (Hemlock Semiconductor Corporation) for silicon.

2. For the preliminary process design, major efforts will be devoted to equipment design, major equipment list and labor requirements.

3. Cost analysis will continue as results issue from the chemical engineering analysis.

APPENDIX A1

PROCESS ENGINEERING: DESIGN SPECIFICATIONS FOR DISTILLATION, D-03

Issue No. 1

1. Process Equipment Name Distillation, D-03 (DCS Column)

2. Process Equipment Function Separation of DCS (Dichlorosilane) and TCS (Trichlorosilane).

3. Feed Specifications

   1. No. of Feeds 1
   2. No. of Feed Components 4
   3. Feed Components MCS, DCS, TCS, TET
   4. Feed Concentration See Item 7
   5. Feed Temperature 91 C
   6. Feed Pressure 90 Psia
   7. Light Key - LK Dichlorosilane (DCS)
   8. Heavy Key - HK Trichlorosilane (TCS)

4. Distillate Specifications

   1. Recovery of Light Key (LK) in Distillate 94.35 %
   2. Concentration Spec. Low TCS, TET

5. Bottoms Specifications

   1. Recovery of Heavy Key (HK) in Bottoms 99.9 %
   2. Concentration Spec. Low in MCS, DCS

6. General Specifications

   1. Pressure for Distillation 90 psia
   2. Condenser Type Partial

   Required amount for feed of CVD reactors is drawn from the top of this column in vapor phase, it is then mixtured with H_2 and feeded to CVD reactor. Only refulx flow is condensed and collected in accumulator, and feeded back to column.

7. Feed Concentration

Component	Feed Concentration
1. SiH_3Cl , MCS	0.0044870
2. SiH_2Cl_2, DCS	0.1034000
3. $SiHCl_3$, TCS	0.8856370
4. $SiCl_4$, TET	0.0064760
Total	1.000000
Temperature (C)	91
Pressure (Psia)	90
Mass Flow (lb-mole/hr)	256.53
Liquid Fraction	1.
Feed Source	Distillate of D-02

Note :

1. Feed concentration and temperature are from design of D-02

APPENDIX A2

PROCESS ENGINEERING: DESIGN RESULTS FOR DISTILLATION, D-03

Issue No. 1

1. Process Equipment Name Distillation, D-03 (DCS Column)

2. Equipment Specifications
   1. No. of Equilibrium Trays N = 20
   2. No. of Equilibrium Feed Tray N_F = 11
   3. Tray Efficiency 65 %
   4. No. of Actual Trays N_{actual} = 32
   5. No. of Actual Feed Tray $N_{F,actual}$ = 16
   6. Tray Spacing 18 in.
   7. Type of Tray Single Pass Crossflow Seive Tray
   8. Column Diameter 4 ft.
   9. Column Height 60 ft.
   10. Reflux Ratio R = 15
   11. Design Temp. Top = 52 C
       Bottom = 97 C
   12. Design Pressure 90 psia
   13. Materials of Construction Steel

3. Product Specifications
   1. Feed Specifications
      1. Feed Concentration See Item 7 of Design Spec.
      2. Light Key - LK Dichlorosilane (DCS)
      3. Heavy Key - HK Trichlorosilane (TCS)
   2. Distillate Specifications
      1. Recovery of Light Key (LK) in Distillate 94.35 %
      2. Concentration Spec. See Item 4
   3. Bottoms Specifications
      1. Recovery of Heavy Key (HK) in Bottoms 99.9 %
      2. Concentration Spec. See Item 4

4. Results for Streams Concentration

Component		Concentration	
		Distillate	Bottom
1. SiH_3Cl ,	MCS	0.0435621	Neglibile
2. SiH_2Cl_2,	DCS	0.9390411	0.0076186
3. $SiHCl_3$,	TCS	0.0173968	0.9837867
4. $SiCl_4$,	TET	neglibile	0.0085947
Total		1.000000	1.0000000

Distillate : 26.3 lb-mole/hr at 52 ^{0}C

Bottom : 230.23 lb-mole/hr at 97 ^{0}C

5. Results for Number of Trays

Reflux Ratio, R	No. of Equil. Trays, N	No. of Actual Trays N_{actual}
12	29 (13)	45
14	23 (12)	36
15	20 (11)	31
20	18 (11)	28
25	17 (10)	27
30	16 (10)	25

MILESTONE CHART