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CONTINUOUS CZOCHRALSKI GROWTH

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

Third Quarterly Progress Report, April 1—June 30, 1978

Work Performed Under Contract No. NAS-7-100-954888

Kayex Corporation
Rochester, New York

MASTER

U.S. Department of Energy

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Solar Energy

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CONTINUOUS CZOCHRALSKI GROWTH

SILICON SHEET GROWTH DEVELOPMENT
OF THE LARGE AREA SILICON SHEET TASK
OF THE LOW COST SILICON SOLAR ARRAY PROJECT

THIRD QUARTERLY PROGRESS REPORT
APRIL 1 - JUNE 30, 1978
PROGRAM MANAGER: DR. R. L. LANE

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ABSTRACT

The continuous Czochralski (CZ) equipment is to be designed and constructed as one portion of this program. Concurrently, methods and process parameters will be developed for continuous CZ in the second portion of the effort. The goal is to demonstrate the growth of 100 kilograms of single crystal material using only one crucible. The approach is to suitably modify a Hamco CG2000 crystal grower to demonstrate that continuous CZ is feasible by periodic melt replenishment (recharging) of 12-inch (and later 14-inch) diameter crucibles.

The program plan for equipment design and construction is on schedule after some delays in the completion of the polyweight/recharge system, which has now been fully constructed and installed on the crystal grower. The grower was disassembled to make modifications to the chambers to enable the recharge system to be installed. Other equipment modifications, i.e., melt level control via weight, 14-inch hot zone, and a powder/lump recharge system are in process and are expected to be completed on schedule.

The process development program is also on schedule. Two recharges (three melts and ingot growth cycles from the same crucible) have been demonstrated. 42.5 kilograms of single crystal material have been pulled from one crucible by melting a total of 48.7 kilograms in three successive melts, for a cumulative pulled yield of 87%. The 10 cm diameter grown crystal material was approximately 80% of zero dislocation structure.

A SAMICS/IPEG analysis of two types of continuous CZ growth is presented.

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1.0 INTRODUCTION

This attempt to produce 100 kilograms of high quality, single crystal silicon from one melt container (crucible) is to utilize a substantially modified Hamco CG2000 crystal grower as the basis for further process development in the areas of recharging, large crystal growth, fast pull speed, and improved automation. All efforts are geared toward production-type equipment and process in order to achieve the JPL cost goals and demonstrate the feasibility of continuous CZ solar grade crystal production.

The modifications of the Hamco CG2000 crystal grower which have been completed and installed include (1) a special pull chamber for storing a supply of polysilicon, (2) a vacuum-tight isolation valve to permit replenishment of the melt and retrieval of grown crystals without contamination, and (3) a polyweight/recharge device as a means of feeding poly rod material into the crucible for melt replenishment (Figure 1).

In order to allow crystal growth and melt replenishment to be performed in a simulated production mode, a crystal/poly transfer device is used to safely handle hot crystals and transfer them to a cooling rack. The transfer device is also used as a convenient way to load the crystal grower with polysilicon (Figure 2).

Equipment modifications which are in process and scheduled for later completion include (1) melt level control via weight which utilizes the crystal weight signal from the cable/pull mechanism to control the crucible lift motor, (2) 14-inch crucible and hot zone, and (3) recharge device for powder or lump silicon.

FIGURE 1
2

FIGURE 2
CRYSTAL/POLY TRANSFER DEVICE

2.0 PROGRESS

2.1.0 GROWTH FACILITY CONSTRUCTION AND MODIFICATION

2.1.1 POLY WEIGHT/RECHARGE SYSTEM

The major effort during this reporting period involved the poly weight/recharge system completion and its installation on the grower. Since continued delays were experienced in the construction and testing of the device, all recharge runs to date have been conducted by placing the poly rod on the seed-lift cable.

During the latter portion of the quarter, the grower was disassembled and adapted with the recharge mechanism.

The modification required that a slot be machined in the top of the pull chamber to allow the recharge cable to be moved backward and forward. Additionally, a chamber overpressure relief valve had to be relocated, as the recharge mechanism covers up the original valve.

The controls for the poly weight/recharge mechanism allow the operator to manually move the poly rod horizontally (hydraulic drive) and vertically (DC motor) either at high speed or at a controlled rate. A DC-PM motor with a tachometer feedback system is used to control the melt-off rate. This is quite critical to the process. The rate of lowering is measured with a digital meter calibrated in inches per hour.

A torque-sensing strain gauge is used to measure the weight of the poly rod. This is calibrated in kilograms and reads to the nearest 0.01 kg. The poly rods presently being used weigh about 20 kg. In actual practice, the weight system is set at the desired melt-off amount, and when the indicated weight reaches zero, the melt-off is terminated by stopping the motor. After about 5 minutes to allow the rod to melt to a

flat bottom, an accurate reading is obtained and the rod is then retracted for crystal growth. Figure 3 shows the JPL growth facility with all current modifications and features.

2.1.2 MELT-LEVEL CONTROL VIA WEIGHT

The desirability of good control of the location of the growth front of the crystal (solid-liquid interface) is well established. Since a CZ crystal grower is designed to have high thermal gradients, both axially and radially, lack of control of the location of the growth front, either during a crystal run or from run to run, will affect crystal properties and growth conditions greatly, resulting many times in unpredictable and undesirable results.

The location of growth front is determined primarily by the location of the surface of the liquid, or the melt level as it is usually called. The growth front is located slightly above the melt level and the liquid is lifted up against the growth front by surface tension.

Usually the melt level is held constant by lifting the crucible at a rate which is proportional to the seed lift rate. The most probable error in this method is caused by errors or deviations in the desired crystal diameter. Thus, if the crucible lift is calculated for a 10 cm diameter crystal and the crystal is too large, the melt level will progress downward during the run and a cumulative error will result. Likewise, if the diameter control system allows the diameter of the crystal to change during growth, again an error in melt level will occur.

Generation of a crystal weight signal during crystal growth makes it possible to obtain better control of the growth process, as the method eliminates melt level errors due to crystal diameter errors. The effect is shown in the following equations:

FIGURE 3

JPL CONTINUOUS CZ GROWTH FACILITY

For a cylindrical crystal and a cylindrical crucible having radii r_s and r_1 respectively, the weight of the crystal must equal the weight (mass) of liquid withdrawn from the crucible:

$$\text{Weight crystal} = \text{weight liquid depleted} \quad (1)$$

$$\pi r_s^2 h_s \rho_s = \pi r_1^2 h_1 \rho_1 \quad (2)$$

in which h_s = crystal length

h_1 = liquid level change (or
required crucible movement up)

ρ_s = density of solid silicon (2.33 g/cc)

ρ_1 = density of liquid silicon (2.54 g/cc)

thus, the distance the crucible must lift for a crystal of radius r_s and length h_s is

$$h_1 = \frac{r_s^2}{r_1^2} \frac{\rho_s}{\rho_1} h_s \quad (3)$$

or the required velocity of crucible lift is:

$$\frac{dh_1}{dt} = \frac{r_s^2}{r_1^2} \frac{\rho_s}{\rho_1} \frac{dh_s}{dt} \quad (4)$$

in which $\frac{dh_1}{dt}$ = crucible lift rate

$\frac{dh_s}{dt}$ = crystal seed lift rate

Thus, the required crucible lift rate is affected by both the seed lift rate and the crystal diameter:

$$\frac{dh_1}{dt} = k_1 r_s^2 \frac{dh_s}{dt} \quad (5)$$

$$\text{in which } k_1 = \frac{\rho_s}{\rho_1 r_1^2} = \text{constant}$$

For a perfectly cylindrical crystal and crucible, the crucible lift rate is proportional to seed lift rate and to the square of crystal diameter. Thus, slight errors in crystal diameter can indeed cause problems. Furthermore, the top of the crystal is typically a cone of uncontrolled volume and another error is introduced at the beginning of the run because of this.

Control of melt level or crucible lift rate by use of the crystal weight signal results in more accurate control of the crystal growth front because it eliminates errors due to errors in crystal diameter. Again, the weight of the crystal must equal the weight of the liquid depleted.

$$\text{weight (mass) of crystal} = \text{weight (mass) of liquid depleted} \quad (6)$$

$$W_s = \pi r_1^2 h_1 \rho_1 \quad (7)$$

At any given time during the crystal growth, the required crucible total travel is:

$$h_1 = \frac{W_s}{\pi r_1^2 \rho_1} = k_2 W_s \quad (8)$$

$$\text{where } k_2 = \frac{1}{\pi r_1^2 \rho_1} = \text{constant.}$$

Thus, the required crucible travel is proportional only to the crystal weight and completely independent of the crystal diameter, and therefore improved control of the crystal growth process results.

In practice, the system is initially calibrated to provide a given crucible travel for a given weight according to the equation (8). An electrical control system generates a corrective error signal if the crucible position does not correspond to the crystal weight. The error signal activates the crucible lift motor and brings it back to the proper position in the furnace.

Thus, in a typical crystal growth run, the crucible moves at a smooth, generally uniform rate depending upon the rate of increase of the crystal weight.

A further advantage of this system is that no adjustments to the controls or calibrations are required if different size crystals are to be grown.

The electrical control circuitry is being constructed to allow for this method of melt level control in the future. The accuracy of the weight readout system of the cable pull mechanism (± 100 g) indicates that the crucible position can be controlled to within 2 mm with a 12-inch diameter crucible.

2.1.3 14-INCH HOT ZONE

The program plan calls for demonstration of the 14-inch hot zone in October. In deriving the program plan, it was believed that the 14-inch hot zone could be retrofitted to the JPL crystal grower without major changes to the chamber. After a more careful study of the problem, it was concluded that the size of the chamber was insufficient to safely accommodate the

hot zone, especially in the case of a molten silicon spill. Since the 14-inch crucible may hold up to 40-45 kg, a considerable volume is required to contain it without damage to the chambers. The net result is that a furnace chamber seven inches higher is required. Since the project cost estimate did not include funding for a new furnace tank, and a 7-inch tank extension would cost almost as much as a new tank, the Company decided to purchase a new replacement tank for the JPL grower using Kayex funds. The design is completed and delivery is expected in late July.

The 14-inch hot zone is considered to be very important in meeting JPL cost goals as it makes the growth of larger diameter crystal economically more attractive and results in a significant reduction in the crystal growth add-on cost.

2.2.0 PROCESS DEVELOPMENT

2.2.1 HOT ZONE MODIFICATIONS

Attempts to fill 12-inch crucibles to a capacity larger than 18-20 kg, although successful as reported earlier, put an excessive load on the upper crucible support. This support is a cylinder of graphite resting on the lower crucible support which conforms to the bottom radius of the crucible. The upper support is slotted vertically to take advantage of the elasticity of the graphite during cool-down to prevent breakage of the parts which is certain to occur without the slot.

The forces on the crucible during the run, combined with the high temperature, cause a continuous and permanent spreading effect on the upper crucible support so that after a small number of 25 kg melts, the support spreads sufficiently to allow the crucible to bulge out, and it must therefore be discarded. The upper support was redesigned with a graphite plate across the

slot to provide support for the crucible. The plate is pinned with graphite pins to support the crucible during the run. During cool-down, the pins shear to relieve stresses, thereby preventing graphite breakage.

2.2.2 RECHARGE RUNS

Two recharge runs were performed during the reporting period: a two-melt run and a three-melt run. Summary data from these runs is presented in Tables 1 and 2.

Run No. 9, the two-melt run, was made by starting with a standard 18 kg melt with growth of a 14 kg crystal. During the recharge cycle, the crucible was replenished with 13.2 kg, along with the suitable amount of dopant, and a second crystal was started. This crystal lost zero dislocation structure after 6.3 kg had been grown and another crystal was grown from the remaining melt. Cumulative pulled yield for the run was 86.9%.

Run No. 11 was the first attempt at a three-melt run utilizing a basic 18 kg melt and two recharge cycles. Two excellent 10 cm diameter crystals were grown from the first two melts, each weighing about 15 kg. Structure loss problems occurred after the second recharge cycle so that two crystals were pulled from the third melt, each being dislocated after about 6 kg had been grown.

A summary of the data from run Nos. 9 and 11 is included in Tables 1 and 2.

Process problems, primarily the loss of zero-dislocation structure, appeared to result from excessive silicon monoxide build-up around one view port. This situation occurred severely in run No. 9 and to a lesser extent in run No. 11. Gas flows in the chamber were adjusted slightly between the two runs, resulting in the improvement. Not to be ruled out, however, is the possibility of a small water leak in the water-cooled chamber near the problem port. This is being checked before further runs are made.

<u>CRYSTAL NO.</u>	<u>MELTED WEIGHT (kg)</u>	<u>PULLED WEIGHT (kg)</u>	<u>CUMULATIVE MELTED WEIGHT (kg)</u>	<u>CUMULATIVE PULLED WEIGHT (kg)</u>	<u>YIELD %</u>	<u>CUMULATIVE YIELD %</u>
1	18.0	14.0	18.0	14.0	77.8%	77.8%
2*	13.2	13.1	31.2	27.1	76.2%	86.9%

*Two crystals were grown from this melt, due to loss of zero dislocation structure.

Crucible diameter x height 12" x 9" (30.5 cm x 22.9 cm)

Crystal diameter 4.4" (11.2 cm)

Total run time 39 hours

Average growth rate 3.42"/hr (8.69 cm/hr)

Throughput 0.695 kg/hr

SUMMARY OF TWO-MELT RUN (NO. 9)

TABLE 1

<u>CRYSTAL NO.</u>	<u>MELTED WEIGHT (kg)</u>	<u>PULLED WEIGHT (kg)</u>	<u>CUMULATIVE MELTED WEIGHT (kg)</u>	<u>CUMULATIVE PULLED WEIGHT (kg)</u>	<u>YIELD %</u>	<u>CUMULATIVE YIELD %</u>
1	18	14.91	18.0	14.91	82.8	82.8
2	15	15.38	33.0	30.29	85.4	91.8
3*	15	12.25	48.7	42.54	68.0	87.3

*Two crystals were grown from this melt, due to loss of zero-dislocation structure.
The crystals were approximately 65% dislocation-free.

Crucible diameter x height 12" x 9" (30.5 cm x 22.9 cm)
 Crystal diameter 4.2" (10.7 cm)
 Total run time 44 hours
 Average growth rate 3.6"/hr (9.1 cm/hr) (1.9 kg/hr)
 Throughput, average 0.97 kg/hr

SUMMARY OF THREE-MELT RUN (NO.11)

TABLE 2

2.2.3 CRUCIBLE DEVITRIFICATION

Of some concern is the crystallization of the vitreous silica crucible as a result of the high temperatures to which the crucible is exposed.

Figure 4 is the crucible after a typical recharge run. The white layer is the devitrified material which tends to flake off from the glassy silica.

Only the external portion of the crucible is affected, and also only that portion which is in intimate contact with the graphite crucible support. Although much has been written on the devitrification of vitreous silica, it is very difficult at this point to identify the cause as it relates in particular to the CZ process.

Approximately 70 hours of running time are required for 100 kg of 18 cm diameter crystal. If devitrification proceeds linearly with time, we expect only about a 15% loss in crucible wall thickness in 70 hours.

Devitrification will be closely watched in the future as the runs become longer in an attempt to identify the major characteristics of the effect. Uniformity among various crucibles, as well as the effects of graphite material, will be observed.

Time and temperature effects will be noted, as it is anticipated that the major portion of the degradation occurs during the melting or recharge cycle when the crucible is subjected to considerably higher temperatures.

2.3.0 ECONOMIC MODEL

A SAMICS/IPEG economic analysis of one method of continuous CZ growth (CZ No. 1) was presented in the last monthly report⁽¹⁾. The method assumed five 10 cm diameter, 20 kg crystals to be grown from five successive melts in the

(1) Lane, R. "Continuous Czochralski Growth". Second Quarterly Report DOE/JPL 954888 78/2, March, 1978.

FIGURE 4

CRUCIBLE DEVITRIFICATION

White devitrified SiO_2 forms
on outside of the crucible
as a scale.

same 12-inch diameter crucible.

The following is a comparison of the CZ No. 1 method with a potentially lower cost method (CZ No. 2). This method assumes four 12.5 cm diameter, 33 kg crystals to be grown from one 14-inch diameter crucible.

Table 3 gives the assumed conditions for CZ No. 1 and CZ No. 2. Table 4 contains time cycles for the two methods.

The criterion for choosing the conditions for CZ Nos. 1 and 2 was to keep the total run time approximately constant for both methods, i.e., approximately 75 hours. CZ No. 2 produces considerably more crystal in the same run time because the crystal diameter is larger while the growth rate remains the same at 10 cm/hr. The resulting significant increase in throughput is the largest single factor in reducing the CZ add-on cost.

Input data for the SAMICS/IPEG cost calculation are detailed in Table 5. The add-on cost for CZ No. 1 is \$19.40/kg and for CZ No. 2 is \$15.07/kg. All costs are expressed in 1975 dollars. The calculations illustrate the economy of increasing the throughput by an increase in crystal diameter.

CZ GROWTH METHODS

<u>CONDITIONS</u>	<u>CONTINUOUS NO. 1</u>	<u>CONTINUOUS NO. 2</u>
CRUCIBLE SIZE (in.)	12 x 9 h	14 x 11-1/2 h
CRYSTAL DIAMETER (cm)	10	12.5
GROWTH RATE (cm/hr)	10	10
TOTAL PCLY MELTED (kg)	105	143.3
TOTAL CRYSTAL PULLED (kg)	100	134
PULLED YIELD	95	94
USABLE AFTER GRINDING (kg)	87.5	117
GROUND CRYSTAL YIELD (%)	83	81.5
NO. CRYSTALS/CRUCIBLE	5	4

TABLE 3

PROCESS CYCLE TIME

<u>OPERATION</u>	<u>CZ NO. 1 (MIN.)</u>	<u>CZ NO. 2 (MIN.)</u>
1. Preparation	180	240
Load Poly	45	45
Close Furnace	5	5
Pump Down	10	10
Melt	120	180
2. Hot Fill/Growth Cycle	765	895
Position Poly	5	5
Lower Poly	5	5
Melt Poly	60	120
Retract Poly*	5*	5*
Lower Seed*	15*	15*
Stabilize Temperature	40	40
Seed Growth	30	30
Crown Growth	60	60
Straight Growth	525	575
Taper End	40	60
3. Recharge/Growth Cycles	3460 (4 cycles)	3210 (3 cycles)
Cool Crystal	30	45
Remove Crystal	10	10
Position Poly	5	5
Lower Poly	5	5
Melt Poly	120	220
Retract Poly*	5*	5*
Lower Seed*	15*	15*
Stabilize Temperature	40	60
Seed Growth	30	30
Crown Growth	60	60
Straight Growth	525	575
Taper End	40	60
4. Shut Down Cycle	120	160
Cool Furnace	60	80
Remove Crystal**	10**	10**
Clean, Set Up	60	80
Total Time (Min.)	4525	4505
(Hr.)	75.4	75.1

*Completed during stabilization of melt temperature.

**Completed during furnace cooling time.

TABLE 4

SAMICS/IPEG INPUT DATA AND COST CALCULATION
FOR CZ NO. 1 AND CZ NO. 2

Conditions: (Per Cycle)	<u>CONTINUOUS CZ NO. 1</u>	<u>CONTINUOUS CZ NO. 2</u>
Total Si Melted (kg)	105	143.3
Crystal Weight (kg)	20	33.4
No. of Crystals/Crucible	5	4
Diameter of Crystal (cm)	10	12.5
Growth Rate (cm/hr)	10	10
Cycle Time (hrs)	75.4	75.1
Crucible Size	12" x 9" high	14" x 11-1/2" high
Yield (%)	83	81.5
Input Data (1975 \$)		
Capital Equipment Cost (EQPT)	\$124,700	\$124,700
Manufacturing Floor Space (SQFT)	100	100
Annual Direct Labor Salaries		
Prod. Operator (1.33 prsn/yr)	\$ 11,572	\$ 11,572
Elect. Tech. (1.33 prsn/yr)	\$ 1,018	\$ 1,018
Inspector (.1 prsn/yr)	\$ 763	\$ 763
Total (DLAB)	\$ 13,353	\$ 13,353
Direct Used Material and Supplies		
85% Usage per year		
Cycles/yr; hrs/cycle	98.7; 75.4	99.1; 75.1
Poly - kg/yr (charged)	10,363 kg	14,201 kg
Seed (\$4.16/cycle)	\$ 410	\$ 412
Dopant (not costed)	---	---
Argon (100 ft ³ /cycle - hr @ \$0.014/ft ³)	\$ 9,563	\$ 9,563
Crucibles - \$166/12"; \$208/14"	\$ 16,384	\$ 20,613
Miscellaneous (including graphite	\$ 16,753	\$ 18,615
\$2.25/cycle-hr (12"); \$2.50/cycle-hr (14"))		
Material Total (MATS)	\$ 43,110	\$ 49,203
Utilities (Process)		
Electricity (C1032B)	\$ 8,932	\$ 8,932
(50 kw)(\$.025/kw) (cycle time-3 hr)(#cycles)		
Cooling Water (C11280)	\$ 1,912	\$ 1,912
(50 kw)(\$.00528/kw)(cycle time-2 hr)(#cycles)		
Utilities Total (UTII.)	\$ 10,844	\$ 10,844

TABLE 5

	<u>CONTINUOUS CZ NO. 1</u>	<u>CONTINUOUS CZ NO. 2</u>
<u>IPEG PRICE</u>		
C ₁ *EQPT = \$0.489/yr-dollar *EQPT	\$ 60,978	\$ 60,978
C ₂ *SQFT = \$96.9/yr-ft *SQFT	9,690	9,690
C ₃ *DLAB = \$2.133/yr-dollar *DLAB	28,482	28,482
C ₄ *MATS = \$1.255/yr-dollar *MATS	54,103	61,750
C ₅ *UTIL = \$1.255/yr-dollar *UTIL	<u>13,609</u>	<u>13,609</u>
TOTAL	\$166,862	\$174,509
QUAN. (total charge x % yield) (kg)	8,602 kg	11,574 kg
Throughput	1.12 kg/hr	1.56 kg/hr
ADD-ON COST (\$/kg or \$/m ²)	\$19.40	\$15.07
(assume 1 kg → 1 m ²)		

TABLE 5

3.0 PLANS

During the next reporting period, more recharge runs will be attempted. Emphasis will be placed on producing more high quality material from one crucible.

Samples will be sent out for solar cell fabrication and evaluation.

Efforts will continue on the 14-inch hot zone, melt level control, and a method of recharging lump silicon according to the Program Plan (Figure 5a and b).

4.0 COSTS AND LABOR SCHEDULES

The following costs and man hours have been expanded:

	<u>Prior Reported</u> <u>(Oct 77 - Mar 78)</u>	<u>This Quarter</u>	<u>Total</u>
Man Hours	3,480	1,360	4,840
Cost	\$132,089	\$50,342	\$182,431

Figures 6 and 7 compare projected with actual costs and man hours.

5.0 CONCLUSIONS AND DISCUSSION

The modified crystal grower is in operation and capable of growing multiple crystals from the same crucible.

Additional equipment modifications are in process according to the Program Plan.

A two- and a three-melt run have been demonstrated. In the three-melt run, 42.5 kg of crystal was grown from 48.7 kg melted for a pulled yield of 87.3%.

The total time of the longest run to date was 44 hours and, although crucible

devitrification was observed, it is considered to be tolerable. There appear to be no other serious problems with long runs.

Economic analyses of two methods of continuous CZ have yielded add-on costs of \$19.40 and \$15.07 per kilogram (excluding polysilicon cost). The lower cost is for four 12.5 cm diameter crystals from one crucible.

Figure 5a

Figure 5b

FIGURE 6

FIGURE 7