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CONTINUOUS CZOCHRALSKI GROWTH

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

MASTER

Ninth Quarterly Progress Report for October 1—December 31, 1979

By
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E. Roberts

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

Kayex Corporation
Rochester, New York

U.S. Department of Energy

Solar Energy

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CONTINUOUS CZOCHRALSKI GROWTH

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

NINTH QUARTERLY PROGRESS REPORT
OCTOBER 1, 1979 - DECEMBER 31, 1979
PROGRAM MANAGER: DR. R. L. LANE
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"The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE".

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ABSTRACT

During this reporting period, four more 100 kg continuous runs were completed to satisfy the six required by the project extension. One of the four (2*) was performed using a new standard CG2000 RC grower, using accessory equipment and process techniques developed under this project. The sixth and final 100 kg continuous run was performed with purified carbon parts and resulted in the best results to date. Run No. 62 resulted in 103.0 kg being pulled from 104.5 kg total charge weight (98.6% pulled yield). Of this 103 kg grown, 89.3 kg were monocrystalline. Moreover, 72.2 kg was "OD" material. Another encouraging result was that 85% of the eighth crystal was monocrystalline and 67% of the ninth and last crystal was monocrystalline after ninety seven hours of continuous growth.

Impurity analysis data indicate that impurity build-up in grown crystals during 100 kg continuous runs is insignificant. However, the results indicate that impurities tend to accumulate in the crucible. Moreover, impurity analysis performed on new crucibles indicates that these new crucibles may not be as pure as the manufacturer's literature states.

Solar cell efficiency data from two 100 kg continuous runs were received this quarter with very encouraging results. This data is contained in Table 9, page 23 and Figure 4, page 26. The average efficiencies of all cells tested from Run No. 49 was 16.35% AM1, which is considerably higher than our 14% AM1 goal. Moreover, the efficiencies remained at or near the same levels throughout the entire 100 kg continuous run. We expect to receive solar cell efficiency results from three more of the 100 kg continuous runs in the next quarter.

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1.0 INTRODUCTION

The purpose of this program is to demonstrate the growth of at least 100 kilograms of monocrystalline ingot from one crucible by the Czochralski (CZ) method.

The approach to the continuous growth process being pursued in this effort relies on conventional CZ technology combined with new equipment designs which allow repeated alternate cycles of crystal growth and hot melt replenishment by methods which are suitable for use in a high volume production facility.

A Hamco Model CG2000 crystal grower was modified with a special chamber for the storage of a supply of polycrystalline silicon and a vacuum-tight isolation valve to permit retrieval of crystals and melt replenishment without contamination. A number of additional modifications to the facility have been completed in the program, and the process study phase is now under way, with a number of multi-ingot runs having been performed.

It is the purpose of this contract extension to describe a program aimed at a more accurate cost analysis of the continuous growth of silicon into material and also an investigative program for the purpose of defining and solving problems which may hinder the development of continuous Czochralski growth.

The proposed work consists of two efforts, the first being to conduct a series of continuous crystal growth runs in a pilot-plant-type operation. This is for the purpose of establishing with more certainty the costs of the process and pushing the technology even farther from the present laboratory operation toward production. The second (and parallel) effort will be to conduct an investigation study in the areas which appear to be related to yield of high quality crystal, specifically the quality of the crucible, the effect of impurities on degradation

of the crucible, the introduction of impurities into the silicon, and the effect of the furnace hot zone parts on the process, specifically in terms of impurities or foreign particles introduced into the silicon melt from its environment.

2.0 PROGRESS

2.1 Crystal Growth Development

During this quarter (October 1, 1979 - December 31, 1979), our goals were to complete the six one-hundred kilogram continuous runs and, at the same time, add to our knowledge of time cycles, power requirements and process parameters and limitations. As of October 1, 1979, two of the six one-hundred kilogram continuous runs had been completed. Two more one-hundred kilogram continuous runs were completed in October (Run No. 55 and Run No. 2*). These two runs were reported in the Second Annual Report, September 30, 1979, and again in the October, 1979 monthly report. In November, the recharge mechanism was refitted to the grower. A continuous run (Run No. 58) was attempted on November 12 with disappointing results. A total of 52 kilograms was grown before the run was terminated due to structure loss problems. It was felt that the original recharge mechanism design could result in poor vacuum sealing capabilities of certain parts of the furnace (door, etc.) when installed on the top of the pull chamber. Therefore, the recharge mechanism was removed after Run No. 58. After one test run in November, a 100 kilogram continuous run was attempted on December 10, 1979 with the results shown in Table 1. The results were much better than Run No. 58. Therefore, after needed maintenance was performed on the grower, a test run (Run No. 61), yielding very good results, convinced us to attempt the sixth one-hundred kilogram continuous run with purified parts on January 7, 1980. The results of this run were good, as seen in Table 1.

2.1.1 100 kilogram Continuous Recharge Runs

Table 2 summarizes the six one-hundred kilogram continuous runs

performed under this contract. They were accomplished between June 26, 1979 and January 11, 1980.

Runs No. 47, 49, 55 and 2* have been reported previously. Therefore, Run No. 60 and Run No. 62 will be emphasized in this report.

Table 1 summarizes Run No. 60 and Run No. 62. Run No. 60 produced 100.4 kg of crystal. Eight crystals were grown, yielding 60.8 kg of monocrystalline silicon. An air leak developed early in the run which eventually caused a serious structure loss problem after the fifth crystal had been grown. Figure 1 shows a composite of the eight crystals grown during Run No. 60. The discoloration on the second crystal indicates that an air leak was present at this time. Crystals 5, 7, and 8 also show the discoloration patterns characteristic of an air leak. Crystal No. 4 exhibits this discoloration to a lesser degree. Starting with crystal 5, circular ring patterns are also visible on the crystals. This pattern usually means that the air leak is a periodic leak - not continuously the same. When the run was completed, an air leak was found in the feed through shaft which operates the seed lift. When the shaft was rotated, the vacuum increased and decreased periodically. If the shaft rotation was stopped at a certain point, the leak would seal and the grower vacuum would decrease to the required limits. (Even though it was possible to grow high quality crystal when the air leak was present, we feel that excessive silicon monoxide buildup occurred along with severe devitrification of the crucible as the run progressed, making it impossible to maintain structure for any length of time during a growth cycle). This can be evidenced by the last three crystals grown. Crystals #7 and #8 lost structure on the crown and contained only 18% and 10% high quality crystal respectively. This, of course, decreased the overall percentage of high quality crystal from 98.6% after 55.7 kg had been grown to 60.6% at the end of the run.

SUMMARY OF RUN NO. 60 AND RUN NO. 62

	<u>NO. 60</u>	<u>NO. 62</u>
Crystal Ingot Diameter	12.2 - 13.2 cm	11.7 - 12.7 cm
Initial Melt Charge	30 kg	30 kg
Crucible Diameter	35.5 cm (14 in.)	35.5 cm (14 in.)
Total Silicon Melted	108.7 kg	104.5 kg
Total Ingot Pulled	100.4 kg	103.0 kg
Pulled Yield	92.4%	98.6%
Total Mono Crystal	60.8 kg (60.6%)	89.3 kg (86.7%)
Number of Ingots	8	9
Throughput	1.18 kg/hr	1.06 kg/hr
Total Run Time	85 hours	97 hours
Recharge Material	100% lump	100% lump

TABLE 1

CONTINUOUS CZ GROWTH SUMMARY

Date	Run No.	Total Melted (kg)	Total Pulled (kg)	No. of Crystals	Diameter (cm)	Average Pull Speed (cm/hr)	Run Time (hr)	Throughput (kg/hr)	Pulled Yield (%)	High Quality Crystal (%)
6/26/79	47*	65.1	60.2	5	12.7-13.5	6.8	51.6	1.17	92.5	88.3
7/17/79	49	114.5	108	9	13.3	7.0	85.5	1.26	94.3	84.8
10/8/79	55	106.1	100.6	10	12.7-16.0	7.2	91.0	1.11	94.8	74.6
10/22/79	2*	104.5	100.3	9	12.7	7.7	109	0.92	96.0	63.7
12/10/79	60	108.7	100.4	8	12.2-13.2	7.6	85	1.18	92.4	60.6
1/7/80	62	104.5	103.0	9	11.7-12.7	7.9	97	1.06	98.6	89.3

*12-inch crucible/hot zone (all others 14-inch crucible hot zone)

TABLE 2

RUN NO. 60

FIGURE 1

Based upon the fact that it was possible to grow good crystal with an air leak present, we are considering a theory that impurities entering the furnace in conjunction with an air leak are not in themselves causing a structure loss problem. It appears to be the excessive silicon monoxide and silicon dioxide that are formed when a supply of oxygen is available (air leak), along with the accelerated crucible devitrification that may be the major cause of structure loss.

Samples from Run No. 60 were sent out for solar cell fabrication and evaluation on January 16, 1980.

Run No. 62 resulted in the highest percentage of monocrystalline silicon being grown to date. A total of 103.0 kg was pulled, of which 89.3 kg was monocrystalline. Moreover, 72.2 kg were of "OD" quality.

We feel that the furnace system was leak-free throughout the entire run. Figure 2 shows the group of nine crystals grown during Run No. 62. The first crystal resulted in a 20.4 kg crystal being grown, which was 90% "OD" and the remainder single crystal. At this point, we noticed a potential problem. The crucible had sagged such that one side was in contact with the graphite upper support while the other side showed a gap of 1/4" to 1/2" from the upper support. A thick layer of silicon monoxide formed on the top lip of the crucible where the gap occurred. When the crucible was raised up into the cold part of the furnace as the crystal was grown, the oxide cracked and broke apart. From time to time during the run, pieces of silicon monoxide were noticed falling from the top rim of the crucible into the melt.

The capability of growing "OD" was demonstrated on eight of the nine

RUN NO. 62
FIGURE 2
8

crystals grown. The sixth crystal was the only crystal that did not contain at least 50% "OD" material. Moreover, the eighth crystal was 85% single crystal while the last crystal was "OD" for 15 of the total 27 inches.

The fact that we were able to grow a high percentage of monocrystalline material at the end of the run tends to indicate that the structure loss problems that were encountered during the run were not consistent or predictable and may have been caused by pieces of the oxide layer falling into the melt. It is impossible to predict when a piece of oxide might fall into the melt. However, we do know from experience that, as the crucible goes higher up into the colder regions of the upper furnace chamber, the oxide tends to crack and peel, increasing the possibility of some coming loose and falling into the melt. An example of this can be seen on the bottom of the straight growth portion of crystal #6 (2.9 kg). Pieces of the oxide were flaking off and falling into the melt during the first four inches of this growth cycle. A large piece actually stuck to the outside of the crystal at the bottom - observe rough area on sixth crystal at the bottom. Refer to Figure 2, page 8.

It is felt that, if a crucible fits the upper support properly - tight to the inside diameter, the upper crucible walls stay hotter and therefore drastically reduce the amount of oxide buildup on the upper portions of the crucible walls.

If Figure 1 and Figure 2 are compared, the discoloration of the crystals in Figure 1 becomes apparent. The crystals in Figure 2 are clean in comparison to those in Figure 1.

The graph in Figure 3 relates the number of kilograms pulled during

Run No. 62 as a function of run time. As can be seen, up until the time that severe structure loss problems occurred (during the growing of the fifth crystal), the growth rate was satisfactory (2.09 kg/hr - 2.38 kg/hr) and the throughput was also satisfactory - 1.26 kg/hr. After the fifth crystal was grown, the throughput fell sharply, concluding at 1.06 kg/hr. Sixty-five (65) kilograms had been grown after fifty-one hours of run time, yielding 80.2% "OD" material.

Tables 3 and 4 further break down the run time cycles into three categories (Recharge Time, Crystal Growth Time and Growth Preparation Time), as has been done in the past. A comparison between Run No. 60 and Run No. 62 indicates that the Recharge Time and Crystal Growth Time are nearly the same during both runs even though one more crystal was grown during Run No. 62. The major difference occurs in the area of Growth Preparation, where 21.4 hours was devoted during Run No. 60 while 32.6 hours was devoted during Run No. 62.

October's monthly report included similar information on the two previous 100 kg runs completed in October - Run No. 55 and Run No. 2*. Run No. 2* was performed in a new standard Hamco CG2000 RC crystal grower using a 12 inch diameter crucible and hot zone parts. Moreover, there were mechanical problems during the run, which increased the Growth Preparation Time significantly.

Table 5 presents a composite of the time cycles for all 100 kg runs completed to date. Runs No. 30, 47 and 2* were performed using 12 inch crucibles and corresponding hot zones. Recharge times have improved while straight growth times have stabilized at about 50% of the run time. The largest variation is experienced in Growth Preparation, usually due to mechanical problems and structure loss problems.

RECHARGE AND INGOT GROWTH TIME (RUN NO. 60)

Melt No.	Recharge Time (Hr) ⁽¹⁾	Comments	Crystal Growth Time (Hr) ⁽²⁾	
1	2.0 (8:15 - 10:15am)	30.0 kg cold charge	5.0 (12:55 - 5:55pm)	(10.9 kg) (2.18 kg/hr)
2	1.8 (5:55 - 7:45pm)	10.0 kg lump	8.8 (9:05 - 4:55am)	(16.1 kg) (1.83 kg/hr)
3	2.5 (4:55 - 7:25am)	15.8 kg lump	1.3 (8:45 - 10:05am)	(3.4 kg) (2.62 kg/hr)
4	0	None (Replaced cable & coupling)	3.7 (12:55 - 3:35pm)	(7.2 kg) (1.95 kg/hr)
5	2.0 (3:35 - 5:35pm)	10.9 kg lump	7.6 (7:05 - 2:40am)	(15.0 kg) (1.97 kg/hr)
6	1.8 (2:40 - 4:30am)	16.8 kg lump 1) Rewired seed holder (5:45am) 2) Lost on crown (7:30am) 3) Lost on crown (9:30am) 4) Lost 1" on crystal (11:00am)	1.2 (10:55 - 12:05pm)	(3.1 kg) (2.58 kg/hr)
7	0	None (Replaced broken cable)	11.0 (4:40 - 3:40am)	(22.5 kg) (2.05 kg/hr)
8	2.7 (3:40 - 6:20am)	26.5 kg lump (Two dump cycles) Lost structure on crown (7:30am)	12.2 (9:30 - 9:45pm)	(22.2 kg) (1.82 kg/hr)
Total 12.8 hr		108.7 kg	50.8 hr	

(1) Recharge time includes:
Removal of grown crystal
Insertion of hopper
Dump hopper charge
Melt down
Seed preparation up to
seeding the melt

(2) At growth diameter 122mm - 132mm

NOTE: Total run time = 85 hours
12.8 hrs (15.1%) was devoted to the recharging procedure including initial melt down of 30 kg cold charge. 50.8 hrs (59.8%) was devoted to straight growth at diameter. 21.4 hrs (25.2%) of the run time was used for Growth Preparation: (1) Stabilize temperature (2) Growing Seed (3) Crown Growth (4) Melt Backs

TABLE 3

RECHARGE AND INGOT GROWTH TIME (RUN NO. 62)

Melt No.	Recharge Time (Hr) ⁽¹⁾	Comments	Crystal Growth Time (Hr) ⁽²⁾	
1	1.5 (8:05 - 9:35am)	30.0 kg cold charge	9.5 (12:15 - 9:45pm)	(20.4 kg) (2.15 kg/hr)
2	2.7 (9:45 - 12:25am)	20.1 kg lump 1) Quartz on seed (2 dumps) 2) Lost structure at 1" - melt back 2:40am 3) Lost structure on shoulder 4:00am	4.2 (6:00 - 10:10am)	(9.5 kg) (2.26 kg/hr)
3	2.0 (10:10 - 12:10pm)	9.5 kg lump	5.6 (1:50 - 7:25pm)	(13.4 kg) (2.39 kg/hr)
4	1.8 (7:25 - 9:15pm)	13.4 kg lump	5.6 (10:25 - 4:00am)	(12.5 kg) (2.23 kg/hr)
5	1.5 (4:00 - 5:30am)	12.5 kg lump	4.5 (7:00 - 11:30am)	(8.9 kg) (1.98 kg/hr)
6	1.7 (11:30 - 1:10pm)	9.0 kg lump 1) Lost structure on crown (2:25pm)	1.7 (4:45 - 6:25pm)	(2.9 kg) (1.71 kg/hr)
7	0 (Put new seed on) (6:25 - 7:30pm)	None 1) Lost structure on crown (8:40pm) 2) Change seed was 1,1,1 (9:30pm) 3) Change seed to 1,0,0 (10:10pm) 4) Lost structure on crown (11:10pm)	3.3 (1:20 - 4:40am)	(6.8 kg) (2.06 kg/hr)
8	1.8 (4:40 - 6:30am)	10.5 kg lump 1) Lost structure on crown (8:00am) 2) Lost structure at about 2" meltback (10:20am)	5.2 (12:40 - 5:50pm)	(12.0 kg) (2.31 kg/hr)
9	0 (5:50 - 7:45pm) Waited for pictures to be taken	None	11.8 (9:10 - 9:00am)	(16.6 kg) (1.41 kg/hr)
Totals		13.0 hrs	104.5 kg	51.4 hrs

(1) Recharge Time includes:
Removal of grown crystal
Insertion of hopper
Dump hopper charge
Melt down
Seed preparation up to
seeding the melt

(2) At growth diameter 117mm - 127 mm

NOTE: Total run time = 97 hours
13.0 hrs (13.4%) was devoted to the recharging
procedure including initial melt down of 30 kg
cold charge. 51.4 hrs (53%) was devoted to

straight growth at diameter. 32.6 hrs (33.6%)
of the run time was used for Growth Preparation:
(1) Stabilize Temperature, (2) Growing Seed,
(3) Crown Growth, (4) Melt Backs.

TABLE 4 (Cont'd)

COMPOSITE OF RECHARGE AND INGOT GROWTH TIMES FOR 100 KG RUNS

RUN NO.	RECHARGE TIME ⁽¹⁾		GROWTH PREPARATION ⁽²⁾		STRAIGHT GROWTH & TAPER		TOTAL RUN TIME HRS
	HRS	%	HRS	%	HRS	%	
30	18.9	(23.9)	20.9	(26.5)	39.2	(49.6)	79
47	8.8	(17.1)	11.9	(23.1)	30.9	(59.9)	51.6
49	16.4	(19.2)	20.3	(23.7)	48.8	(57.1)	85.5
55	12.9	(14.2)	33.5	(36.8)	44.6	(49)	91
2*	19	(17.4)	38.3	(35.1)	51.7	(47.4)	109
60	12.8	(15.1)	21.4	(25.2)	50.8	(59.8)	85
62	13.0	(13.4)	32.6	(33.6)	51.4	(53)	97

(1) Recharge Time includes:
Removal of grown crystal
Insertion of hopper
Dump hopper charge
Melt down
Seed preparation up to seeding
the melt

(2) Growth Preparation includes:
Stabilize Temperature
Growing Seed
Crown Growth
Melt Backs

TABLE 5

2.1.2 Individual Process Runs

Three one-crystal runs were made in October. Run No. 54 was made to test the equipment prior to attempting a continuous run - Run No. 55.

Before Run No. 54, numerous preventive maintenance and repair items were performed on the grower. The heat shield was modified to improve the exhaust characteristics of the furnace.

Run No. 56 was performed at the request of the JPL monitor. Wafers of a crystal grown during this run were sent to the JPL monitor for further testing.

Run No. 57 was an experimental run to determine the effect of a graphite radiation shield which was attached to the furnace top plate such that the crystal would be drawn through the radiation shield as it was grown. The objective was to eliminate the silicon monoxide buildup on the crystal and increase the growth rate. The crystal did remain clean throughout the run, indicating the argon flow patterns had changed. However, we were not able to make any conclusions as to the effect the radiation shield had on the growth rate. More testing is planned in this area in the future.

Run No. 58 was an unsuccessful attempt at growing 100 kg. The recharge mechanism was refitted before this run and was removed after this run. It is our opinion that the recharge mechanism may have caused distortion in the pull chamber door and created sealing problems in that area.

Run No. 59 was a proving run after repairs and maintenance had been performed on the grower after Run No. 58. A new pull chamber and door were installed prior to Run No. 59.

Run No. 61 was performed to test the grower after routine preventive maintenance was performed following Run No. 60 (100 kg run). A 21 kg, five inch diameter crystal was grown "OD" all the way to the end of the taper.

Based on these results, we decided to make the last 100 kg run with purified graphite parts as soon as possible.

2.2 Sample Preparation and Analysis

Extensive testing for impurity contaminations in silicon crystal samples as well as samples taken from new and used fused quartz crucibles has been performed in the past with a majority of the results being received this quarter.

All results continue to indicate that impurities in the furnace/melt environment are not absorbed into the crystal to any significant degree. For the most part, residual melt samples have not shown a significant increase in impurities either (except for Run No. 30). However, it is very difficult to be sure that we are getting a representative sample of the residual melt. It is possible to get segregation within the sample taken from the residual melt.

Based upon these present results, our attention has focused more on the fused quartz crucibles which may act as a getter of certain furnace/melt impurities. However, test results that we have received on new crucibles do not correspond to impurity levels of new fused quartz crucibles as supplied by the crucible manufacturers. See Tables 7 and 8.

In addition to impurity analysis results received this quarter, solar cell efficiency results were received in November for samples from Runs No. 47 and 49. These results were very encouraging and tend to indicate that solar cell efficiencies do not deteriorate throughout an entire 100 kg continuous run. Moreover, efficiencies have surpassed the 14% AM1 goals of this project with an efficiency of 16.94% being realized on one cell tested from Run No. 49. More detailed data is presented in Section 2.2.2 of this report.

For a more detailed discussion of both impurity analysis and solar testing data, refer to the November, 1979 monthly report.

2.2.1 Impurity Analysis

More test results have been received since the September report. These results continue to indicate that there is no significant buildup of impurities in the grown crystals during a 100 kilogram continuous run.

Table 6 lists seven impurities that have been found in high concentrations in samples of crucibles used for 100 kilogram continuous runs. Data from four continuous runs are presented in Table 6. This table lists impurity results taken from crystal samples and the residual melts only. As can be seen, there is no significant increase in impurity levels except for the residual melt from Run No. 30. The high concentration of calcium, potassium and fluorine cannot be explained for sure. However, it is possible that a microscopic water leak may have been present in the furnace during this continuous run. Other possibilities for this sample showing these relatively high concentrations are:

- 1) Poorly prepared (cleaned) polysilicon
- 2) Method of obtaining the residual melt sample
- 3) Impure virgin poly
- 4) Impure quartz
- 5) Testing method and procedure.

Table 7 lists the impurity concentrations found in samples of crucibles used for the runs indicated. The same seven elements listed in Table 6 are listed in this table. These samples of quartz contain much higher concentrations of the elements listed than the corresponding crystal samples taken from the Toshiba crucible used during Run No. 49. The calcium concentration on Run No. 49 crucible drastically increased as compared to the calcium concentration found in a new Toshiba crucible. The exact cause for this increase, or the mechanism which is causing it, is not understood at this

time.

To better verify our test results, one crucible manufacturer (Quartz Products) has agreed to take identical quartz crucible samples and run impurity analysis tests. Table 8 lists impurity concentrations of crucibles as supplied by the crucible manufacturers.

2.2.2 Solar Cell Results and Analysis

Table 9 lists the results of the solar cells fabricated and tested by Applied Solar. These cells were tested under AMO conditions. We converted the efficiency results from AMO to AM1 by using a 1.4:1 conversion factor as previously indicated by JPL. The resultant AM1 efficiency results range from 14.98% to 16.94%. The results from Sample No. 47-3-T were not a true representation of the sample's efficiency capabilities because they were polished beyond the normal lower limit for cell thickness. Therefore, the results were lower than would be expected using cells of normal thickness.

The graph depicted in Figure 4 relates the results from Applied Solar in relation to the project goals of 14% AM1 efficiencies. The top line represents the average of the four cells. The range of results for each wafer (four cells) is indicated by each vertical line.

The cells from the top of the first crystal, taken after approximately four (4) kilograms had been grown, had an average efficiency of 16.3%. The cells from the bottom of the first crystal at the 10 kilogram mark averaged 16.1%. The cells from the sixth crystal grown at the 50 kilogram mark yielded an average efficiency of 16.2%. The last crystal grown yielded average efficiencies of 16.1% at the top (90 kilograms) and 15.8% towards the bottom after 100 kilograms had been grown.

The heavy dark line represents the 14% project goal.

As you can see, efficiency results have exceeded the 14% AM1 goal of

Impurity Analysis (Concentration in PPM Weight)
Silicon Crystal Samples

Sample #	Na	Mg	Ca	K	Fl	Cl	Ba
47-1 Top	7	0.3	0.1	0.1	0.1	0.7	
47-3 Bottom	0.8	0.3	0.1	0.1	0.1	0.3	≤ 0.1
47 Residual Melt	*1	0.3	0.1	0.1	0.1	0.6	≤ 0.1
49-1 Top	0.4		0.1	0.1		*0.3	≤ 0.1
49-3 Top	4		0.1	0.2		*0.6	
49-9 Top	*7	0.5	0.1	*0.6	0.1	0.3	0.1
49-9 Bottom	1	0.5	0.1	0.1	0.1	*0.7	≤ 0.1
55-1 Top	0.9	0.3	0.1	≤ 0.1	≤ 0.1	0.2	
55-10 Top	0.4	0.2	*0.3	≤ 0.1	≤ 0.1	0.2	≤ 0.1
55-10 Bottom	4	≤ 0.3	0.1	0.6	≤ 0.1	*0.7	2
55 Residual Melt	0.7	0.2	*0.3	0.1		0.7	≤ 0.1
30-1A	0.9	0.2	*4	1	0.06	0.2	
30-1 Top	0.5	0.3	0.2	0.04	0.02	0.3	0.09
30-1 Bottom	0.7	0.2	0.2	0.4	0.04	0.2	
30-6 Top	3	4	0.4	0.3	0.2	0.2	
30-6 Bottom	4	4	0.7	0.6	0.6	0.4	
30 Residual Melt	3	2	150	12	≈ 30	5	< 0.1

*Heterogeneous

All elements not detected are normally < 0.3 PPM.

MC - Major Component

Int - Interference

TABLE 6

Impurity Concentrations (PPM)
Crucible Samples and Other Related Samples

Sample #	Na	Mg	Ca	K	Fl	Cl	Ba
47-Crucible 12" (Amersil)	66	6	33	35	≈ 20	29	6
49-Crucible 14" (Toshiba)	41	7	410	80	11	41	35
55-Crucible 14" (Quartz Products)	24	≤ 0.2	36	52	≈ 170	9	20
2* Crucible 12" (Quartz Scientific)	57	82	68	220	MC $> 0.1\%$	25	480
New 14" Crucible (Quartz Products)	61	19	14	120	≈ 270	11	0.9
New 14" Crucible (Toshiba)	20	3	3	24	≤ 1	20	*7
Run 2* Top of Crucible (cleaned) (Quartz Scientific)	92	4	20	89	≈ 460	18	≤ 0.9
Run 55 Hopper Deposit formed during Recharge Cycle after water leak developed (White Powder)	> 160	11	21	> 770	≈ 550	28	*1

TABLE 7

CRUCIBLE IMPURITIES
(CONCENTRATION IN PPM BY WEIGHT)

1 Impurity	2 Amersil	3 Quartz Products	4 General Electric	5 Corning
Al	12	20	28	5
B	1	NR	1	0.5
Ca	4	3	3	1
Cu	1	NR	1	0.05
Fe	3	1	4	5
Li	1	1	0.5	NR
Mg	1	1	1	0.1
K	2	3	3	0.1
Na	3	1	3	2
Ti	1	2	2	10
Cr	NR	NR	3	NR
Zn	NR	NR	2	NR
Mn	NR	NR	2	NR

NR: Not Reported

INT: Interference

Maximum impurities in Fused Quartz (vitreous SiO₂) crucibles provided by vendors.
Expressed as parts per million by weight.

TABLE 8

APPLIED SOLAR ENERGY INC.
SOLAR CELL ELECTRICAL DATA

Sample No.	Voc mV	Isc mA	Iscb mA	Iscr mA	I1 mA	V1 mV	P mW	CEff %	AM0 n %	AM1 n %
47-1T-1	590	144.1			132.6	485	64.31	75.6	11.9	16.67
-2	565	143.4			131.0	460	60.26	74.4	11.1	15.54
-3	590	141.8			124.2	480	59.62	71.3	11.0	15.4
-4	580	143.9			130.3	480	62.54	74.9	11.6	16.24
47-3T-1	552	129.1			119.5	450	53.78	75.4	9.9	13.86
-2	553	130.5			121.3	450	54.59	75.6	10.0	14.0
-3	545	133.0			113.0	440	49.72	68.5	9.2	12.88
-4	552	131.4			119.3	450	53.69	74.0	9.9	13.86
47-3B-1	570	140.5			126.0	470	59.22	73.9	10.9	15.26
-2	582	142.7			131.5	480	63.12	76.0	11.7	16.38
-3	586	143.0			131.0	480	62.88	75.0	11.6	16.24
-4	573	144.1			132.2	470	62.13	75.2	11.5	16.1

TABLE 9

APPLIED SOLAR ENERGY INC.
SOLAR CELL ELECTRICAL DATA

Sample No.	Voc mV	Isc mA	Iscb mA	Iscr mA	I1 mA	V1 mV	P mW	Cff %	AM0 η %	AM1 η %
49-1T-1	599	146.1			135.1	485	65.52	74.9	12.1	16.94
-2	597	144.2			132.2	485	64.12	74.5	11.8	16.52
-3	596	144.1			124.0	465	57.66	67.1	10.7	14.98
-4	598	144.2			132.3	485	64.17	74.4	11.9	16.67
49-1B-1	593	140.7			127.1	490	62.28	74.6	11.5	16.1
-2	593	140.7			127.1	490	62.28	74.6	11.5	16.1
-3	592	138.2			127.1	490	62.28	76.1	11.5	16.1
-4	594	140.0			127.1	490	62.28	74.9	11.5	16.1
49-6T-1	595	142.8			132.0	485	64.02	75.3	11.8	16.52
-2	593	142.3			129.0	485	62.57	74.1	11.6	16.24
-3	595	143.1			128.6	485	62.37	73.3	11.5	16.1
-4	595	140.4			128.0	485	62.08	74.3	11.5	16.1

TABLE 9 (Cont.)

APPLIED SOLAR ENERGY INC.
SOLAR CELL ELECTRICAL DATA

Sample No.	Voc mV	Isc mA	Iscb mA	Iscr mA	I1 mA	V1 mV	P mW	Cff %	AMO η %	AM1 η %
49-9T-1	598	143.3			131.2	490	64.29	75.0	11.9	16.67
-2	593	141.0			120.0	490	58.8	70.3	10.9	15.26
-3	594	141.5			126.5	490	61.99	73.7	11.5	16.1
-4	596	142.5			130.0	490	63.7	75.0	11.8	16.52
49-9B-1	589	138.0			123.0	480	59.04	72.6	10.9	15.26
-2	589	142.0			128.2	480	61.54	73.6	11.4	15.96
-3	590	137.3			121.0	480	58.08	71.7	10.7	14.98
-4	596	139.1			126.1	490	61.79	74.5	11.4	15.96
Control-1	602	143.7			129.3	500	64.65	74.7	11.9	16.67
-2	600	142.5			129.0	495	63.86	74.6	11.8	16.52
-3	598	143.0			128.0	480	61.44	71.8	11.4	15.96
-4	595	143.2			128.5	490	62.97	73.9	11.6	16.24

TABLE 9 (Cont.)

Figure 4

this project on all cells tested. Moreover, there does not appear to be any significant decrease in efficiency throughout the run.

Four control cells yielded efficiencies from 16.0% to 16.7%, which is similar to the samples tested.

3.0 CONCLUSIONS AND DISCUSSION

This reporting period has seen the completion of the six 100 kg continuous recharge runs that were outlined in the original contract extension. This ninth quarterly reporting period culminated in four of the six 100 kg continuous runs being completed. Two were completed during the month of October, one during December and the last during the first week of January. The data for these runs is listed in Table 2, page 5, of this report.

Based upon the results of the six 100 kg continuous runs, it is felt that the grower and component parts are capable of growing at least 100 kg of silicon from one crucible at any time. However, a leak-free system must be maintained throughout the run in order to grow a high percentage of monocrystalline or zero dislocation material.

Details of time cycles for these runs can be found in previous reports. Runs No. 55 and 2* are included in the October, 1979 monthly while the last two, Run No. 60 and Run No. 62, are contained in this report - Tables 3 and 4.

Observations taken during Run No. 60 and Run No. 62 have caused us to question the primary cause of structure loss during continuous runs. We still feel that a leak-free furnace system is imperative for growing high quality crystals over an extended period of time. However, even with a tight, leak-free system, the possibility exists that silicon monoxide or silicon dioxide may build up on the upper portion of the crucible wall and, from time to time, fall

into the melt, causing a loss of structure if it comes into contact with the growth interface of the crystal.

Further data from impurity analysis has indicated that new crucibles may have higher impurity concentrations than indicated by the manufacturers. However, it still appears that furnace/melt impurities do not combine with the silicon as the crystal is being grown to any significant degree. The data still indicates that the crucible may absorb most of the impurities.

Solar cell efficiency results for Run No. 49 and Run No. 47 were very encouraging. Results indicate that efficiencies are higher than the 14% goals of this project and the efficiencies do not appear to deteriorate throughout the entire 100 kilogram continuous run. Refer to Figure 4, page 26.

It has also been shown that high quality crystal ("OD") can be grown using continuous growth methods after ninety (90) hours of operation. Refer to Table 1 and Table 2 and Figures 2 and 3.

A three month extension of this contract commences January 1 and requires parameters for six inch diameter crystal growth and parameters for the growth of 150 kg of six inch diameter material using the continuous methods developed under this project.

4.0 PLANS

In January, we will be investigating the parameters involved in growing six (6) inch diameter crystals in the grower using several test runs before we attempt a 150 kg continuous run growing six inch diameter crystals.

Samples will be sent out for solar cell fabrication and efficiency testing from Runs No. 60 and 62.

A Residual Gas Analyzer will be interfaced with the crystal grower during this next quarter to see if we can learn something about the furnace atmosphere during crystal growth cycles.