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DEVELOPMENT OF ADVANCED METHODS FOR CONTINUOUS CZOCHRALSKI GROWTH

Silicon Sheet Growth Development for the Large Area Silicon Sheet Task of the Low Cost Silicon Solar Array Project

FIRST QUARTERLY PROGRESS REPORT

By

R. G. Wolfson

C. B. Sibley

January 11, 1978

Reporting Period October 4, 1977 to December 30, 1977

JPL Contract No. 954884

Varian Associates
Lexington Vacuum Division
121 Hartwell Avenue
Lexington, Massachusetts 02173

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, under NASA Contract NAS7-100 for the U.S. Department of Energy.

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TABLE OF CONTENTS

1.0 SUMMARY

The overall purpose of this two-stage program is the demonstration of continuous Czochralski growth. In Stage 1, an existing Varian 2850 Czochralski furnace is to be modified for continuous growth by recharging; in Stage 2, a new puller is to be designed and shown capable of producing 100 kg of crystal from the same crucible with an after-grind yield of at least 70%. The three components required to modify the Varian furnace for batch and continuous recharging with granular silicon have been designed and are currently being fabricated. The feasibility of extended growth cycles up to 40 hours long has been demonstrated by a recharge simulation experiment; a 6 inch diameter crystal was pulled from a 20 kg charge, remelted, and pulled again for a total of four growth cycles, 59-1/8 inch of body length, and ∿65 kg of calculated mass. A preliminary economic analysis indicates that the 1982 SAMICS price goal allocation can be met by sheet production predicated upon the recharging of otherwise standard Czochralski furnaces; the projections are, in 1975 dollars: add-on cost for growth -- $37 per m^2, direct poly cost -- $46 per m^2, margin available for wafering -- $45 per m^2.

2.0 INTRODUCTION

The overall purpose of this program, which began on October 16, 1977, is the demonstration of continuous Czochralski growth, defined as a throughput of silicon that produces 100 kg of single-crystal material, 10 cm or greater in diameter, from one common container. The two prime constraints on the process to be developed are as follows:

-- It is to be amenable to scaling up to large-quantity, minimum-cost processing of silicon consistent with the established cost goals of ≲$2,000 per peak kilowatt for completed arrays by 1982 and ≲$500 per peak kilowatt by 1986.

-- It is to be capable of producing silicon with the purity and the degree of structural perfection required for the subsequent fabrication of solar cells with a terrestrial efficiency greater than 14%.

The program is in two coordinated stages, each of which yields a functioning Czochralski puller. In Stage 1, an existing Varian 2850 Czochralski furnace is to be modified for continuous growth by recharging. The feasibility of 100 kg growth cycles is to be demonstrated by simulation, and continuous Czochralski growth is then to be accomplished using batch recharging with granular silicon; in addition, continuous recharging with granular silicon is to be attempted, and recharging with molten silicon is to be considered. In Stage 2, a new, prototype Czochralski puller is to be designed and shown capable of producing 100 kg of crystal from the same crucible with an after-grind yield of 70% or greater.

The work of this first quarter addressed the Stage 1 objectives, with specific emphasis on the modification of the Varian 2850 puller and the simulation of 100 kg growth cycles.

3.0 RESULTS

3.1 Furnace Modification

The three additions required to modify the Varian 2850 Czochralski furnace for batch and continuous recharging are as follows:

-- a vacuum valve to replace the present gate valve between the growth chamber and the crystal-removal chamber; this valve is to isolate the hot crucible while recharging and crystal removal are in progress.

-- a pellet-feed charger capable of handling granular silicon up to centimeter size; this is to enter the growth chamber through a port in the existing furnace wall.

-- a charge-isolation lock to permit recharging without contamination by the ambient atmosphere; this is to include an auxiliary pumping system with its own vacuum pump and port for evacuation and backfilling.

These three components are shown in the schematic diagram of the modified Czochralski puller, Fig. 1.

The vacuum valve to replace the present gate valve between the growth chamber and the crystal-removal chamber has been designed and fabricated, utilizing a commercially available 6-inch gate valve. In order to adapt this valve for use in the Varian 2850 furnace, the surfaces of both the body and the disc were traced

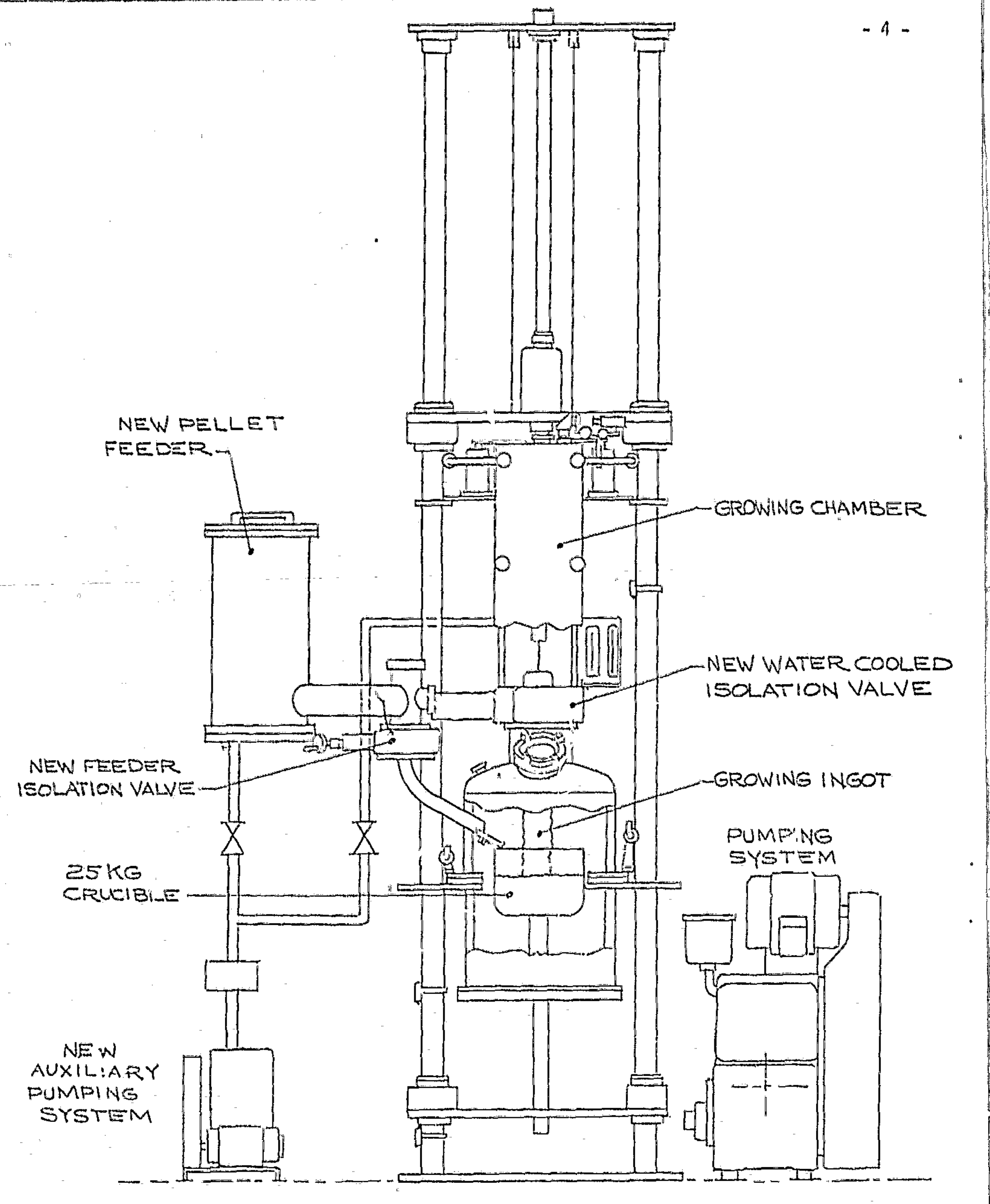


FIG 1 CZOCHRALSKI CRYSTAL PULLER FURNACE WITH BULK CHARGER & ISOLATION VALVE

with 1/4-inch copper tubing for water cooling. Shielding was added to the valve carriage assembly to protect the disc from mechanical damage, and the flanges were modified to permit accurate, dependable mating with the growth chamber and the crystal-removal chamber.

T' pellet-feed charger and the charge-isolation lock have been designed and are currently being fabricated; they are shown schematically in Fig. 2. Several feed hopper geometries were studied; that with an included (cone) angle of 60° was selected, built, and tested successfully. Four feed trays were built and tested: V-bottom, flat-bottom, round-bottom, and enclosed-tubular; the V-bottom tray was found to be the most satisfactory for 1-2 cm nuggets and crushed repull silicon. A horizontal-axis charge-isolation valve was tested with a retractable upper feed tube, which was rejected in favor of a vertical free-fall design. Tests of this latter arrangement, with the vibratory feeder dropping granular silicon through the valve, were successful. They did, however, point up the need for a simple inner guide tube to prevent fines from lodging in the isolation valve; the guide tube has been designed to retract into the lower feed tube when the valve is to be closed. Lower feed tubes were fabricated in several sizes and configurations, and it was determined that a 5.7 cm tube at 30° to the horizontal would best serve the purpose. The lower feed tube terminates in a refractory extension which can be retracted to cover the end of the tube. In this position, which will be normal during growth, the extension will prevent silicon fines from entering the chamber and SiO from depositing on the inner surface of the tube; it will also act as a heat shield.

A full-size mock-up of the recharging system -- comprising the feed hopper, the vibratory feeder and tray, the isolation valve, and the lower feed tube -- was used in ambient to charge

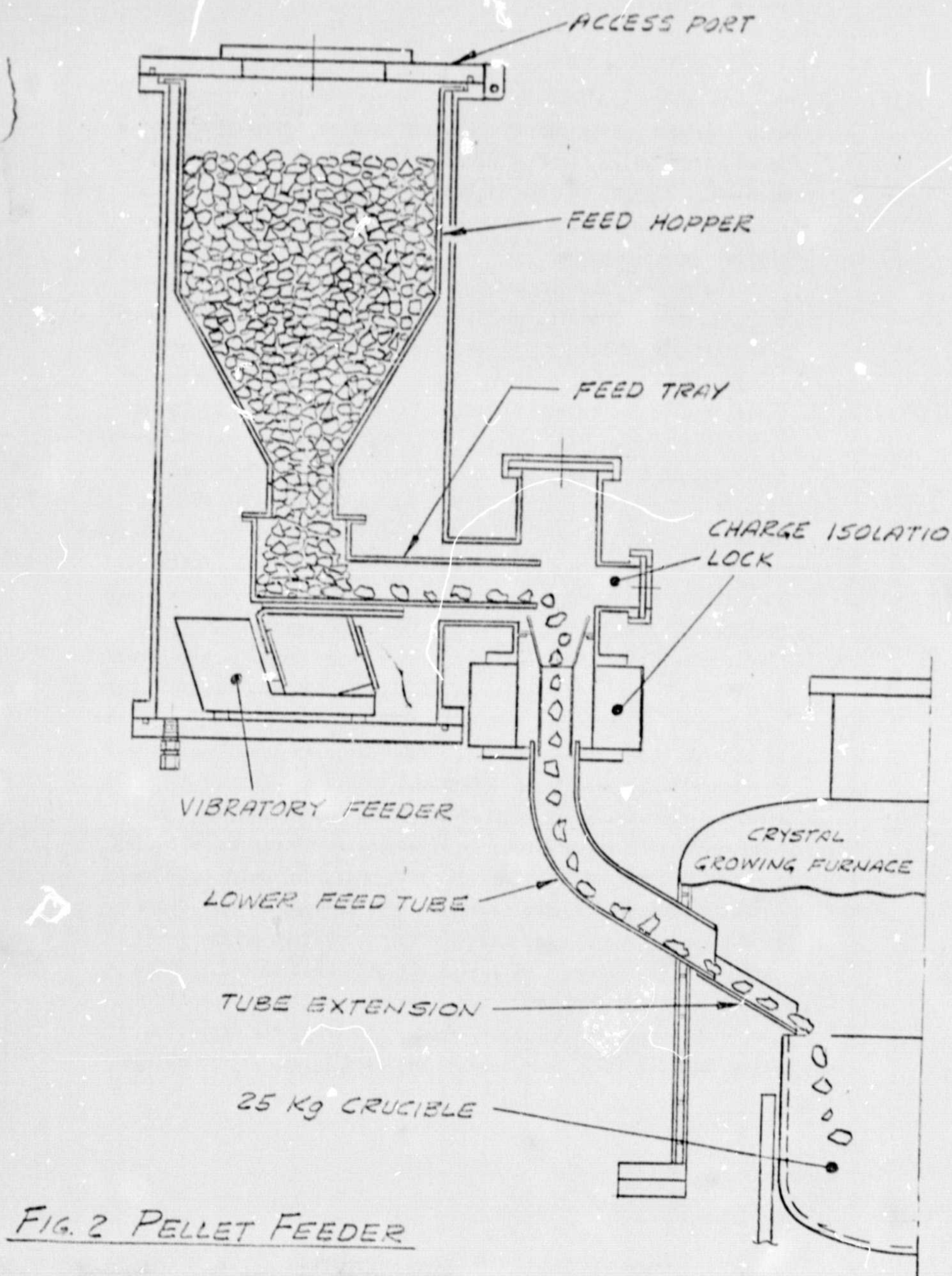


FIG. 2 PELLET FEEDER

a standard 12 inch diameter quartz crucible with 1-2 cm crushed repull silicon. The tests indicated that it would be prudent to limit the free fall of the silicon feed to a maximum of ~25 cm and to bring the lower feeder tube through the furnace wall at a point lower than the existing dopant port.

3.2 100 kg Growth Cycle

The feasibility of 100 kg growth cycles is to be demonstrated, and the salient process parameters determined, prior to the development of batch continuous recharging. This demonstration is to be accomplished by simulation; viz., by pulling and remelting one ingot for a sufficient number of cycles.

The first attempt at recharge simulation was terminated prematurely by an accident attributable to lack of operator experience in the melting back of large crystals. Four 6 inch diameter ingots had been grown from the same 20 kg charge. During the remelt portion of the fourth cycle, the crystal was advanced too rapidly and touched the bottom of the crucible, snapping the seed holder and falling athwart the crucible, which was subsequently found to have been undamaged. Fortunately, the upper few inches of this fourth crystal survived intact the emergency shut-down and the subsequent freezing of the melt, as did large portions of the crucible; these have been retained for analysis.

Although the goal of 100 kg was not reached in this first trial, it was nevertheless demonstrated that Czochralski growth at reduced pressure (vacuum growth) can continue for 40 hours with neither machine malfunction nor excessive accumulation of silicon monoxide. The simulation of the growth of four 6 inch (nominal) diameter ingots from one crucible yielded a total body length of 59-1/8 inch, with a calculated mass of ~65 kg. With reference to process parameters, it appears than an average growth rate of ∿6.5 cm per hour (2.5 inch per hour) for 6 inch diameter crystals, corresponding to a solidification rate of ∿2.7 kg per hour, is compatible with dislocation-free growth, whereas rates much in excess of 7.5 cm per hour (3 inches per hour) lead to structural degradation. Remelting of the crystal can be accomplished at ∿20 cm per hour (nearly 8 inches per hour) with a power input of 80 kilowatt, provided that 3 kg of melt is retained in the crucible; this corresponds to a recharging rate of 8.5 kg per hour. Under the conditions of this experiment, attempts to remelt the crystal at rates approaching 30 cm per hour (12 inches per hour) are very risky, indeed.

3.3 Economic Analysis

The interim price estimation guidelines standards for SAMICS impose the following price goal allocation for wafer production in the year 1982:

$$\$128 \text{ per } m^2$$

(Here, as in the remainder of this discussion, 1975 dollars are used.) A preliminary analysis of the economics of continuous Czochralski growth indicates that this goal can be met by sheet production based upon recharging. Specifically, it suggests that the 1982 goal can be achieved by the rechargeable Czochralski furnace proposed in Stage 1 of the present program.

The cost reduction attendant upon the addition of recharging capability to an otherwise standard Czochralski furnace is developed in the calculations summarized in Tables I and II of Apendix III. Table I shows the present state of the art: one crystal grown from a 20 kg charge. At 55% after-grind yield, the add-on cost for Czochralski growth is $53 per kg, with the cost of poly silicon set at the 1982 price goal allocation of \$25.00 per kg. Using the 1980 slicing yield goal of 1.00 m^2 per kg, the cost of Czochralski silicon projected on the basis of current technology would be \$99 per m^2 of wafer. Subtraction of this value from the wafer price allocation of \$128 per m^2 yields the margin available for the wafering add-on cost: \$29 per m^2. Table II extrapolates the same conditions to the rechargeable furnace of Stage 1, with 100 kg of single-crystal silicon pulled from one crucible; here, the cost of the Czochralski furnace was increased by 36% in order to reflect the addition of the recharging capability. Batch recharging lowers the add-on cost at 55% yield to \$37 per m^2. The direct poly cost remains constant, of course, and the margin available for wafering increases to \$45 per m^2. This last-mentioned value appears to be a reasonable projection for the multiblade slurry saw.

4.0 DISCUSSION

4.1 Relevance to Program Goals

The modification of the Varian 2850 furnace and the simulation of extended crystal growths, together, address the 1982 cost goal of \$2,000 per peak kilowatt. In order to decrease the add-on cost for Czochralski growth to a value commensurate with this figure, the crucible cost per unit mass of usable crystal must be reduced to about one-fifth of its present value. This is to be effected by continuous growth, which requires the capability to recharge the crucible while maintaining suitable growth conditions for long periods.

Recharging with solid silicon has been achieved repeatedly by the semiconductor industry, and the mechanical design is well within the current state of the art. In the past, however, subsequent crystal yields have been unacceptably low because of the effects of heavy silicon monoxide build-up within the furnace. Thus, the twofold requirement of recharging and of maintaining proper growth conditions reduces to minimizing silicon monoxide deposition, which Varian has accomplished by the development of a process of crystal growth at reduced pressures -- the so-called vacuum process.

Silicon monoxide is evolved by the melt as the product of its reduction of the quartz crucible. The monoxide deposits upon the cooler surfaces within the growth chamber, and, under conditions of rapid accumulation, flakes off to drop into the melt and terminate dislocation-free growth. Indeed, it appears that vapor-phase agglomeration can produce the same result. The vacuum process avoids this problem by using a comparatively low argon flow at reduced pressure to sweep the monoxide from the growth chamber before either deposition or agglomeration can occur. In principle, crystal growths on the order of days in duration are possible, but it would be prudent to obtain experimental corroboration as early in this program as is possible.

4.2 Conclusions and Recommendations

Since this work has been in progress only a little more than two months, no definite conclusions can be drawn. The progress so far, however, supports the view that the goals of the program are attainable and that the time constraints are realistic.

The sole recommendation is that ongoing efforts adhere to the original program plan.

5.0 PLANS

Plans for the immediate future are as follows:

- -- Completion of the modification of the Varian 2850 Czochralski furnace.
- -- Simulation of 100 kg growth cycles.

Plans for the next quarter conform to the program plan and include the following:

-- Demonstration of batch recharging with solid silicon by pulling 100 kg of ingot from one crucible in a continuous growth process.

-- Start of design of the transducer array and the control system for the prototype puller.

APPENDIX I

New Technology

Engineering Drawings and Sketches

NEW TECHNOLOGY

No new technology was generated during the reporting period.

ENGINEERING DRAWINGS AND SKETCHES

The engineering drawings of the three additions to the Varian 2850 Czochralski furnace -- viz., the vacuum valve, the pellet-feed charger, and the charge-isolation lock -- will be included in the next quarterly report. Sketches of the modified puller and of the pellet-feed charger were included in Section 3.1 as Figures 1 and 2, respectively.

APPENDIX II

Man Hours and Costs

Program Plans, Updated

-- Milestone Chart

-- Direct Labor Summary

-- Incurred Cost Summary

MAN HOURS AND COSTS

The previous totals of both man hours and costs were zero. During the repo ting period of October 4 - December 30, 1977, total man hours were 1,466 and total costs were $56,378. As of December 30, 1977, the cumulative totals were thus 1,466 man hours and $56,378.

DEVELOPMENT OF ADVANCED METHODS FOR CONTINUOUS CZOCHRALSKI GROWTH

Varian Associates/Lexington Vacuum Division
JPL Contract 954884

Recharging

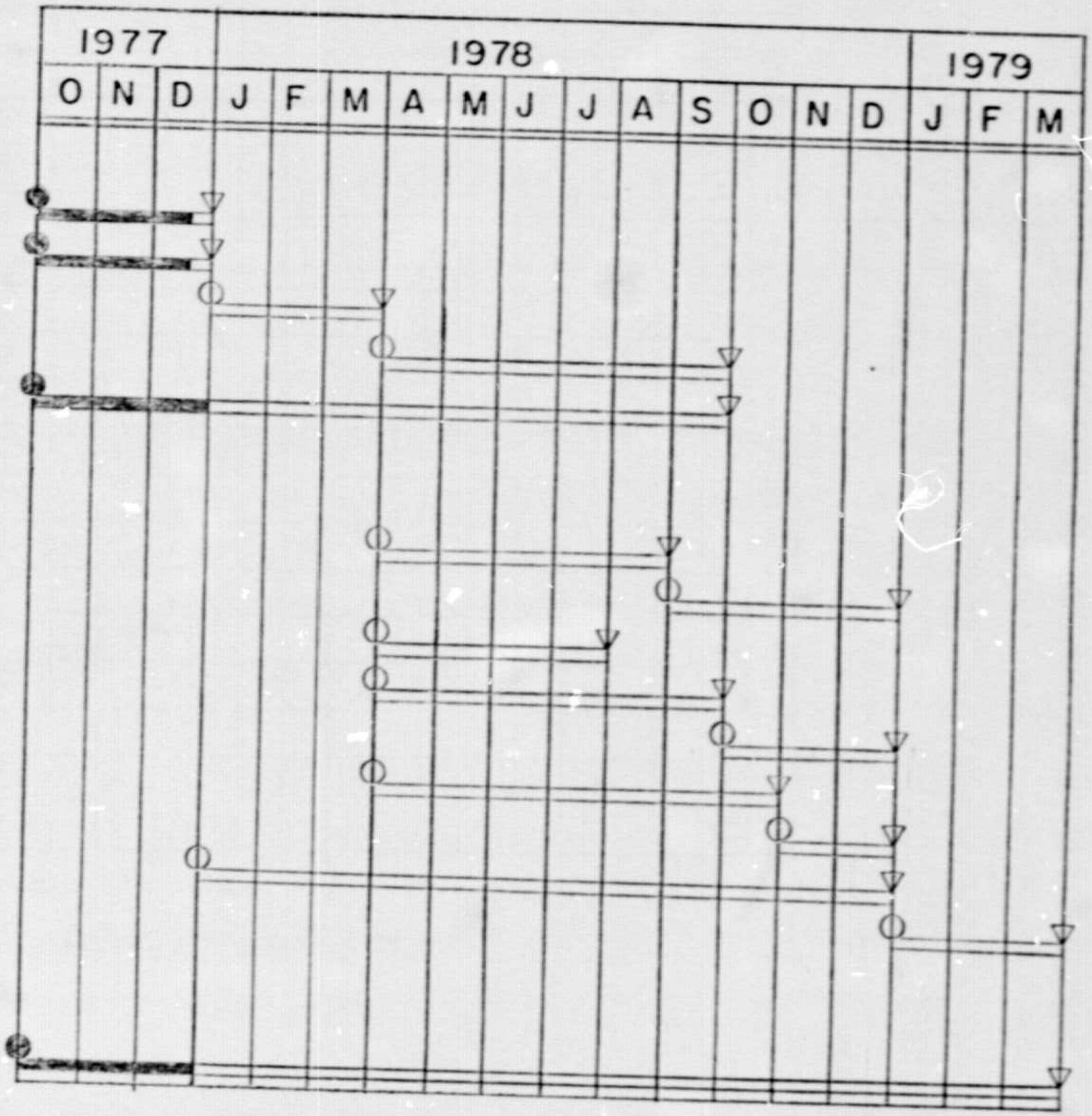
1. Furnace Modification
2. 100 kg Growth Cycle
3. Batch Recharging with Solid
4. Continuous Recharging with Solid
5. Continuous Recharging with Liquid

Prototype Puller

6. Lower Assembly: Design
   Manufacture
7. Hot Zone and Power Supply
8. Crystal Lift Mechanism: Design
   Manufacture
9. Upper Assembly: Design
   Manufacture
10. Transducers and Control
11. Process Demonstration

Design Analysis Cost Study

12. Economic Model



RGW 10/22/77
Updated 1/11/78

MILESTONE CHART

DEVELOPMENT OF ADVANCED METHODS FOR CONTINUOUS CZOCHRALSKI GROWTH

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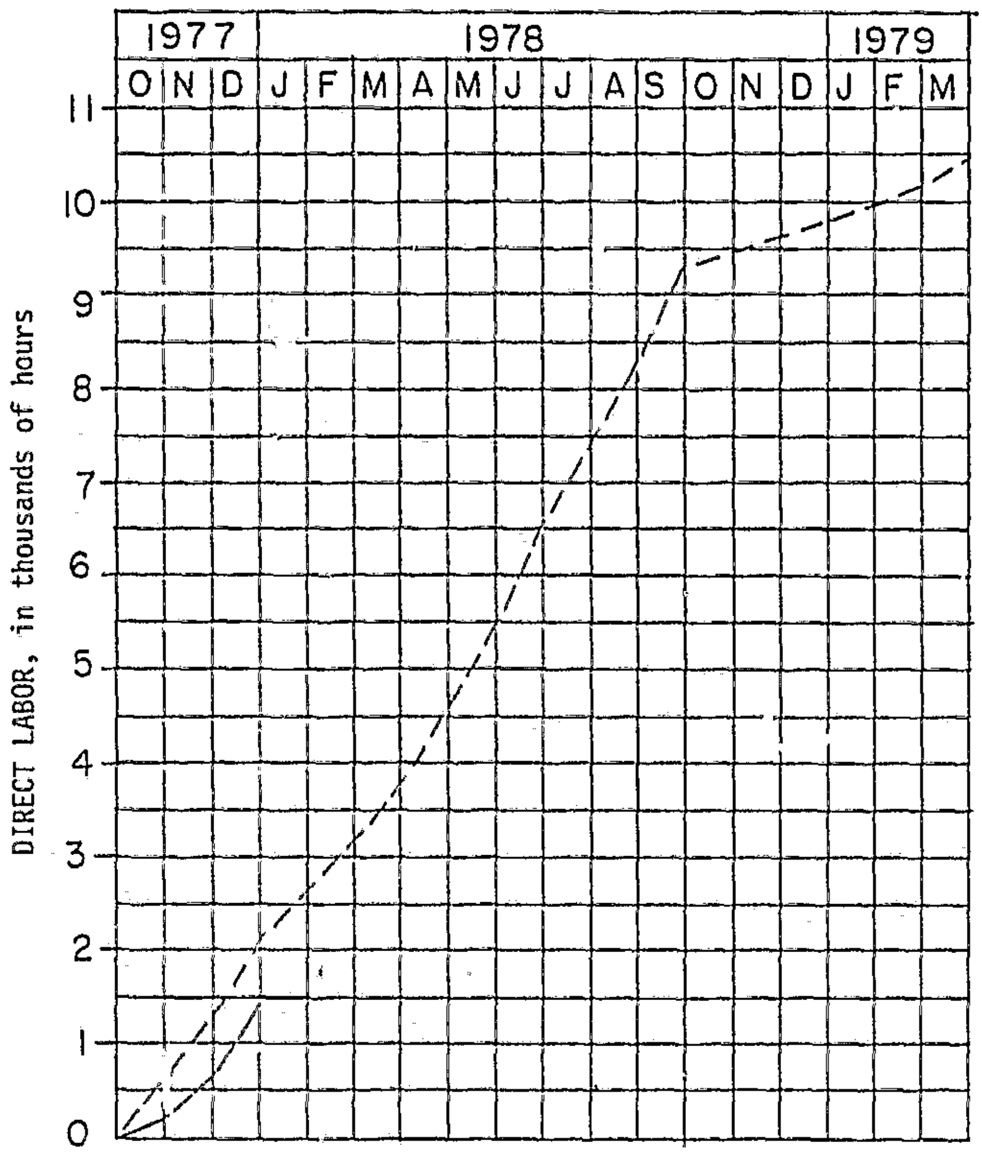
SC-2 Program Plan
Page 2 of 3 pages



RGW 10/20/77
Updated 1/11/78

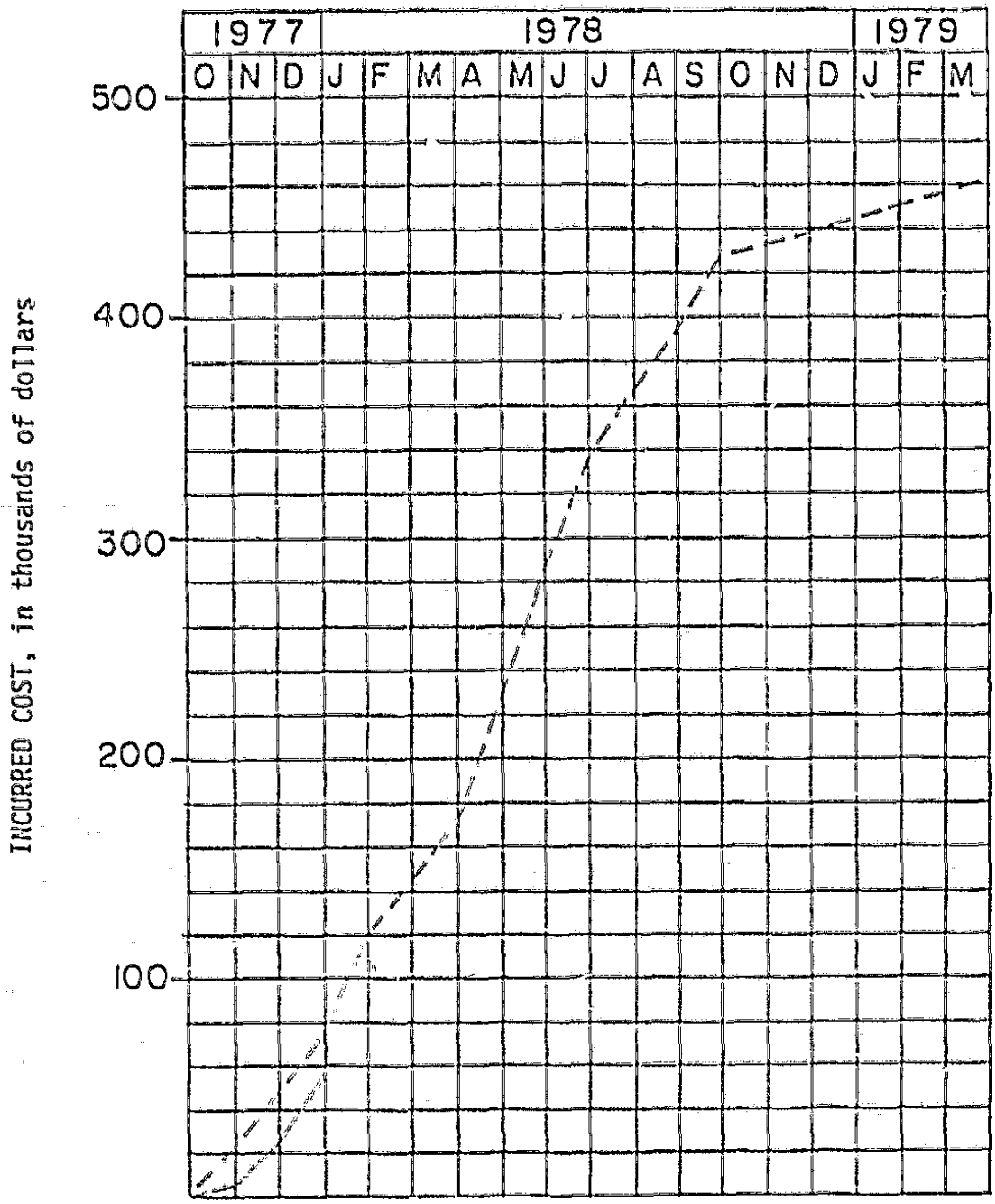
Total Hours: 10,440
Hours to Date: 1,466

Planned - - - -
Incurred ———

DIRECT LABOR SUMMARY

DEVELOPMENT OF ADVANCED METHODS FOR CONTINUOUS CZOCHRALSKI GROWTH

Varian Associates/Lexington Vacuum Division
JPL Contract 954884



RGW 10/20/77
Updated 1/11/78

Total Cost: $460,155
Cost to Date: $ 56,378

Planned - - - -
Incurred ———

INCURRED COST SUMMARY

APPENDIX III

Summary of Economic Analysis Data

TABLE I

ONE CRYSTAL GROWN FROM A 12 INCH DIAMETER CRUCIBLE

Description of Czochralski Growth Process

A. Facilities and Operation

- -- Cost of Czochralski furnace: $110,000
- -- Floor space per Czochralski furnace: 100 ft^2
- -- Utilization of Czochralski furnace: 85%
- -- Czochralski furnaces per operator: 3
- -- Operating days per year: 345
- -- Labor rate: $4.50/hr.
- -- After-grind yield: 55%

B. Process

- -- Average solidification rate: 2 kg/hr
- -- Argon flow: 40 ft^3 (standard)/hr
- -- Power for melting: 80 kw
- -- Power for steady-state growth: 60 kw
- -- Mass of taper: 1 kg
- -- Mass left in crucible: 1 kg

C. Cycle Time

-- Clean furnace	0.5 hr
-- Load and melt charge (20 kg)	2.5 hr
-- Seed; grow neck, crown, and shoulder	1.0 hr
-- Grow body of crystal (18 kg)	9.0 hr
-- Grow taper (1 kg)	0.5 hr
-- Cool and remove crystal	1.5 hr
-- Maintain and repair furnace	2.5 hr
	17.5 hr

TABLE I (continued)

Input Data

1. Direct Equipment Initial Capital Cost

-- Czochralski furnace		$ 110,000
	EQPT	$ 110,000

2. Direct Manufacturing Floor Space

-- Czochralski furnace		100 ft^2
	SQFT	100 ft^2

3. Annual Direct Labor Salaries

-- Operator/furnace-year (8/3 operator-hr/furnace-shift)(3 shift/day) (345 day/year) ($4.50/hr)		$ 12,420
	DLAB	$ 12,420

4. Directly Used Materials and Supplies

-- Poly charge/furnace-year @ $30.08/kg* (20 kg/cycle) (473 cycle/year) ($30.08/kg)		$ 284,557
-- Crucible/furnace-year @ $210 ea. (1 crucible/cycle) (473 cycle/year) ($210 ea)		$ 99,330

---

* This is the 1982 price goal allocation for FPOLY expressed in 1978 dollars.

TABLE I (continued)

	-- Argon/furnace-year @ $\$0.012/ft^3$ (13.5 hr/cycle) (473 cycle/year) ($40\ ft^3/hr$)($\$0.012/ft^3$)		\$ 3,065
	-- Single-crystal seed/furnace-year @ \$150 ea. (1/15 seed/cycle) (473 cycle/year) (\$150 ea.)		\$ 4,730
	-- Miscellaneous/furnace-year, including graphite-component usage (473 cycle/year)(\$15/cycle)		\$ 7,095
		MATS	\$ 398,777
5.	Direct Process Utilities Usage		
	-- Electricity/furnace-year @ \$0.022/kw-hr [(1.5 hr/cycle)(80 kw) + (10.5 hr/cycle)(60 kw)] (473 cycle/year)(\$0.022/kw-hr)		\$ 7,805
		UTIL	\$ 7,805
6.	Annual Production		
	-- (20 kg/cycle)(473 cycle/year) (0.55)		5,203 kg
		QUAN	5,203 kg

TABLE I (continued)

IPEG Price

$$\text{Price} = [(C_1*EQPT) + (C_2*SQFT) + (C_3*DLAB) + (C_4*MATS) + (C_5*UTIL)]/QUAN$$

where C_1 = \$0.49/year per dollar

C_2 = \$97/year per ft^2

C_3 = \$2.1/year per dollar

C_4 = \$1.3/year per dollar

C_5 = \$1.3/year per dollar

In 1978 dollars,

price = \$618,239/5,203 kg

= \$ 119 per kg

In 1975 dollars,

price = \$ 99 per kg

This price is comprised of two major components: the direct poly cost and the Czochralski add-on cost, the latter including the overhead applied to the poly cost. Further, if the crystal mass is converted to sheet area, this price can be combined with the 1982 wafer price goal allocation to yield the margin available for the wafering add-on cost.

Using the 1980 slicing yield goal of 1.00 m^2 per kg, and expressing all costs in 1975 dollars,

1982 standardized price, PWAFERS	\$128 per m^2
direct poly cost	\$ 46 per m^2
Czochralski add-on cost	\$ 53 per m^2
margin available for wafering add-on cost	\$ 29 per m^2

TABLE II

FIVE CRYSTALS (100 kg) GROWN FROM ONE 12 INCH DIAMETER CRUCIBLE

Description of Czochralski Growth Process

A. Facilities and Operation

Same as Table I with the following exception:

-- Cost of Czochralski furnace: $150,000

B. Process

Same as Table I with the following exception:

-- Mass left in crucible: 4 kg (crystals 1-4); 1 kg (crystal 5)

C. Cycle Time

Crystal 1:

-- Clean furnace	0.50 hr.
-- Load and melt charge (20 kg)	2.50 hr.
-- Seed; grow neck, crown, and shoulder	1.00 hr.
-- Grow body of crystal (15 kg)	7.50 hr.
-- Grow taper (1 kg)	0.50 hr.
-- Cool and remove crystal during recharge	- -

Crystal 2:

-- Recharge (21½ kg)	1.50 hr.
-- Seed; grow neck, crown, and shoulder	1.00 hr.
-- Grow body of crystal (20½ kg)	10.25 hr.
-- Grow taper (1 kg)	0.50 hr.
-- Cool and remove crystal during recharge	- -

TABLE 'I (continued)

Crystal 3:		
Same as crystal 2		13.25 hr.
Crystal 4:		
Same as crystal 3		13.25 hr
Crystal 5:		
-- Recharge (21½ kg)		1.50 hr.
-- Seed; grow neck, crown, and shoulder		1.00 hr.
-- Grow body of crystal (23½ kg)		11.75 hr.
-- Grow taper (1 kg)		0.50 hr.
-- Cool and remove crystal		1.50 hr.
Maintain and repair furnace		12.00 hr.
		80.00 hr.

Input Data

1.	Direct Equipment Initial Capital Cost		
	-- Czochralski furnace		$ 150,000
		EQPT	$ 150,000
2.	Direct Manufacturing Floor Space		
	-- Czochralski furnace		100 ft^2
		SQFT	100 ft^2
3.	Annual Direct Labor Salaries		
	-- Operator/furnace-year (8/3 operator-hr/furnace-shift)(3 shift/day) (345 day/year) ($4.50/hr)		$ 12,420
		DLAB	$ 12,420

TABLE II (continued)

4. Directly Used Materials and Supplies	
-- Poly charge/furnace-year @ \$30.08/kg* (106 kg/cycle) (103.5 cycle/year) (\$30.08/kg)	\$ 330,008
-- Crucible/furnace-year @ \$210 ea. (1 crucible/cycle) (103.5 cycle/year) (\$210 ea.)	\$ 21,735
-- Argon/furnace-year, @ \$0.012/ft^3 (66.5 hr/cycle) (103.5 cycle/year) (40 ft^3/hr) (\$0.012/ft^3)	\$ 3,304
-- Single-crystal seed/furnace-year @ \$150 ea. (1/3 seed/cycle) (103.5 cycle/year) (\$150 ea.)	\$ 5,175
-- Miscellaneous/furnace-year, including graphite-component usage (103.5 cycle/year)(\$75/cycle)	\$ 7,763
MATS	\$ 367,985
5. Direct Process Utilities Usage	
-- Electricity/furnace-year, @\$0.022/kw-hr [(7.5 hr/cycle)(80 kw) + (57.5 hr/cycle) (60 kw)] (103.5 cycle/year)(\$0.022/kw-hr)	\$ 9,222
UTIL	\$ 9,222
6. Annual Production	
-- (106 kg/cycle)(103.5 cycle/year) (0.55)	6,034 kg
QUAN	6,034 kg