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LSSA LARGE AREA SILICON SHEET TASK: CONTINUOUS CZOCHRALSKI
PROCESS DEVELOPMENT

Quarterly Report No. 2, January—March 1978

Texas Instruments Report No. 03-78-11

By
Samuel N. Rea

April 1978

Work Performed Under Contract No. NAS-7-100-954887

Texas Instruments Incorporated
Dallas, Texas

MASTER

U.S. Department of Energy

Solar Energy

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Samuel N. Rea
April 1978

JPL Contract No. 954887

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ABSTRACT

A commercial puller having a 12-kg main crucible capacity is being modified extensively to permit installation of an auxiliary crucible with attendant silicon storage and feed system. The various puller modifications have been designed and are currently being fabricated. All major externally purchased parts such as a metallurgical vacuum valve, auxiliary power supply, and silicon feed system are on order. Puller assembly and checkout will be accomplished during the second quarter of this year.

Several experiments have been run in existing equipment examining suitable configurations of auxiliary melters. Heating of the auxiliary melt can be accomplished fairly easily using graphite resistance heaters. Power inputs around 4.5 kVA have proven sufficient to maintain 700 g melts. A major problem uncovered in these experiments is getting the molten silicon to flow properly from the quartz crucibles. Also, thin quartz crucibles tend to devitrify excessively, limiting the run time. These problems can be circumvented by using SiC-coated graphite crucibles, but primary melt contamination may be a problem with graphite crucibles.

Economics of a continuous Czochralski process with 100 kg furnace runs of 10-cm diameter crystal indicate a crystal cost in the \$24.00/m² range exclusive of polysilicon and slicing costs.

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CONTINUOUS CZOCHRALSKI PROCESS DEVELOPMENT

Texas Instruments Incorporated

Quarterly Report
January - March, 1978

I. INTRODUCTION

This program is to demonstrate feasibility of a continuous Czochralski process employing liquid silicon melt replenishment during growth. An incoming flow of solid granular or nugget polysilicon will be premelted in a small auxiliary crucible from which liquid silicon will be introduced into the main or primary crucible. Liquid melt addition is being pursued since this replenishment technique should cause minimal thermal and mechanical disturbances to the primary melt thereby allowing high crystal productivity.

Figure 1 illustrates the continuous crystal growth machine being developed at Texas Instruments. A commercial puller is being modified extensively to permit continuous silicon feed. Modifications include a new lower chamber, addition of a high-temperature vacuum valve, and a modified upper chamber to accept a Hamco cable seed pull mechanism equipped with crystal weight readout. In addition, the polysilicon storage and feed components depicted in Figure 1 are being constructed.

A careful assessment of costs associated with this continuous Czochralski process is presented below based on the IPEG option of SAMICS. The economic model indicates a 1982 Czochralski wafer cost around \$90/m² compared with the SAMICS goal allocation of \$128/m². Crystal cost, expressed in equivalent sheet area, exclusive of polysilicon and slicing costs is in the range \$24-30/m².

II. TECHNICAL DISCUSSION

The technical discussion will roughly parallel the milestone chart shown in Figure 2. As a perusal of that figure indicates, approximately one-third of the program consists of equipment design, one-third in construction, and the rest in demonstration runs and miscellaneous activities such as auxiliary melter testing and economic modeling. As of this report

date, the program is approximately 40% complete with major emphasis on equipment modification, design, and construction. A Varian 2848 A furnace is being used as the basic building block of the continuous puller. The standard 12-kg (10-inch) hot zone will be utilized.

A. Design, Test and Construction

1. Lower Dome

A new lower chamber was designed which will accept the vacuum valve, auger feed, and auxiliary melter. A flat-top chamber was designed in order to minimize overall puller height. The chamber is double-wall construction with water cooling. A 25-cm (10-inch) diameter side appendage 13 cm (5 inches) long is integral with the lower chamber to provide room for the auger feed and auxiliary melter. The chamber assembly is designed, detailed, and currently being fabricated in the TI shops.

2. Upper Chamber

The existing upper chamber will be utilized with a few modifications. Transition sections were designed and built to mate the vacuum valve and to attach the Hamco cable pull mechanism. Other minor modifications were effected in order to use the existing chamber lift mechanism.

3. Liquid Feed System

The key technical challenge in this approach to a continuous puller is designing a suitable auxiliary crucible which will supply the liquid feed to the primary crucible. By premelting the silicon in a small crucible the auxiliary power requirements can be minimized and crucible/heater costs can be held to a minimum. To maintain a constant melt level in the primary crucible while growing a 10-cm crystal at 10 cm/h, a 1838 g/h flow of silicon into the crucible is required. The power requirement to effect melting of this flow starting from room temperature is around 1.5 kW. Experimental work to date with a small RH power supply has indicated 5-6 kVA is required for the auxiliary power supply. For adequate safety margin, a 9 kVA supply has been ordered.

A wide variety of melter configurations have been tested. This experimental work has been done in a small reactor chamber 24 cm ID by 46 cm long under a 1-atm argon ambient. Best results to date using separate crucibles and heaters have been achieved with the heater shown in Figure 3. This graphite heater is 5 cm ID by 16 cm long with electrodes attached to the flanges. Silicon fines are fed into an inner quartz crucible through the top opening in the heater.

Efforts to utilize small fused silica crucibles have been unsuccessful due to nonwetting of the quartz by the silicon. This causes the silicon to ball up and not flow out of the crucible in a continuous stream. Instead, the silicon exists the crucible in globules which would cause extreme melt agitation in the primary crucible. In addition, the quartz softens appreciably at the elevated temperatures required to effect flow from the crucible and these high temperatures result in extreme devitrification.

To overcome these problems, several crucibles were constructed of graphite coated with silicon carbide. These crucibles were successful in that molten silicon wets silicon carbide and flow from the SiC-coated crucibles more nearly resembled a steady stream although some globular chunks still were evident.

The encouraging results of the SiC-coated crucibles led one step further to an integral heater/crucible depicted in Figure 4. This arrangement is made quite easily by gluing graphite end plates to a heater like that in Figure 3 using Dylon GC graphite cement. A graphite exit nozzle is screwed into a threaded hole drilled through the bottom of the heater and the entire assembly is SiC-coated.

Results using integral crucibles are very encouraging. The intimate contact between silicon and heater provides rapid, efficient melting and the exit flow exhibits fewer globules than the other approaches. Of course, a number of problems are obvious; the primary one being contamination of the silicon by carbon. Also, a heater will probably be consumed in each run but the design is simple enough so that this should not be a big cost factor. Work along this line is continuing with some additional heater designs being fabricated. In particular, the end piece gluing operation mentioned above is rather crude and may be circumvented by test-tube shaped crucibles mounted at an angle toward the primary melt.

4. Hot Zone

A careful assessment of the heater, heat shield performance, and internal furnace space allowable indicated that the present Varian 2848 A design should be adequate.

5. Melt Level Control

The Hamco cable pull seed lift will have crystal weight readout. By calibrating the auger feed rate it should be possible to achieve adequate melt level control by manual adjustment of the auger speed. Thus, no active feedback control is envisioned at present although this could be effected once the basic puller concept is proven.

6. Silicon Hopper

A 100-kg hopper having conical exit and vibration feed is completed.

7. Auger Feed

The Thomas Conveyor Company of Fort Worth, Texas, designed and is constructing the auger feed. Their original delivery date has slipped approximately two months but this is not a pacing item. A variable speed 3/4 hp motor to drive the auger has been purchased and received.

8. Miscellany

The Hamco seed lift mechanism has been received. The VAT vacuum valve is due in early April. Thus, barring unforeseen delays, the schedule of Figure 2 still holds.

B. Economic Analysis

The IPEG option of SAMICS was used to estimate costs of the continuous Czochralski process under development. Fairly conservative assumptions were made regarding labor, supplies costs, and yields. A 100-kg furnace run results in 544 cm of 10-cm diameter crystal. For the process under development, it is assumed that seven crystals, each of 78 cm length, will be grown in one run for a total of 546 cm crystal at 100% yield. A summary of the basic model assumption is given below with backup information in the Appendix.

1. Basic Assumptions

- a. 100-kg furnace run
- b. 10-cm diameter crystal pulled at 10 cm/h
- c. Equipment cost is \$121,000
- d. Equipment utilization is 83%
- e. Furnace floor space is 150 ft²
- f. Run cycle time is 78.6 h
- g. Two pullers per operator
- h. Direct labor costs \$5.00/h
- i. Operating supplies are \$516/furnace run
- j. Power per run is 3800 kW-h at \$0.03/kW-h
- k. 100% yield/run is 85.77 m² in slice equivalent area based on 20 slices/cm crystal
- l. Multiblade slicing cost is \$27/m² at 95% saw yield

The above cost assumptions are in 1978 dollars. Later these costs will be adjusted back to 1975 dollars per the IPEG inflation factors in order to remain compatible with the 1982 cost goals.

2. IPEG Inputs

a. Furnace Runs/Year

With the assumed 83% utilization factor applied to a 24 h day, 364 day year the furnace runs/yr are:

$$\text{Runs/year} = (0.83)(24)(364)/78.6 = 92.6$$

b. Direct Labor

$$\text{Labor/run} = (78.6 \text{ h})(\$5.00/\text{h})/2 = \$196.50$$

$$\text{Direct labor/year} = (196.50)(92.6) = \$18,196$$

c. Operating Materials

From the Appendix the materials costs/run are \$515.92.

The annual materials costs are:

$$\text{MATS} = (\$515.92)(92.6) = \$47,774.19$$

d. Utilities

The only utilities cost of consequence is the electrical power per run assumed at \$0.03/kW-h.

$$\text{Cost/run} = (3800 \text{ kW-h})(\$0.03) = \$114$$

$$\text{Utilities/year} = (\$114)(92.6) = \$10556.40$$

e. Crystal Output

At 100% yield, 546 cm crystal will be produced, which, at 20 slices/cm yields 85.765 m^2 per furnace run. Then,

$$\text{Output/year} = (85.765)(92.6) = 7941.88 \text{ m}^2/\text{yr}$$

3. IPEG Cost

The IPEG cost model uses the equation:

$$\text{Cost} = (0.49 * \text{EQPT} + 97 * \text{SQFT} + 2.1 * \text{DLAB} + 1.3 * \text{MATS} + 1.3 * \text{UTIL}) / \text{QUAN}$$

where

EQPT = equipment cost = \$121,000
SOFT = space requirements per furnace = 150 ft²
DLAB = annual direct labor = \$18,196
MATS = annual materials cost = \$47,774
UTIL = annual utilities cost = \$10,556
QUAN = annual output = 7941.88 m²

With these parameters the IPEG cost is \$23.65/m². Deflating this cost back to 1975 dollars using the 1.203 inflation factor gives a 1975 equivalent cost of \$19.66/m² at 100% crystal yield.

4. Total Crystal Cost

Polysilicon expense can be added easily to the above crystal processing cost using 104.58 kg/run and the same slice equivalent area. Total crystal costs are shown in the table below for unburdened polysilicon in 1975 dollars.

<u>Poly Cost</u>	<u>1975 Crystal Cost-100% yield</u>
\$0/kg	\$19.66/m ²
10	31.85
25	50.14
60	92.82

Figure 5 shows the above crystal costs as a function of yield. On a grams in - grams out basis a crystal yield of 80% or better should be feasible in a continuous process.

5. Total Wafer Cost

Crystal slicing costs must be added to the above to arrive at the final slice value. Based on past experience with multiblade sawing¹, a 1975 IPEG sawing cost in the neighborhood of \$27/m² appears feasible. When this value is added to the various crystal costs, total wafer costs represented in Figure 6 are obtained. With an 80% crystal yield a wafer cost around \$90/m² is indicated which is comfortably below the JPL 1982 goal of \$128/m². Since there is no profit included in the \$90/m² figure the possibility exists of showing a profit on wafers selling at \$128/m².

The 1986 target of \$18.20/m² is considerably more formidable. Even at the 1986 goal of \$10/kg for polysilicon, a wafer cost of \$65/m² is indicated. In fact, polysilicon alone accounts for \$15.20/m² of the wafer cost under the present assumptions leaving only \$3/m² for all other processing at the 1986 price.

6. Cost Sensitivity

Figure 7 graphically shows the cost elements for the continuous Czochralski process at the 1982 polysilicon goal of \$25/kg. Excluding polysilicon for the moment, the total wafer costs are fairly evenly distributed among equipment, labor, materials, and space/utilities. In fact, equipment, labor, and materials are equally divided between crystal growth and slicing. Thus, a frontal attack on all cost elements is justified with crystal slicing as important as crystal growth.

Of the various materials items, argon stands out as a major target for cost control. From past experience, argon runs about \$3/operating hour and in Figure 7 accounts for \$3.40/m². Clearly, argon usage will be monitored closely and efforts made to minimize its consumption.

All the modeling results presented depend heavily on the underlying assumptions. One key assumption employed here is that 20 slices/cm are produced. With 10-cm crystal this translates into 0.855 m²/kg crystal which is below the 1.0 m²/kg targeted for 1980². The wafering costs in Figure 7 assume a moderate extension of current multiblade sawing technology. Machines are under development to slice 1000 slices per run³ which could lower sawing costs by 30-40%. This greater sawing capacity coupled with the 1.0 m²/kg target should lower wafering costs included in Figure 6 and 7 by 50% as well as improve the polysilicon area yield and crystal growth costs. All these advances combined should put continuous Czochralski within shooting distance of the 1986 cost goals.

C. Additional Considerations

1. Continuous Czochralski Doping

An analysis of dopant impurity levels in a continuous Czochralski process indicates that the crystal resistivity can be held constant during growth. Details of the analysis are omitted but the general result for crystal resistivity under continuous growth is:

$$\rho/\rho_i = \frac{k C_{li}}{C_r - (C_r - k C_{li}) \exp(-k V_s / V_o)} \quad (1)$$

where

C_{1i} = initial melt dopant concentration
 C_r = incoming melt replenishment concentration
 V_s = crystal volume grown
 V_o = primary crucible melt volume
 k = dopant segregation coefficient
 ρ_i = initial crystal resistivity, $V_s = 0$.

Equation (1) indicates that, without special precaution, the crystal resistivity will continually decrease as more crystal is grown due to the exponential term in the denominator. However, if $C_r = k C_{1i}$, the exponential term drops out and the resistivity will remain constant, independent of crystal volume, V_s , grown. Thus, the incoming feed stream should be doped to a level $k C_{1i}$ in order to maintain constant axial crystal resistivity profiles.

In the case of minor impurities inherent in the polysilicon (Al, Cu, Ti, etc), $C_r = C_{1i}$ and the reciprocal of equation (1) reduces to the result given in reference 4.

2. Crystal Pull Rate

The crystal growth model developed in reference 1 was utilized to model 10-cm growth. Figure 8 shows theoretical maximum pull rates for continuous versus single charge pulls from 12-kg crucible melts. At the longer crystal lengths, continuous growth provides a 25% pull rate advantage over batch growth. The indicated 19 cm/h continuous pull rate offers a comfortable margin over the 10 cm/h goal of this program.

III. CONCLUSIONS AND RECOMMENDATIONS

The program thus far has been primarily in a design and construction phase and is on schedule with the exception of a few minor areas. Development of a suitable auxiliary crucible still is the major technical obstacle.

An IPEG analysis of the economics of continuous Czochralski indicates the 1982 price goal of \$128/m² can be met. However, achieving the 1986 goal will require major breakthroughs, especially in the slicing area to achieve a maximum number of slices per ingot.

IV. NEW TECHNOLOGY

No new technology has been developed on this program during this quarter.

V. PLANS FOR NEXT QUARTER

No significant deviation from the program plan in Figure 2 is anticipated. Particular events scheduled include:

1. Fabricate the lower chamber assembly.
2. Assemble the modified puller.
3. Continue development of the feed melter.
4. Update the economic model as necessary.
5. Conduct a design review with JPL.

VI. PROGRAM COST

During the latest quarter 1075 man hours were expended and a total cost including fixed fee of \$75,367 was incurred. To date, through March 31, 1978, cumulative man hours are 2330 and total cost including fee is \$118,847. Figure 9 shows actual versus planned labor and Figure 10 depicts program costs to date.

VII. REFERENCES

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4. R.H. Hopkins, et al., "Silicon Materials Task of the Low Cost Solar Array Project (Part 2), " Fifth Quarterly Report and Summary, ERDA/JPL-954331-77/1, December 1976, p. 78.

FIGURE I. CONTINUOUS CZOCHRALSKI SILICON FURNACE

Figure 2. Program Plan

Figure 3. Auxiliary Crucible Heater: Split-Tube Design

Figure 4. Integral Auxiliary Heater/Crucible

Figure 5. SAMICS/IPEG Continuous Czochralski Crystal Cost Expressed in Equivalent Slice Area

Figure 6. SAMICS/IPEG Cost Projections of a Continuous Czochralski Wafer Process

Figure 7. SAMICS/IPEG Cost Breakdown of a Continuous Czochralski Wafer Process

Figure 8. Theoretical Maximum Pull Rates of Continuous Versus Single Charge Czochralski Silicon Crystal Growth

Figure 9. Program Labor Hours

Figure 10. Total Program Cost

APPENDIX

ECONOMIC MODEL BACKUP

The attached pages detail pertinent assumptions and costs associated with the continuous process under development. These data were utilized in the IPEG model presented in this report.

CONTINUOUS CZOCHRALSKI ECONOMIC MODEL

CRYSTAL GROWTH EQUIPMENT COST

1.	Basic Furnace	\$100,000
2.	Auxiliary Melter/Power Supply	3,000
3.	Silicon Hopper/Feed System	4,000
4.	Melt Level Control	5,000
5.	Vacuum Valve	4,000
6.	Contingency	5,000
	Total	\$121,000

CONTINUOUS CZOCHRALSKI ECONOMIC MODEL

CYCLE TIME

<u>Event</u>	<u>Time</u>	
1. Cleanup	0.5 h	
2. Load and Melt (12 kg)	1.5	
3. Seed and Top	1.0	
4. Growth (14.34 kg)	7.8	78 cm
5. Taper (10.6 kg)	1.0	
6. Unload	1.0	
7. Repeat - 6 Crystals	64.8	468 cm
8. Cooldown and Unload	1.0	
Totals:	78.6 h	546 cm
Polysilicon Charged:	104.6 kg	
Power Consumption:	3800 kW-h	

CONTINUOUS CZOCHRALSKI ECONOMIC MODEL

CRYSTAL GROWTH OPERATING SUPPLIES COSTS

	Item	Cost/Run
1.	Crucible Shaft	\$ 18.00
2.	Graphite Crucible	32.00
3.	Quartz Liner	100.00
4.	Graphite Shaft Parts	9.00
5.	Graphite Heater	16.67
6.	Misc. Graphite Heater Parts	3.75
7.	Graphite Heat Shield	20.00
8.	Graphite Felt Insulation	3.00
9.	Outer Stainless Heat Shield	4.00
10.	Misc. Heat Shield Parts	6.00
11.	Shaft Seals	1.00
12.	Auxiliary Heater	12.50
13.	Auxiliary Crucible	10.00
14.	Crystal Seeds	35.00
15.	Vacuum Pump Filter	9.00
16.	Vacuum Pump Oil	3.00
17.	Argon	<u>233.00</u>
	Total	\$515.92/run