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LARGE AREA SILICON SHEET BY EFG

Second Quarterly Report for Period April 1—June 30, 1980

October 31, 1980

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Work Performed Under Contract No. NAS-7-100-954355

**Mobil Tyco Solar Energy Corporation
Waltham, Massachusetts**

DIST-348
NTIS-25

U.S. Department of Energy

Solar Energy

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LARGE AREA SILICON SHEET BY EFG

Program Manager: F.V. Wald

Second Quarterly Report - Subcontract No. 954355

Covering Period: April 1, 1980 - June 30, 1980

October 31, 1980

"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

In Machines 17 and JPL No. 1, experiments are being continued to better understand the limitations on growth speeds over 4 cm/minute, imposed by the details of the present cartridge design. Also, work is progressing on the uniform introduction of oxygen into high-speed growth systems and the resulting 10 cm wide ribbon.

In the multiple furnace (3A), further progress in understanding the obstacles preventing the "technical features demonstration" has been made. This is reported in detail in Section III.

In the characterization area, a principal result has been to verify the reproducibility of the behavior of oxygen enriched ribbon insofar as thermal processing during solar cell fabrication is concerned.

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I. INTRODUCTION

The signal success of the five ribbon multiple run in May 1979, which produced from five cartridges 150 m of 5 cm wide ribbon with an average growth speed of ~3.5 cm/minute at a machine duty cycle over 90%, has now clearly demonstrated that "cartridge type" multiple EFG machines can be considered "prototypical" for the development of the future production equipment with which to fabricate silicon ribbon base material at the low cost necessary to achieve the 1986 LSA project goals.

Upscaling of the cartridge to the ultimate 10 cm width which, at growth speeds below 5 cm/minute, is considered necessary to achieve the final cost goals, is also well underway, along with the development of a viable automatic control system, which at this point appears to be sufficiently advanced and also sufficiently inexpensive, to allow perhaps more than the originally projected ten ribbons to be under the control of a single machine operator.

The program has thus made great strides in arriving at a scalable production technology for multiple EFG ribbons, and it appears that in the coming year conclusive final design choices for a production unit will become possible.

Also, in the five-ribbon multiple run, it was demonstrated that material from such units can indeed have reasonable electronic properties, since large area ($\sim 50 \text{ cm}^2$) cells of ~9% efficiency could be prepared from silicon ribbon grown in that experiment.

Such efficiencies, although significant as a general demonstration that this type of equipment in principle can produce ribbon with acceptable electronic properties, are not considered sufficient to meet the LSA project goals for 1986.

Hence, very significant efforts in this program were directed toward the identification and study of mechanisms which could optimize or degrade the electronic properties of the ribbon during growth as well as during

processing into solar cells, and these studies will be further emphasized during the coming year.

So far as crystal growth is concerned, the work has focused on an examination of material quality changes arising from variations in die design, construction materials, thermal conditions and the furnace ambient.

Although we consider optimized die designs and thermal conditions to be worthy of further study, and of importance in particular as they allow us to influence the meniscus height during growth which appears to be a significant variable, it was the furnace ambient manipulation which has led to reproducible, unambiguous, and positive changes in growth system behavior and material quality, such that a definite breakout in solar cell performance over earlier baseline levels in these machines has been observed.

From these experiments, large area (50 cm^2) cells of 11.5% (AM1) efficiency have now resulted, and it is believed that optimization of the gas ambient in the furnace and particularly around the meniscus will lead to further efficiency increases. At this point, it is realized that the gas ambient, in particular with respect to carbon and/or oxygen-containing species such as CO , CO_2 , and CH_4 , is perhaps the one most important unrecognized and uncontrolled parameter affecting growth and properties of EFG silicon ribbon prepared from carbon dies.

However, a distinct model linking ribbon properties and gas phase composition, flow, etc., must now be evolved and proven, and subsequently the results must be applied in appropriately designed machines which control and optimize the relevant parameters. Therefore, because of the apparent importance of the observations so far, we plan to devote a very significant effort to these questions in the year ahead.

Finally, it has become clear that solar cell fabrication sequences can have a significant influence on the material properties in the final cell through the high temperature processing now commonly associated with cell fabrication. It is also suggested that such influences can be both positive or negative, i.e., the diffusion length may decrease during processing, or, through such mechanisms as gettering, precipitation, or complexing, it may perhaps be made to increase.

In either case, the development of processing sequences which at least conserve, and hopefully could improve the initial minority carrier lifetime of imperfect materials, such as EFG ribbon, can conceivably have a large influence on the final solar cell efficiency. These questions, therefore, will continue to be explored. However, at this point in time, multiple EFG ribbon technology appears to be extremely well placed toward actual achievement of the 1986 LSA program goals.

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II. HIGH SPEED GROWTH AND QUALITY STUDIES by J.P. Kalejs

A. Overview

Experiments with the 10 cm cartridge in this quarter have been directed toward optimization of performance (speed and quality) at speeds of 4 cm/minute and above. Growth conditions in the speed range from 3.5 to 4.0 cm/minute have generally been good, and efforts have been made to gain a better understanding of factors which limit the speed capabilities of the cartridge above 4 cm/minute. A study of the effect of some die design parameters on high-speed performance has been carried out. The investigation next will be directed toward examining the limitations imposed by the cartridge cooling (cold shoe) and heating (after-heater) loads.

Quality studies have continued with growth at reduced main zone purge rates and with the use of a mixture of CO_2 in argon. Evaluation of several different cartridge gas inlet configurations has shown that it is difficult to obtain uniform introduction of these gases over the full width of the 10 cm ribbon with the available flow arrangements. However, encouraging indications that ambient effects will be useful in improving material quality at high growth speeds have been obtained. Minority carrier diffusion length increases to over 40 μm have been directly as a consequence of the use of CO_2 .

Conversion of Machine 18 (JPL No. 1) to 10 cm wide ribbon growth has been completed and initial growth runs made. Growth conditions to date have been very poor due to inadequacies in some of the power supplies and in the design of the main zone insulation.

B. Machine 17

A summary of the experimental growth runs carried out in this reporting period is given in Table I. A number of runs with mixtures of CO_2 in argon were made, as gas flow conditions were sought that would

Table I. Run and Ribbon Data for Furnace 17. All Runs are for 10 cm Cartridge with Cold Shoes, Except as Noted.

Run No.	Speed Range (cm/min)	Gas Flow Conditions (ℓ /min)		Die Design Slot/Displacement (cm)	L_D (μ m)	Comments
		Main Zone	Cartridge			
17-091	3.5 - 4.0	5	1	0.040/0.012	21	Continuation of reduced main zone experiments. Some growth instability at highest speed.
17-092	3.7 - 4.0	5	1	0.025/0.008	30	Reasonable growth up to 3.8 cm/min. Die heater shorting affects growth conditions.
17-093	3.7 - 4.0	5	1	0.040/0.012	38	Repeat of reduced main zone experiments.
17-094	2.5 - 2.8	5	1	0.040/0.012	-	Main zone thermocouple problems and die top silicon flood limited growth.
17-095	1.4 - 1.5	6	1	0.025/0.008	-	Test of 10 cm cartridge without cold shoe for use in Machine 18. Very poor growth conditions, no full-width growth possible.
17-096	3.7 - 4.0	6	0	0.040/0.012	25	Test of 10 cm cartridge from Machine 16 with new design of cold shoes (increased ribbon edge cooling). Good growth at 3.8 to 4.0 cm/min.
17-097	3.3 - 4.0	7	1	0.050/0.015	35	Good growth conditions up to 3.7 cm/min; some ribbon cracking problems.

Table I continued.

Run No.	Speed Range (cm/min)	Gas Flow Conditions (ℓ/min)		Die Design Slot/Displacement (cm)	L _D (μm)	Comments
		Main Zone	Cartridge			
17-098	3.5 - 4.0	6	0.5 - 1.0 1% CO ₂	0.050/0.015	16 (28) *	Test of new interface gas flow arrangement with CO ₂ .
17-099	3.0 - 3.9	5	1 - 3 1% CO ₂	0.050/0.015	40	Repeat of run 17-098. CO ₂ used entire run. Some growth instabilities, die top deposits at highest flow rates.
17-100	3.0 - 3.5	5	2.5 1% CO ₂	0.050/0.015	32	Repeat of 17-098 with CO ₂ .
17-101	3.2 - 3.5	5	1 1% CO ₂	0.050/0.015	20 (30) *	Additional gas inlet modification to extend gas exit tubes. Heavier surface film, die-top deposits.
17-102	3.5 - 3.7	6	0.5	0.050/0.015	28	Continue speed optimization studies.
17-103	3.5 - 4.0	7	0.5	0.050/0.015	27	Good growth stability up to 3.8 cm/min.
17-104	3.5 - 4.0	6	0 - 4	0.050/0.015	22	Study gas flow influence on growth stability.
17-105	2.5 - 2.7	6	1	0.040/0.012	-	Broken ribbon caused die top flooding and limited growth.

* First value is without the test gases present; number in parentheses is with the test gases.

Table I continued.

Run No.	Speed Range (cm/min)	Gas Flow Conditions (L/min)		Die Design Slot/Displacement (cm)	L _D (μm)	Comments
		Main Zone	Cartridge			
17-106	3.5 - 4.1	6	1	0.040/0.008	37	Good stability at 4.1 cm/min.
17-107	3.0 - 3.7	6	1	0.040/0.008	48	Test of reduced length end heater suitability for high-speed growth. Undoped melt. Poor growth at high speeds.
17-108	3.0 - 3.5	6	1	0.050/0.015	34	Repeat of -107 with thicker die-top slot die. Poor growth at high speeds.
17-109	3.5 - 4.1	6	1	0.040/0.015	24	Good growth conditions at 4.1 cm/min.
17-110	3.5 - 4.1	6	1	0.040/0.012	28	Good growth conditions at 4.1 cm/min.
17-111	3.5 - 4.1	6	1 1% CO ₂	0.040/0.012	21	Growth limited by broken seed.
17-112	2.5 - 2.8	6	0.5 1% CO ₂	0.040/0.012	12 (23)*	Rapid SiO buildup on cold shoe resulted in thermal imbalance and poor growth conditions.
17-113	2.5 - 2.8	5	2 1% CO ₂	0.040/0.012	12 (23)*	Similar growth difficulties to run -112.
17-114	2.5 - 3.5	6	0.75 1% CO ₂	0.040/0.012	-	Cracked ribbon resulted in silicon spill and growth difficulties.

* First value is without the test gases present; number in parentheses is with the test gases.

improve minority carrier diffusion lengths uniformly across the full width of the ribbon. The other emphasis was on a search for parameters that are important in optimization of the performance of the 10 cm cartridge at speeds of 4 cm/minute and above. Details of the experiments are given below.

Modified gas tube arrangements were introduced in run 17-098 and again in 17-101, whereby the quartz tubes used to convey the test gas to the cartridge were extended so as to more directly exit on the interface. Difficulties with observing effects with the CO_2 in earlier experiments (e.g., no interstitial oxygen in the ribbon) were attributed to the fact that the test gas exited from the quartz tubes at locations too far removed from the interface (at the corners of the cartridge). Clear evidence of the presence of the test gas near the interface was observed with the modified gas inlets, and significant increases in the minority carrier diffusion length at high speeds (up to 4 cm/minute), which are attributable to the gas, were observed for the first time in this high-speed growth system (see Section IV).

In the first series of runs (17-098 to -101), a mixture of 1% CO_2 in argon was used in an attempt to increase the partial pressure of oxygen near the growth interface. Increases in the flow rate of the CO_2 mixture produced noticeable effects on growth conditions. Freezes were more frequent and eventually SiO deposits were observed to build up on the cold shoe to levels which appeared to introduce thermal imbalances at the die top. In one run (17-104), no CO_2 was used in order to examine the influence of pure argon gas flow patterns on growth conditions. Since specific growth instabilities were not apparent with argon, only the effects observed with CO_2 appear to be caused by the presence of the oxygen and/or changes in thermal conditions arising from the build-up of film. A return was made to the use of 1% CO_2 in argon with the new gas flow configuration in an additional sequence of runs, starting with 17-111. Again, the use of the CO_2 was associated with growth instabilities and SiO_x film build-up on the cold shoes.

In the experiments on optimization of cartridge performance at 4 cm/minute and above, growth conditions were generally found to be acceptable

in the speed range from 3.5 to 4.0 cm/minute for the various system configurations used. Die design changes, with respect to capillary slot width and displacement, were not observed to significantly influence performance below 4 cm/minute. However, the largest displacements of 0.015 cm used appeared to be a factor in limiting ribbon edge stability above 4 cm/minute within the context of the present die-top thermal configuration. The capillary channel width (in the ribbon thickness dimension) may also be a parameter important in achieving more stable growth above 4 cm/minute. In general, more leeway in adjustment of the thermal conditions at the die-top is available for the wider channels (e.g., 0.050 cm vs. 0.025 cm). This may be an important factor in attempting to match die design changes with those made in other cartridge components, such as the cold shoe, die-top shields or heaters, for more stable growth at the higher speeds. On the other hand, these benefits must be traded off against the lower available meniscus height (for a given h_{eff}) and increase in ribbon thickness at the edges, which itself limits the available maximum growth speed. Tests of several capillary and displacement combinations have shown that slot thicknesses of 0.035 to 0.040 cm and displacements of not more than about 0.012 cm afford the best possibilities in allowing the maximum speed capabilities of the present system to be attained. The highest speeds of 4.0 to 4.2 cm/minute were achieved only with growth of ribbon of thickness below 250 μ m.

Exploration of the different die design parameters' influence on the upper growth speed limit will also be coupled to testing of modified cold shoe designs in the future. One such design was tried out in run 17-096. This configuration provides additional cooling at the ribbon edges relative to the center and was introduced in Machine 16 in order to attempt to boost its speed capabilities. Growth conditions up to 4.0 cm/minute were good. Further testing of this cold shoe design, and of other die configurations, is planned.

One run (17-095) utilized a cartridge without a cold shoe, which was later used in Machine 18. Growth conditions were very poor, and no full-width growth could be sustained. This experiment showed that some modifications will be needed to make the 10 cm cartridge without the

cold shoe operative. In particular, a reduction of die-shield heating by the afterheater, which radiates directly onto it when the cold shoe is removed, appears to be desirable in improving die-top thermal conditions.

C. Machine 18 (JPL No. 1)

A summary of the experimental activity is given in Table II. Work with the 7.5 cm cartridge was terminated this quarter and conversion to 10 cm wide ribbon growth completed. The last run (18-207) with the 7.5 cm cartridge was carried out with the deliberate introduction of hydrogen to the growth interface. A 1% mixture of hydrogen in argon was used as the test gas in the growth slot. Considerable ribbon breakage and poor growth conditions were experienced in this run, although these conditions cannot conclusively be attributed to the presence of the hydrogen (and its embrittling influence) on the basis of this single run.

Efforts to establish acceptable growth conditions for the 10 cm cartridge in Machine 18 resulted in the diagnosing of several deficiencies in the growth system which prevented successful growth in initial testing of the cartridge. The first several runs were carried out with a cartridge without cold shoes. Growth conditions and die-top thermal conditions were considerably worse than experienced in testing of this growth configuration in Machine 17 and full-width growth was not possible. Subsequently, a cartridge with cold shoes was tried out in order to compare growth conditions in Machine 18 with the well-established baseline in Machine 17. Again, growth conditions were poorer than would be expected on the basis of performance in Machine 17. The growth difficulties were traced to the main zone insulation. A new insulation package has been introduced with the 10 cm cartridge in order to provide better insulation around the cartridge walls. However, it was found that this produced too much heating of the cartridge walls. Although the insulation was beneficial from the point of view that the cartridge heaters could be run at lower power levels, it also made die-top thermal conditions unacceptable for growth. The main zone insulation used with the 7.5 cm cartridge was remachined to accept the 10 cm cartridge and tried out. This resulted in marginal growth conditions, in that seeding and growth were possible.

Table II. Summary of Experiments Carried out in Machine 18 (JPL No. 1).

Run No.	Purpose	Observations
18-206	Repeat of 18-205 with CO ₂	No growth. Run failed because of melt feed problems in die.
18-207	Growth with 1% H ₂ in argon.	Considerable growth difficulties, ribbon splitting in growth slot.
18-208	Repeat of 18-207.	No growth. Run failed due to water interruption. Last run with 7.5 cm cartridge.
18-209	First test of 10 cm cartridge without cold shoes.	No growth. Cartridge power supplies failed.
18-210	Repeat of 18-209.	Limited growth of narrow ribbon. Poor end stability.
18-211	Repeat of 18-210.	Seeding not possible due to die-top thermal imbalances.
18-212	First test of 10 cm cartridge with cold shoes. Remachined main zone insulation from 7.5 cm cartridge.	No growth. Seed broke and blocked growth slot.
18-213	Repeat of 18-212.	Erratic growth. Ribbon very thick (~0.050 cm) and ribbon ends unstable, but crucible emptied of melt.
18-214	Repeat of 18-213 but with regular 10 cm cartridge insulation.	Poor growth conditions attributed to change in insulation. Water failure in cold shoe terminated run. Main zone power supply failure.
18-215	Test of combined 7.5 and 10 cm furnace insulation packages.	Growth conditions improved. Limited growth due to broken seed in growth slot.

However, both the face heater and afterheater power supplies were no longer capable of providing sufficient levels of power to make growth acceptable. In particular, afterheater temperatures of only about 900°C could be reached, and much of the ribbon split.

Remedies to the above problems have been sought by combining the insulation packages used with the 7.5 cm and the 10 cm cartridges. Also, the power supply capabilities have been increased slightly to provide more margin in operation at their highest levels. The first test (run 18-215) of these modifications gave indications that growth conditions have benefited from the changes that were made. Growth was limited because of seed breakage which blocked the growth slot. The furnace was inoperable for a considerable period of time after run 18-214 because of a breakdown in the main zone power supply, which had to be sent out for repairs.

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III. MULTIPLE GROWTH AND AUTOMATIC CONTROLS by B.H. Mackintosh

A. Overview

Development of the 10 cm multiple ribbon growth technology in this quarter proceeded in two principal areas. The first of these was the improvement of growth control and stability through the use of profiled cold shoes. The second was the evaluation and development of the optically-based automatic ribbon edge control system.

In nine of the runs of this period, one version of a profiled (thick-ended) cold shoe yielded consistently easier ribbon spreading and better stability in steady-state growth at about 3.0 cm/minute than the previously standard non-profiled shoes. In the last run of this period, a thicker version of the same profile appeared to retain the same advantages for growth rates around 4.0 cm/minute.

Initial use of the optically-based automatic growth control system gave encouraging results. The equipment operated reliably when connected to the furnace, and stable closed-loop control over the ribbon edge positions was possible. Nine periods of continuous growth under automatic control lasting between one and over three hours were demonstrated in this period.

Two multiple-growth runs were attempted with profiled cold shoes in all three stations, and automatic controls on one. The first of these runs demonstrated significant progress in the multiple 10 cm technology when compared with the outcome of the multiple demonstration runs attempted in February.

B. Summary of Growth Runs in Second Quarter

Pertinent details of these runs are listed in Table III.

TABLE III

SUMMARY OF EXPERIMENTAL RUNS OF FURNACE 3A

RUN NO.	PURPOSE	DIE DIMENSIONS (cm)		TYPICAL GROWTH RATE (cm/min)	DURATION OF AUTO CONTROL PERIODS (minutes)	TOTAL QUANTITY OF RIBBON GROWN (meters)	COMMENTS
		SLOT	DISPL.				
220	First test of profiled cold shoes.	.038	.019	-	-	0	Thick sections of cold shoe too far inboard --- created cold spots.
221	Test cold shoes with profile altered to correct shortcomings seen in run 220.	.038	.013	-	-	0	Severe lateral gradient prevented any full-width growth.
222	Reverse cold shoes in cartridge and re-test for lateral gradient.	.038	.005	-	-	0	Lateral gradient essentially unchanged from previous run.
223	Use lower cold shoe setup height and modified end heaters.	.038	.005	3.0	-	1.4	Good full-width growth was possible at end of run when melt replenisher was turned down.
224	Use large-capacity crucible to avoid needing to operate melt replenisher.	.043	.015	3.2	-	3.0	Good growth behavior with replenishment heaters off.
225	New main zone heaters installed; initial test of auto control system.	.043	.014	3.2	70	2.1	Run was non-replenished. Stable control action with low proportional control gains.
226	Experiment with higher control gains. Test two-layer die-top shield.	.043	.005	3.0	95 90	5.5	Attempted to make open-loop frequency response measurements of edge position. See text.
227	Experiment with use of auto control system in start-up. Test long-term stability.	.043	.015	3.1	210	9.3	Corrections periodically made to position of right edge on die top.
228	Test long-term stability. Observe signal from center (meniscus height) window.	.043	.015	2.8	205	8.5	Growth completely unattended. Ended when ribbon became caught under overhead structure.
229	Measure end-heater power vs. position on die. Test setpoint tracking circuits.	.043	.008	2.6	75	4.1	
230	Three-cartridge run with profiled cold shoes in all and auto control on No. 1.	.043	.015	3.1	201	12.4	Cartridges 1 and 3 performed well. No. 2 inoperable due to assembly error. Melt replenisher failure ended run.
231	Repeat of 230.	.038	.015	3.2	90	3.0	Numerous faults and failures. No significant growth from Cartridges 2 or 3.
232	Single-cartridge run to test thicker version of profiled cold shoes.			-	-	-	Run terminated during heat-up due to water system shut-down.
233	Repeat of 232.	.043	.006	3.8	125 56	7.0	Good stability at 4.0 cm/minute. Rate raised to 4.6 cm/minute before control was lost.

C. Discussion of Developmental Activities

1. Cold Shoe Optimization

As discussed in previous reports, the growth performance of the 10 cm cartridge, operated in Furnace 16, has been poor with regard to stability, ease of growth initiation, and growth rate.

The principal symptom, from the crystal growth operator's point of view, is that the die ends must be cooled to very near the melting point of silicon (using the end heater controls) to maintain the ribbon edges at full-width positions. This low gradient condition at the ribbon ends results in frequent freezes, invariably at these locations.

We have undertaken to remedy this situation by designing cold shoes in which the vertical distance between the die top and the cold shoe, as well as the vertical extent of the cold shoe faces adjacent to the ribbon, vary as a function of position across the ribbon width. These two parameters, H (height above die top) and T (thickness of cold shoes), are shown in Figure 1a, which is of a non-profiled cold shoe. Figure 1b shows the type of profile evaluated during this period, in which there are two combinations of height and thickness, namely H_1 , T_1 and H_2 , T_2 for the center and end regions, respectively. The widths of the die-end regions (W_2) and transition regions (W_1) are also parameters. Table IV shows the dimensions of the non-profiled cold shoes in general use since November 1979, and those of the profiled cold shoes which were utilized in this reporting period.

In the first run using profiled cold shoes, No. 220, it was immediately apparent that the thick end regions extended too far inward for successful growth with the existing die heater set, creating cold spots about 0.5 cm from the die ends. This condition was largely corrected by changing the widths of the end and transition regions of the cold shoes prior to run 221, but a severe lateral temperature imbalance across the die made it impossible to establish full-width growth. The heater controls could be set for stable growth on either the left or the right half of the die, giving a first impression of the improved spreading performance and more stable meniscus at the ribbon edge obtained by the use of the profiled cold shoes. The cold shoes were reversed laterally in run

FIGURE 1A
NON-PROFIED COLD SHOE

FIGURE 1B
PROFIED COLD SHOE

TABLE IV
COLD SHOE DIMENSIONS IN CENTIMETERS
(REFER TO FIGURE 1)

A. NON-PROFILED

<u>Run Nos.</u>	<u>H</u>	<u>T</u>
210 - 219	.09	.28

B. PROFILED

<u>Run Nos.</u>	<u>(CENTER)</u>		<u>(END)</u>		<u>W₁</u>	<u>W₂</u>
	<u>H₁</u>	<u>T₁</u>	<u>H₂</u>	<u>T₂</u>		
220	.21	.21	.13	.28	.65	1.00
221, 222	.21	.21	.13	.28	.75	.39
223 - 231	.16	.21	.08	.28	.75	.39
233	.16	.41	.08	.49	.75	.39

222 to see if some aspect of their construction caused the imbalance; this was not the case. The end heaters were thinned slightly for better heating control of the extreme die end, and the cold shoes were repositioned closer to the die. These changes allowed good, controllable full-width growth to be achieved when the melt replenishment heater was turned down. The ribbon would spread slowly, with a low meniscus, when inboard of the thick section of the cold shoes. Upon approaching these thick areas, the edge meniscus would drop and the spreading rate would increase, requiring careful attention of the operator to progressively raise end heater power levels to avoid "freezing."

Although the cartridge, configured in this way, is inherently unstable with regard to edge position when the ribbon edge is within a few millimeters of the die end, it appears to promote the attainment of a full-width, end bulb stabilized condition in a shorter time than previous configurations. With the ribbon growing in full-width condition, the bulbous-ended dies and the profiled cold shoes both contribute usefully to stable growth as follows. As has been previously described in these reports, the end bulb promotes a large radius of curvature around the ribbon end, a large radius of curvature to the meniscus about an axis normal to the face of the ribbon, and a correspondingly greater equilibrium height of the growth interface above the die. The thick end sections of the cold shoe generate a larger vertical temperature gradient in this region. These combined factors render the overall system more tolerant of fluctuations in die temperature and pull rate. The role of the optical automatic growth control system is discussed below.

The performance improvement with profiled cold shoes was confirmed in run 224, as was a deleterious influence of the replenishment heater in combination with the then-current set of main zone heating elements. A crucible with approximately twice the normal silicon capacity was used to permit longer duration of growth without running the melt feeder. Full-width growth was again found to be impossible when the melt feeder was turned on.

A short chronology of the development of this furnace is useful at this point to explain the apparent cause of this problem. This hot zone

was built in late 1978, and with slight modifications to the heating elements as initially designed, gave a sufficiently uniform temperature profile for highly successful growth of five 5 cm ribbons to be demonstrated. These heating elements were retained through the conversion of the hot zone for three 10 cm cartridges. Temperature measurements taken along the length of the hot zone without cartridges in place showed these elements to give a "W"-shaped profile, hot at ends and center, and cold between. The heater design was changed slightly to flatten this profile, and elements of the new configuration were installed after run 216, just prior to this reporting period. The new elements indeed produced a significantly more uniform profile, as measured without the cartridges in place.

The growth performance of the system, as described above, has made it apparent that the thermal nonuniformity produced at cartridge position No. 1 by the original heaters complements, to a certain extent, the imbalance caused by the melt replenisher heater to the left of this cartridge. A return to the original heaters made replenished growth possible once again in runs 226 onward. Thermal conditions at position No. 3 in the hot zone were found to be satisfactory for full-width growth in run 230, whereas the growth performance of position No. 2 could not be assessed in runs 230 and 231 because of other equipment problems.

At the time of run 225, the full set of equipment comprising the automatic growth control system became available, so the further experimentation with profiled cold shoes was postponed until run 232. The version used in this interval consistently allowed stable growth at around 3.0 cm/minute, which was also the maximum practical growth rate for the previous standard non-profiled shoes. The difference in behavior between the non-profiled and profiled cold shoes is that with the former, "freezes" invariably occurred at the ribbon ends, and long-term stability was poor, while with the latter, "freezes" occur with approximately equal frequency at the end and central positions of the die when the growth rate is raised beyond the practical limit (with simultaneous readjustments of heater power). The small amount of experience with the thicker version of the profiled cold shoes on which we can report at this time indicates that these perform in a qualitatively similar way, with an optimum growth rate of 3.8 to 4.0 cm/minute.

2. Automatic Controls Development

Following completion of the video analysis circuitry, which derives information on the geometric features of the growth meniscus from a TV camera's output, the remaining components of the complete control system were built during this quarter. The overall system is shown schematically in Figure 2. The newly-constructed portions are as follows:

a) Cartridge temperature control panel: This is similar to the previously used panel, but with added wiring to connect the outputs of the optical controller to the end heater power controllers and to the set-point terminal of the face heater temperature controller. The face heater temperature controller is newly connected to the rest of the system through isolation amplifiers, to reduce the influence of ground currents in the thermocouple circuit.

b) Operator's control panel: The new version is similar to the previous one, but toggle switches have been added for connecting the optically sensed feedback signal to the left, center, and right die-heater control circuits. The manual control potentiometers and startup ramp circuit for the end heaters were rearranged so that their functions are more logical from the operator's point of view.

c) Controller circuit card: A controller circuit card was built to produce a control signal proportional to the difference between the position signal from the video analysis circuitry, and a reference input for each channel. As the control system is developed, additional control functions may be built onto the card, e.g., filters, P-I-D control, alarms, etc.

When mounted to the machine, the equipment was found to operate without any difficulties due to electromagnetic noise pickup.

In this initial period of experimentation with the control system, the loops to control end-heater power were outfitted with deviation amplifiers with gains adjustable over a range of one to five. Combined with

Automatic Ribbon Growth Control System

FIGURE 2

the transducer gain of the video analysis unit and the current gain of the end-heater power supplies, this range of amplification gave a control gain of between 14 and 70 end-heater amperes per centimeter of deviation of ribbon position. Given a typical end-heater operating point of 45 amperes, the control gains at these two settings of the error amplifier, expressed in terms of percent power (current squared) per increment of edge-position deviation, were 0.6 to 3.1 percent power per 0.01 cm. The unit of 0.01 cm is chosen for this expression because it is approximately the edge-position control accuracy which is required of the automatic width control system. The center channel was not used to control the face heater to date.

Experimentation was begun in run 225 with control gains set at the lower values; these were gradually increased and the edge-position signals were observed for a tendency for oscillatory instability. None was observed, even at the highest gains in this range, so these gain settings were used throughout the remaining runs of the period. In the following run (226), an attempt was made to perform an open-loop frequency-response measurement on one end of the ribbon while the other was maintained by closed loop control. The data obtained were not of sufficient accuracy to construct a satisfactory frequency-response plot. There exist in the overall system at least two strong noise sources with frequencies in the range of interest, which made the interpretation of the data difficult.

The automatic growth control system was used in run 229 to make another type of measurement which may be more useful in the near term. The performance of the die/end heater/cold shoe combination was characterized by measuring the end-heater power for steady-state growth as a function of ribbon edge position. The local slope of the power vs. position curve indicates the regions on the die of stable and unstable edge position in open-loop control. These measurements could provide useful guidance for optimization of these components. Additional measurements will be made in the near future, time permitting, to assess the repeatability of the technique.

In run 227, the usefulness of the width control system to aid the operator in growth startup was assessed. Instead of manually controlling

end heaters to spread and stabilize the ribbon at full width before switching-on the signals from the video analysis circuitry, the operator employed the width controller to move the ribbon edges to the correct positions on the die ends by shifting the video-analysis windows on the TV screen. The controllers for the ribbon ends operate to maintain the edges at fixed positions within the windows. The windows can be shifted at will, subject to the limitation that moving them outward with the control loop "on" causes the controller to cool the die ends. Ribbon at less than full width grows with a very low meniscus and therefore the die is within a degree or two of the freezing point. Hence, the windows must be moved by very small increments. Slow shifting of the windows, or the reference points within the windows, could be performed electronically, and this would be a key element of a semi-automatic growth startup system which might considerably reduce the operator's required skill level.

The operator's task in using the auto control system was perceived as unnecessarily complicated in the first few runs. A pair of potentiometers was originally used to establish the reference points within the video windows, and analog meters were provided to permit the output of each error amplifier to be nulled just before closing the control loop. To eliminate this manual nulling procedure, "automatic setpoint tracking" circuitry was designed which digitally remembers the position within the video analysis window occupied by the ribbon edge at the time the auto control switch is turned on, and defines this as the operating point or reference input.

The long-term performance of the auto control system was demonstrated in run 228 by a duration of completely unattended growth of nearly 3.5 hours, which ended when the ribbon became caught under the overhead structure of the furnace. The signal from the central meniscus-position window was recorded during this period in an attempt to assess its usefulness for controlling the face heater. One of the principal effects seen was the sensitivity of the video analysis system to silicon carbide particles, which appear as white patches in the dark meniscus and create errors in the position signal. There is not much contrast available in the meniscus, and hence the setting of the video quantization level is critical. Further experimentation is needed to show whether or not the long-term

stability of growth can be improved by optical sensing of center meniscus height. Our plans for development of the control system in the near future also include operation with higher control gains and proportional-plus-integral control.

D. Multiple Growth Runs

Three-ribbon simultaneous growth was attempted twice in June to demonstrate the recent advances in the technology. The first version of the profiled cold shoe was duplicated to update the two additional cartridges to the configuration of the one used for development.

Throughput data on the first of these runs, shown in Table V, illustrate the role of the automatic growth control system installed on Station 1 only. This cartridge operated continuously for the duration of the run, with occasional checking for optimum positioning of the ribbon ends on the die bulbs. Cartridge 3 also operated very well with only one freeze soon after startup, but with operator checks at intervals of five to ten minutes, and a number of readjustments of the end heaters. The run was foreshortened by the failure of the melt replenishment system. Cartridge 2 was assembled with mounting pins for the linear cooling plates inserted too far and blocking the ribbon slot. An attempt was made to repeat this run after correcting these faults, but in run 231 a number of additional problems occurred. One of these was the use of dies with smaller tip width than those used in 230. With these, a less uniform meniscus-height profile could be established. Full-width growth could not be achieved in Cartridges 2 and 3.

E. Plans for Next Quarter

The goals of this project include a "combined technical features" demonstration by the end of July. Substantial progress remains to be made in two areas before the required levels of throughput and solar cell efficiency can be demonstrated by this machine. First, the cartridge must be developed for stable growth at 4.0 to 4.5 cm/minute. The cold shoe set operated in the last run of this quarter appears to increase the optimum growth rate to nearly 4.0 cm/minute; further evaluation will be carried

TABLE V

MULTIPLE GROWTH RUN 16-230Run Duration (minutes): 208

	<u>Cartridge No. 1</u>	<u>Cartridge No. 2</u>	<u>Cartridge No. 3</u>
Length of ribbon grown (m)	6.3	0*	5.5
Length $\geq$ 10 cm wide ribbon (m)	6.3		5.3
Percent $\geq$ 10 cm wide ribbon	100		96
Total growth time (minutes)	208		199
Longest growth time (minutes)	208		196
Number of freezes	0		1
Average growth rate (cm/minute)	3.03		2.76
Percent of run time operating	100		95.6

* Cartridge No. 2 was not operable due to assembly error.

out. Second, the machine needs to be operated in a routine, full-clean mode for several runs to assess the solar cell efficiency of the resultant material. The means for enhancing the photovoltaic output of this material through the introduction of oxygen will have to be developed in techniques applicable to the multiple ribbon machine. The usefulness of this approach has recently been demonstrated in preliminary fashion in single-station Furnace 17. These tasks will not be completed by the end of July, but will continue to be the focus of our efforts during the next quarter.

IV. CELL AND MATERIALS CHARACTERIZATION

A. Cell Characterization by C.T. Ho

1. Overview

The solar cell results of a repeat ambient gas experiment, run 18-199, have shown that the photovoltaic properties of the ribbon grown from the controlled CO₂ ambient environment are indeed reproducible.

Another experiment involving the flow of H₂O vapor into the furnace system during growth has not produced particularly clear effects on the solar cell characteristics. Efforts to introduce CO₂ in Furnace 17, however, have not yet shown positive results in terms of solar cell properties.

B. Detailed Description of Experimental Results

1. Ambient Gas Experiments in Machine No. 1

Run 18-199 was a repeat of growth with a CO₂ ambient. The purpose of the run was to see if the photovoltaic characteristics of the CO₂-on/CO₂-off ribbons had the same respective quartz-like/graphite-like behavior as shown in the previous experimental run, 18-191.

Representative 5 x 2.5 cm² ribbon blanks were selected from the CO₂-on and CO₂-off portions of the ribbons and processed into solar cells by both PH₃ and CVD diffusion procedures. The cell output data under ELH simulated measurement are summarized in Table VI. All the photovoltaic parameters were nearly identical to these. In 18-191, the quartz-like behavior of the CO₂-on portions manifested itself clearly in both types of the processing groups. Here we confirm once again that one can improve the solar cell quality of the RH-grown material with PH₃ processing by introducing CO₂ gas into the growth system. The growth process is also shown to give reproducible results with respect to the final solar cell output characteristics.

Another ambient gas experiment has been conducted in growth run 18-180. In the run, H₂O vapor mixed with Ar carrier was introduced above the menis-

TABLE VI

SOLAR CELL DATA OF RUN 18-199

(REPEAT OF 18-191)

ELH LIGHT, 100 mW/cm², 28°C, AR COATED, AREA = 14 cm²

Process	Ambient Condition	J _{sc} (mA/cm ²)	V _{oc} (V)	FF	η (%)
PH ₃	CO ₂ off	22.2	.523	.738	8.7
		18.7	.499	.723	6.8
		22.7	.530	.732	8.9
		18.9	.501	.731	7.0
		23.1	.534	.760	9.4
	CO ₂ on	28.0	.570	.721	11.6
		28.8	.580	.698	11.8
		28.1	.574	.717	11.7
		26.4	.562	.695	10.4
		26.9	.565	.730	11.2
		27.5	.573	.727	11.6
		26.3	.560	.730	10.4
		27.3	.573	.714	11.3
		26.1	.562	.739	11.0
		26.1	.560	.741	10.9
		25.2	.555	.738	10.4
		26.2	.563	.765	11.4
CVD	CO ₂ off	26.5	.558	.709	10.6
		26.7	.559	.702	10.6
		26.4	.559	.666	9.9
	CO ₂ on	27.8	.567	.688	11.0
		26.7	.559	.632	9.5
		25.9	.551	.692	10.0
		26.1	.553	.699	10.2
		26.1	.554	.715	10.4
		26.9	.548	.653	9.7
		26.5	.565	.732	11.1

cus in the same manner as in the CO_2 experiment. From the IR transmission measurement, the interstitial oxygen concentration was found $\sim 5 \times 10^{17} \text{ cm}^{-3}$ in the H_2O -on grown portion, and $< 5 \times 10^{16} \text{ cm}^{-3}$ in the H_2O -off grown portion of the ribbon. Solar cells of $5.5 \times 2.5 \text{ cm}^2$ size were then fabricated by the PH_3 diffusion process. The cell output data under simulated AM1 condition are summarized in Table VII. These results are quite erratic, and a further exploration of the effect of H_2O needs to be made. It is very likely, though, that the erratic nature of the results is due to the fact that the H_2O interacts with carbon parts in the growth setup to produce CO , which then acts on the growing ribbon.

2. Ambient Gas Experiments in Machine 17

Several CO_2 ambient growth runs have also been made in the high-speed, 10 cm growth System 17. In runs 17-084 through 17-087, the ribbons were grown with the CO_2 gas being introduced by mixing the gas with the cartridge purging Ar gas. In contrast to a heavier film on the ribbon surface grown from Machine 18 under the CO_2 -on condition, the ribbons showed only a light hazy film on the surface. No interstitial oxygen was detected in the IR absorption measurement at $\lambda = 9 \text{ }\mu\text{m}$. The oxygen concentration detection threshold was about $5 \times 10^{16} \text{ cm}^{-3}$ in this material.

Cell fabrication was made concurrently with material from 18-199. As shown in Table VIII, there was a limited improvement in J_{sc} and V_{oc} in the CO_2 -on group as compared with the CO_2 -off group after the PH_3 processing. By comparing the results of the PH_3 -processed group with the CVD-processed group, we conclude that the ribbon is predominantly graphite-like. It is possible that the CO_2 gas has not been effectively transported into the silicon melt. The same speculation was also made after the growth run and already discussed in the First Quarterly Report.

B. Materials Characterization by J.F. Long

1. Overview

Both Furnaces 1 (18) and 3A (16) have been operated in developmental modes; only a few runs have been examined for material quality eval-

TABLE VII

SOLAR CELL DATA OF RUN 18-180. THE CELLS
WERE FABRICATED FROM PH₃ DIFFUSION PROCESS

ELH LIGHT, 100 mW/cm², 28°C, AR COATED, AREA = 14.5 cm²

Ambient Condition	J _{sc} (mA/cm ²)	V _{oc} (V)	FF	η (%)
H ₂ O off	21.8	.525	.734	8.4
	22.4	.515	.728	8.4
	21.1	.515	.750	8.2
	22.6	.517	.725	8.5
	22.9	.523	.700	8.4
H ₂ O on	24.3	.535	.640	8.3
	23.9	.542	.714	9.2
	23.2	.533	.690	8.5
	24.8	.545	.678	9.2
	27.5	.565	.650	10.1
	24.5	.551	.656	8.9
	24.7	.543	.648	8.7
	24.2	.541	.652	8.5
	26.8	.566	.718	10.9
	27.8	.574	.735	11.7

TABLE VIII

SOLAR CELL DATA FOR RIBBONS
GROWN FROM FURNACE 17

100 mW/cm², ELH LIGHT, 28°C, AR COATED

Run No.	Ambient Condition	Process	J _{sc} (mA/cm ²)	V _{oc} (V)	FF	η (%)	Notes
17-084	CO ₂ off	PH ₃	23.6	.533	.710	9.0	Cell area 55 cm ²
			24.6	.531	.635	8.3	
			22.6	.531	.615	7.4	
		CVD	24.4	.544	.621	8.2	
			23.5	.546	.590	7.6	
			22.4	.545	.548	6.7	
	CO ₂ on	PH ₃	25.1	.542	.690	9.4	
			24.3	.541	.727	9.6	
			23.3	.522	.610	7.4	
		CVD	25.2	.551	.568	7.9	
			23.9	.549	.721	9.5	
			24.4	.546	.719	9.6	
17-085	CO ₂ off	PH ₃	19.4	.494	.714	6.9	Cell area 14 cm ²
			19.7	.500	.701	7.0	
			19.6	.487	.674	6.5	
		CVD	25.0	.547	.721	10.0	
	CO ₂ on	PH ₃	18.7	.495	.674	6.3	
			20.2	.497	.717	7.4	
			20.0	.503	.710	7.2	
			22.4	.515	.679	7.9	
			22.7	.517	.728	8.6	
		CVD	25.4	.550	.654	9.2	
			25.7	.551	.690	9.9	
			24.9	.541	.644	8.8	
			24.7	.542	.714	9.7	

TABLE VIII continued.

Run No.	Ambient Condition	Process	J_{sc} (mA/cm ²)	V_{oc} (V)	FF	η (%)	Notes
17-086	CO ₂ on	PH ₃	23.1	.533	.670	8.3	Cell area 50 cm ²
			25.2	.549	.672	9.3	
			24.5	.510	.665	8.8	
			24.0	.544	.724	9.4	
			24.1	.538	.677	8.8	
		CVD	26.2	.560	.650	9.5	
			25.5	.555	.731	10.3	
			26.0	.558	.671	9.7	
			25.0	.547	.715	9.8	
			25.4	.543	.648	8.9	
	CO ₂ on	PH ₃	23.5	.524	.708	8.7	Cell area 55 cm ²
			23.1	.525	.683	8.3	
			21.3	.512	.625	6.8	
			21.8	.518	.629	7.1	
			21.1	.511	.668	7.2	
		CVD	25.1	.544	.676	9.2	
			25.2	.553	.671	9.3	
			24.3	.547	.614	8.1	
			25.2	.547	.685	9.4	

uation. The bulk of the material characterization has been performed on ribbon grown in Furnace 17, in which quality experiments have included growth speed and ambient composition variations.

2. Detailed Description of Results

a. Furnace No. 1 (18)

Two runs were examined this quarter. The first was the last in a series of ambient experiments during 5 cm wide growth; the second was the first clean attempt at 10 cm wide growth in this furnace.

Run 18-207 involved the introduction of hydrogen gas into the interfacial region through the gas jet. Minority carrier diffusion lengths are tabulated below:

Sample No.	Main Zone Argon Flow Rate (ℓ /minute)	1% H ₂ in Argon Flow Rate (cc/minute)	$\bar{L}_D$ (μ m)
18-207-1G	10	0	28.4
-2H	10	500	31.2
-4A	4	500	45.9

As there was little improvement in sample 18-207-2H, it seems more likely that the elevated diffusion lengths in sample 18-207-4A are due to the decreased main zone purge rate than to any effect of the introduction of hydrogen.

Run 18-213 was the first attempt at clean 10 cm wide growth in Furnace 1. The diffusion lengths were low, with an average minority carrier diffusion length of 9.0 μ m.

b. Furnace No. 3A (16)

Three runs were examined this quarter. All three runs produced 10 cm wide material. Runs 16-227 and 16-228 were both single-cartridge runs which employed automatic width control (see Section III). Six samples from each of these runs, spanning several hours of growth, were examined for average minority carrier diffusion length. The results, tabulated as follows, indicate no change in diffusion length as a func-

tion of run time. The average diffusion lengths for each run (22.4 and 20.7, respectively) are consistent with prior Furnace 3A material performance.

Sample No.	$\bar{L}_D$ (μm)
16-227-3B	22.4
-3C	22.8
-3F	20.9
-3H	26.2
-3J	21.1
-3L	21.2
16-228-4C	25.0
-4D	19.8
-4E	20.6
-4F	18.6
-4G	20.0
-4H	19.8

Run 16-230 was a multiple cartridge run. Cartridge 1 was controlled automatically, while it was intended that Cartridges 2 and 3 be operator-controlled. Cartridge 2, however, produced no material, as improper assembly precluded growth. Two samples from each of Cartridges 1 and 3 were examined. The average minority carrier diffusion length for the entire run (24.4 μm) is again consistent with prior levels in this furnace.

c. Furnace No. 17

The series of runs undertaken this quarter included several runs which investigated the effects of growth speed on material quality, several which examined the effects of the introduction of CO_2 in argon into the interfacial region of the growing ribbon, and one which was undoped. Additionally, in several of the runs minor die design changes were made in an attempt to improve growth stability. No effects on material quality were observed as a result of any of these die alterations.

The runs which involved growth speed variations are summarized in Table IX. These show that the diffusion length averages achieved at speeds

TABLE IX

SUMMARY OF GROWTH SPEED
EXPERIMENTS, FURNACE 17, SECOND QUARTER 1980

Run No.	Growth Speed (cm/min)	$\bar{L}_D$ (μm)
17-091	3.7	20.9
-092	3.8 - 4.0	29.9
-093	3.8 - 4.0	37.8
-096	3.7	24.6
-097	3.7	35.3
-102	3.5	27.8
-103	3.7	27.0
-104	3.7 - 3.8	22.5
-106	3.8	36.6
-108	3.0	34.2
-109	3.7 - 3.8	23.8
-110	3.8 - 4.0	27.5

of up to 4.0 cm/minute are among the best performance of any materials grown to date in a cold shoe system.

The runs which involved the introduction of the argon-CO₂ mixture are summarized in Table X. In each case, and especially at the higher flow rates, the introduction of this gaseous mixture appears to beneficially affect the minority carrier diffusion length. This effect is well documented in Furnace 1 (18).

Some question, however, has arisen regarding the uniformity with which the gaseous mixture is being delivered to the interfacial region in this furnace. The gas delivery configuration is treated more extensively in Section II of this report. Figure 3 demonstrates two things: First, that the "CO₂-on" samples are in fact superior to the "CO₂-off" case (see also Table X), and second, that there exists a large "hole" in the diffusion length spatial profile for all the "CO₂-on" samples approximately 4 cm from one edge. It is felt that this result reflects nonuniformity in the current gas introduction scheme.

Finally, run 17-107 was grown without the addition of boron dopant. The average resistivity (19.1 Ω -cm) corresponds to $\sim 6.5 \times 10^{14}/\text{cm}^3$ acceptor sites, which may be due to residual boron in the system. The spatial resistivity profile was reasonably uniform, which tends to reinforce this possibility (boron will not redistribute under the influence of fluid flow patterns; aluminum, another acceptor, will do so quite readily, resulting in a more inhomogeneous spatial resistivity profile). The average diffusion length for this run (47.5 μm) was high.

d. ESCA Results on Ambient-Induced Film-Bearing Samples

Results are reported here of ESCA (electron spectroscopy for chemical analysis) which was performed on two film-bearing samples grown in Furnace 1 (18). The two samples were submitted to Photometrics, Inc., of Lexington, Mass., for the analysis. Sample No. 1, hereafter referred to as "wafer No. 1," was a 1 cm x 1 cm section of sample 18-186-2H, which was grown under reduced main zone argon flow. Sample No. 2 ("wafer No. 2") was a similarly sized section of sample 18-199-2G, grown with CO₂ introduced into the interfacial region via a gas jet.

TABLE X

SUMMARY OF AMBIENT EXPERIMENTS,
FURNACE 17, SECOND QUARTER 1980

Run and Sample No.	(1% CO ₂ + Argon) in Ar Flow Rate (ℓ /min)	$\bar{L}_D$ (μm)
17-098-1A	0	16.3
-1B	0.5	27.2
-1C	1.0	27.0
-1D	2.0	29.9
17-099-1A	1.0	37.0
-1C	3.0	43.6
17-100-1A	2.5	26.4
-1B	2.5	38.0
17-111-2A	1.0	20.6
17-112-1A	0.5	35.5

FIGURE 3

Spatial distribution of minority carrier diffusion lengths for run 17-098 (CO₂ experiment)

The elemental compositions in atomic percent estimated from the data are as follows:

	<u>C</u>	<u>O</u>	<u>Si</u>
Wafer No. 1	43	20	37
Wafer No. 2	13	59	28

High resolution spectra on the C(1S), Si(2P), and O(1S) peaks indicate the following allocations:

	<u>C</u>			<u>O</u>		<u>Si</u>		
	<u>C(O)</u>	<u>C(H)</u>	<u>SiC</u>	<u>SiO₂</u>	<u>O(C)</u>	<u>SiO₂</u>	<u>SiC</u>	<u>Si</u>
Wafer No. 1	2	12	28	17	2	5	28	6
Wafer No. 2	4	9	-	55	4	28	-	-

In this table, O(C) and C(O) are ether or hydroxy type O and C, and C(H) is environmental hydrocarbon.

The principal difference between the wafers is the additional SiO₂ on wafer No. 2 which replaces some near-surface SiC on wafer No. 1.

The presence of 17% O as SiO₂ and only 5% Si as SiO₂ in wafer No. 1 (the ratio should be 2:1) can be accounted for on the hypothesis that the SiO₂ is a thin layer, probably less than 100 Å thick. The lower kinetic energy of the O(1S) electrons means that this line will be relatively enhanced in a thin layer. These data are the result of an examination of the top 20 Å of the film, and cannot, therefore, be construed as complete characterization of the film in either case. However, these data agree qualitatively with prior analyses of these films done with infrared spectrophotometry and ellipsometry.

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APPENDICES

1. Updated Program Plan

The original program plan, as amended, is still in effect.

2. Man Hours and Costs

Previous cumulative man hours were 89,581 and cost plus fixed fee was \$3,183,144 through March 31, 1980. Man hours for the second quarter of 1980 are 6,151 and cost plus fixed fee is \$277,584. Therefore, total cumulative man hours and cost plus fixed fee are 95,732 and \$3,460,728, respectively, through June 30, 1980.

3. Engineering Drawings and Sketches Generated During the Reporting Period

See Figure 2, Section III.

4. Summary of Characterization Data Generated During the Reporting Period

See Section IV.

5. Action Items Required by JPL

None.

6. New Technology

Some of the efforts reported here may have generated items of potentially "new" technology. If so, they will be separately reported, pending possible patent action.

7. As-grown material sent to JPL.

APPENDIX 7

AS-GROWN MATERIAL SENT TO JPL

Ribbon 18-199-2H

Grown in JPL No. 1, no cold shoes

Clean system, doped to 1 Ω -cm, p-type

Graphite crucible

Growth speed: 2 cm/min

Displaced die, back marked with "X" in top left-hand corner

Sample size: 5.5 cm x 2.5 cm

Ambient conditions: main zone 3 ℓ /min; 1% CO₂ in argon mixture
300 cc/min

AS-GROWN MATERIAL SENT TO JPL

Ribbon 18-191-5F

Grown in JPL No. 1, no cold shoes

Clean system, doped to 1 μ -cm

Graphite crucible

Growth speed: 2 cm/min

Displaced die, back marked with "X" in top left-hand corner

Sample size: 5.5 cm x 2.5 cm

Ambient conditions: main zone 10 ℓ /min; 1% CO₂ in argon mixture
300 cc/min

AS-GROWN RIBBON SAMPLES FOR JPL
(from Furnace JPL No. 1)

Material

12 blanks 10 cm x 5.5 cm = 660 cm²

6 blanks 5 cm x 5 cm = 150 cm²

TOTAL 810 cm²

Run Data

Run 18-205 grown in JPL No. 1, no cold shoes

Clean system, doped to 1 Ω -cm, p-type

Graphite crucible

Displaced die; low meniscus side marked by "X" scribed in corner

Growth speed: 2 cm/min

Ambient conditions: main zone flow 10 ℓ /min, 1% CO₂ in argon mixture
400 cc/min in growth slot