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Mobil Tyco Solar Energy Corporation
16 Hickory Drive
Waltham, Massachusetts 02254

LARGE AREA SILICON SHEET BY EFG

Program Manager: Fritz V. Wald

Fourth Quarterly Report - Subcontract 954355

Covering Period: October 1, 1980 - December 31, 1980

February 17, 1981

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ABSTRACT

Extensive characterization of the multiple ribbon Furnace 3A main zone temperature profile has been performed and the information used to improve uniformity of heating. Irregularities in the main zone heater have been associated with growth difficulties at specific cartridge locations, and growth conditions subsequently improved by profiling the main zone heater. This work has resulted in good growth conditions being established in all three cartridge positions. These improvements have allowed multiple growth of three 10 cm wide ribbons to be demonstrated for periods of an hour on several occasions. A full eight-hour technical features demonstration run has not been achieved due to malfunctions in auxiliary equipment.

A new gas distribution system for the 10 cm cartridge has been introduced and demonstrated to lead to improved ambient control during growth. Growth without and with CO_2 has shown that quality improvement in 10 cm ribbon grown with cold shoes results from ambient manipulation. Optimization of this process is continuing with studies of different means by which to vary oxygen levels in the ribbon.

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I. INTRODUCTION

In the past year, all essential elements have been demonstrated in accordance with the 1986 LSA project goals to qualify EFG multiple ribbon technology as a candidate for production of low-cost substrates for terrestrial solar cell fabrication. Solar cells of efficiencies over 13% have been prepared from resistance furnace-grown ribbon, 10 cm wide ribbon has been grown at speeds of 4 cm/minute, and automatic controls have been developed and successfully used during growth of 10 cm wide ribbon in a multiple ribbon furnace. It remains to integrate all these demonstrated elements into a single multiple-ribbon furnace for 10 cm wide ribbon that is the prototype unit of a large-scale facility for production of EFG silicon ribbon at the low costs necessary to achieve the LSA project goals. The milestone toward completion of the integration task scheduled for 1980, referred to as the "technical features demonstration," has not yet been achieved, however, and so the program is behind schedule. Nevertheless, the necessary information to accomplish this milestone has been gathered, and suggests that it can be achieved by design iterations on the equipment presently in use; that is, there is no technical basis for believing that the obstacles are of a fundamental nature. This view is better expressed by considering in more detail the progress that has been made in the various areas of development of the EFG prototype multiple growth units and related work.

By the end of 1979, the program had made significant progress toward a technology for production of silicon ribbon by the EFG technique that is capable of being scaled to levels required for low-cost substrates for solar cells. Growth of five ribbons, each 5 cm wide, with continuous melt replenishment for 15 hours was demonstrated, with a total output of 150 m of ribbon at speeds of 3.5 cm/minute and a machine duty cycle of over 90%. Growth of 10 cm wide ribbon in an upscaled version of the basic system had been shown to be viable, and the fundamentals of an automatic control system evolved to a point of understanding sufficient to permit construction of prototype units. Cell efficiencies up to 11.5% (AM1) had been demonstrated for large areas (~50 cm^2) for ribbon grown in single-ribbon (cartridge) furnaces at speeds of the order of 2 cm/minute, and cell efficiencies for ribbon grown in the more complex multiple-ribbon furnace at speeds of up to 4 cm/minute averaged 9%.

The work in the past year accordingly focused on development of a multiple ribbon system for 10 cm wide ribbon capable of growing three ribbons with continuous melt replenishment and automatic control capabilities, and on gaining further understanding of factors that influence material quality in high-speed growth. The technical features demonstration requirement for the multiple ribbon furnace for 1980 was for a run length of eight hours, a growth rate of 4.5 cm/minute, a machine duty rate of 85% or better, operational automatic controls on one ribbon, and a ribbon quality sufficient for a 10.2% cell efficiency. In addition, an efficiency-related goal of the demonstration of a 13+% cell was required. Significant developments related to the work toward these goals which occurred in this past year have been:

(i) Characterization and development of the 10 cm cartridge to gain a better understanding of the parameters that control the die top isotherms, hence growth stability and the maximum speed capability. Reproducible growth conditions and greatly increased stability and control over ribbon width have resulted, and growth at speeds of 3.5 to 4.0 cm/minute is routinely possible with a control level sufficient to make multiple ribbon growth feasible.

(ii) A complete automatic control system for ribbon width control has been demonstrated. It has been proven highly successful in routinely allowing growth of 10 cm wide ribbon over time spans of the order of hours with minimal operator intervention. This system has been now installed for use with all three 10 cm cartridges of the multiple-ribbon furnace.

(iii) Improvements of cell efficiencies to the range of 10 to 11% have been demonstrated for 10 cm wide ribbon grown at speeds of up to 4 cm/minute. Although all factors relevant to quality improvement at higher speeds are not yet understood, the ambient composition influence demonstrated in a lower speed growth system, and which has now been used to achieve cell efficiencies over 13% there, has also been shown to be operative in the case of higher speed growth. Optimization of these effects and study of additional factors which may have an important influence has proceeded through the utilization of computer modeling to study interface shape effects and mass transport phenomena in the meniscus.

(iv) Important processing effects on solar cell efficiency have been demonstrated. These show the need for the presence of oxygen during the growth process in ribbon pulled from melt contained in graphite crucibles, combined with special thermal treatment sequences during the processing, in order to maximize cell efficiencies in this ribbon. With proper attention to these factors, small (~6 cm^2) cells of over 13% have been prepared from resistance-furnace cartridge-grown ribbon.

Several factors have prevented the technical features demonstration, with incorporation of the above developments into multiple 10 cm wide ribbon growth, from achieving required performance levels. Simultaneous growth of three 10 cm wide ribbons for periods of several hours has been demonstrated, but at speeds and duty rates well below those desired. Currently, solar cells made from this ribbon are in the 8% range. Unexpected irregularities in the multiple furnace main zone and interaction between thermal fields in it and the cartridge, viz., die-top isotherms, are the main causes for reduced growth stability that result in lower speeds and productivity. Seed ribbon breakage has been an additional contributor to lower productivity. Work is being continued on finding tractable solutions to remedy these situations, as well as characterizing cartridge configurations that have produced ribbon at the desired speeds and with the necessary growth stability. With respect to the quality, the design changes needed to achieve control over the growth ambient have not been implemented because of the continued state of development of the multiple furnace during preparation for technical features demonstrations. In summary, the differences between the individually demonstrated goals in single-cartridge growth and the multiple-ribbon furnace performance have well-defined origins, which are believed to be rooted in the inadequacy of the present design of the multiple ribbon furnace main zone configuration. The confidence that this shortfall and related program tardiness can be resolved simply by design iterations is consequently very high.

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

A. Overview

Improvements have been made in the gas distribution system in the 10 cm cartridge to facilitate experiments in the study of ambient influence on material quality. A new die-top shield has been tested and shown to lead to a uniform distribution and better control of test gases in the growth interface environment. Ribbon has been grown with CO and CO_2 ambients.

Upgrading of power supplies has been undertaken for both Furnaces 17 and 18 to improve the reliability of operation. New main zone and afterheater power supplies with redesigned control circuits have been ordered.

Standardization of cartridge and main zone components in all the 10 cm wide ribbon growth systems has been started to reduce as much as possible existing design differences in these furnaces. This effort at present is directed at building up an inventory of standard components, and investigation of the influence of design differences in dies, cold shoes and main zone insulation that are not of a standard design in these furnaces. Interchange of cartridges among the three 10 cm ribbon systems has shown that these differences result in growth condition variations which make comparisons of performance difficult.

B. Experimental

An improved gas distribution system has been implemented in the 10 cm cartridge, where the wider ribbon has made it difficult to obtain uniform gas penetration to the meniscus with the arrangement used in 5 cm wide ribbon growth. In initial trials, it was found that the maximum available growth speeds in these experiments were not as great as those attained previously, viz., 3.8 to 4.2 cm/minute. Several aspects of the initial shield design may be contributing to this situation and modifications are being considered to improve interface heat transfer so as to recover a higher growth speed capability. The experiments are summarized in Table I.

Continued testing and development of the 10 cm cartridge has revealed that the existing power supplies in Furnaces 17 and 18 are only marginally capable of providing the flexibility of operation required in these tasks. Increased face heater and afterheater power is needed when using larger cold shoes with a greater cooling capacity, for example. An increased frequency of supply failures has led to a decision to replace the main zone power supplies in both Furnaces 17 and 18. Afterheater transformers are also being replaced. This is particularly needed in Furnace 18, where the present power supply does not allow afterheater operation at a temperature high enough for stress-free ribbon growth.

Development work with the 10 cm cartridge in Furnace 18 was particularly impaired by power supply malfunctions in combination with the persistence of poor growth conditions. Part of the problem was due to the power supply inadequacy itself, as noted above. This often forced operation of the cartridge in a regime of parameters marginal for 10 cm wide ribbon growth. Interchange of identical cartridges among Furnaces 16, 17, and 18 has revealed other factors which also have been contributing to this situation. In particular, the initial design of main zone insulation was proven to be unsatisfactory. The design of this has now been changed to approximate more closely the insulation in use in Furnace 16, and this will be installed in Furnaces 17 and 18.

Good growth conditions were not consistently obtained in Furnace 18 operation until the use of a Furnace 16 cartridge with profiled cold shoes in runs 18-242 and 18-243 (see Table II). This showed that the increased cooling provided by this

TABLE I
RUN DATA FOR 10 CM WIDE RIBBON GROWTH IN FURNACE 17

Run No.	Speed Range (cm/minute)	Gas Flow Conditions (ℓ/minute) Main Zone	Cartridge	Comments
17-129	2.8 - 3.2	6 Ar	0.5 Ar	Rebuilt cartridge run to re-establish baseline.
17-130	3.0 - 3.6	6 Ar	0.5 Ar	Repeat of 17-129.
17-131	3.0 - 3.6	6 Ar	0.5 Ar	Start of series with melt doped to 4 Ω-cm. Baseline run.
17-132	3.4 - 3.5	6 - 12 Ar	0.5 - 1 Ar	Testing of residual gas analyzer for furnace gas composition measurements — unsuccessful due to sample line leak.
17-133	3.3 - 3.5	6 - 12 Ar	0.5 - 1 Ar	Repeat of 17-132. Sampling line problems as above.
17-134	2.5 - 3.3	6 Ar	0 - 2, 1% CO in Ar	First test of new die shield, baseline run with CO ambient.
17-135	3.5 - 3.8	6 Ar	0	Cartridge from Furnace 16. Good growth conditions.
17-136	3.C - 3.2	6 Ar	0.5 - 1, 1% CO_2 + 100 ppm O_2 in Ar	Repeat of run 17-134 with CO_2 and O_2 mixture in ambient.
17-137	3.0 - 3.3	6 Ar	0.5 - 1, 1% CO_2 + 100 ppm O_2 in Ar	Repeat of 17-136 with quartz added to melt.
17-138	3.0 - 3.3	6 Ar	0.5 - 1, 1% CO_2 in Ar	Repeat of 17-137 with CO_2 only in ambient.
17-139	3.5 - 3.8	6 Ar	0 - 1 Ar and 1% CO_2	Testing of high speed capabilities with new die-top shield.
17-140	2.8 - 3.5	6 Ar	0 - 1 Ar and He	Experimentation with argon and helium mixtures.
17-141	3.4 - 4.3	6 Ar	0	Growth with "standardized" cartridge with thicker profiled cold shoes in use in Furnace 16. Good growth conditions at 4 cm/minute.
17-142	3.6 - 3.8	6 Ar	0.5	Testing of "standardized" main zone insulation modified to conform to multiple hot zone design. Melt doped to 2 Ω-cm.

TABLE II

RUN DATA FOR 10 CM WIDE RIBBON GROWTH IN FURNACE 18 (JPL No. I)

Run No.	Comments
18-230	No growth. Insulation misalignment.
18-231	Good growth conditions. Operator training. 1.5 meters grown, crucible emptied. Growth speed from 2.8 to 3.2 cm/minute.
18-232	Repeat of 18-231.
18-233	No growth. Water supply failure.
18-234	Good growth conditions with modified die, no bulbous ends. Ribbon highly stressed due to inadequate afterheater power levels. Growth speeds of 3 to 3.5 cm/minute. Crucible emptied.
18-235	Growth conditions good in speed range of 3 to 3.5 cm/minute. Thin ribbon (~150 to 200 μm), stress levels reduced. Crucible emptied.
18-236	No growth, power supply malfunction.
18-237	No growth, water supply failure.
18-238	Poor growth conditions due to silicon spill on die shield.
18-239	Poor growth conditions. Hot die center.
18-240	No growth. Broken seed obstructed growth slot.
18-241	Erratic growth conditions, similar to run 18-239.
18-242	Test of cartridge from multiple Furnace 16. Excellent growth conditions. Run terminated early due to broken seed.
18-243	Repeat of run 18-242 with cartridge from Furnace 16. Excellent growth conditions in speed range 3.0 to 3.4 cm/minute. Edge stability good with die without bulbous ends.

design was very necessary to produce growth conditions comparable to those available in other furnaces. The impact of the cold shoes was thus similar to that demonstrated in Furnace 16. On the other hand, the non-profiled cold shoes had been used with success in Furnace 17 in early optimization studies, and shown to be capable of allowing growth speeds above 4 cm/minute there. With a better appreciation of the influence of the main zone insulation on growth conditions gained from experience, these differences in performance have now become more understandable.

A further development arising out of the experience gained in these latest experiments is the initiation of an effort to standardize cartridge and main zone components in use in the three 10 cm cartridge furnaces as much as possible. This has been started with the installation of as identical main zone insulation packages as is allowed by the furnace configurations in Furnaces 17 and 18. The process of standardization is being extended to cartridge components to the extent operating conditions will allow. This process is expected to be completed when the new power supplies for Furnaces 17 and 18 are operational.

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

A. Overview

Much of the work of this reporting period was directed toward resolution of the problem of irregular die-top isotherm profiles, which was encountered when attempts were made in August and September to operate cartridges in all three positions in this furnace. A large number of temperature and power measurements were made in the overall system to accumulate knowledge about the relationship between the design of components of the main furnace, and growth performance of the cartridges. These measurements guided the re-machining of existing main furnace heating elements to improve thermal conditions at cartridge positions 2 and 3. These modifications achieved the desired result, and in December two runs were made which included significant periods of simultaneous three-ribbon operation of the furnace.

B. Furnace Temperature Profiles

By the end of the third quarter, the evidence gathered in attempting to perform multiple 10-cm growth runs clearly indicated that local irregularities in the temperature profile of the main furnace were the factor preventing the cartridges in furnace positions 2 and 3 from operating properly.(1) During the early development of the 10-cm cartridge in single-ribbon operation utilizing position No. 1, a problem had at one time been encountered with a lateral gradient at the die top, caused by the heat from the adjacent melt replenishment unit. A modification of the main heating elements which effectively corrected the problem at cartridge position No. 1 was made at that time. However, it was not until quite recently that the temperature distributions at furnace positions 2 and 3 were seen also to be unacceptable for proper cartridge operation.

To effect a definitive solution to this problem, a large number of temperature measurements were made in the furnace, in three areas, as follows:

(i) "Dummy load:" The longitudinal temperature profile of the furnace has been measured many times in the past using a graphite bar installed in place of the crucible, and with the cartridge openings plugged with thermal insulation. For the recent set of measurements, additional thermocouple wells were drilled in the dummy load so that the temperature could be measured at five places per cartridge position rather than the previous three.

(ii) Die Tops: A die blank was drilled with nine thermocouple pockets and installed in a cartridge. For each set of these measurements made, this cartridge was operated in the three furnace positions on successive days. No silicon was melted, but in other respects the conditions closely simulated normal operation: All three cartridges and melt replenisher were in place, with heating power applied and with control settings used which were typical for growing conditions (except as further explained below).

(iii) Heating element segments: The temperatures of the individual segments of the multi-slotted heating elements were measured on two occasions, and visually observed on two other occasions. For these measurements and observations, the heaters were powered without the furnace's thermal insulation in place.

In general, the measurements made in these locations correlated well with one another, and with the observed growth difficulties. The primary purpose of these measurements and the alterations made to the main heating elements was to obtain satisfactory operation of the present furnace as quickly as possible. A secondary aim was to develop rules of thumb which could be used to design hot zone components for future furnaces which would produce better profiles and reduce the need for "cut and try" development. The most significant representative profiles taken in this period are shown in Figures 1 and 2, and are discussed in the following paragraphs.

Figure 1 is of the temperature distribution in the dummy load when heated by the set of heating elements in place during run 243. Only in cartridge 1 could the die-top isotherms be sufficiently balanced so that full-width ribbon could be grown. The two full-length traces in this figure were made at three points per cartridge location (in September) and at five points per cartridge location (in October), respectively. Also shown are the die top temperature measurements under two conditions: end heaters off, and end heaters on, at the "best effort" settings used in run 243. The highly asymmetric temperature distribution at 6 mm deep in the die top in cartridge position 2 is clearly related to the growth difficulties experienced with this cartridge. The asymmetry of the "end-heaters on" profile of position 1 could be adequately trimmed for full-width growth by readjustment of the growth controls, while the "end-heaters on" profile for position 3 appears to show a serious cold spot at the second point from the left end. This cold spot was also observed during growth attempts, and could not be overcome by adjustment of the heater controls.

Following the taking of the measurements of Figure 1, the heating elements were reworked and exchanged several times, additional profiles were taken, and growth runs were made, as listed in Table III. The dummy load profiles made on five of the seven heater sets utilized in this period are shown in Figure 2.

In order to alter the temperature distribution in a furnace of this design, where three heaters of slotted design are placed end to end and operated as a three-phase delta load, the

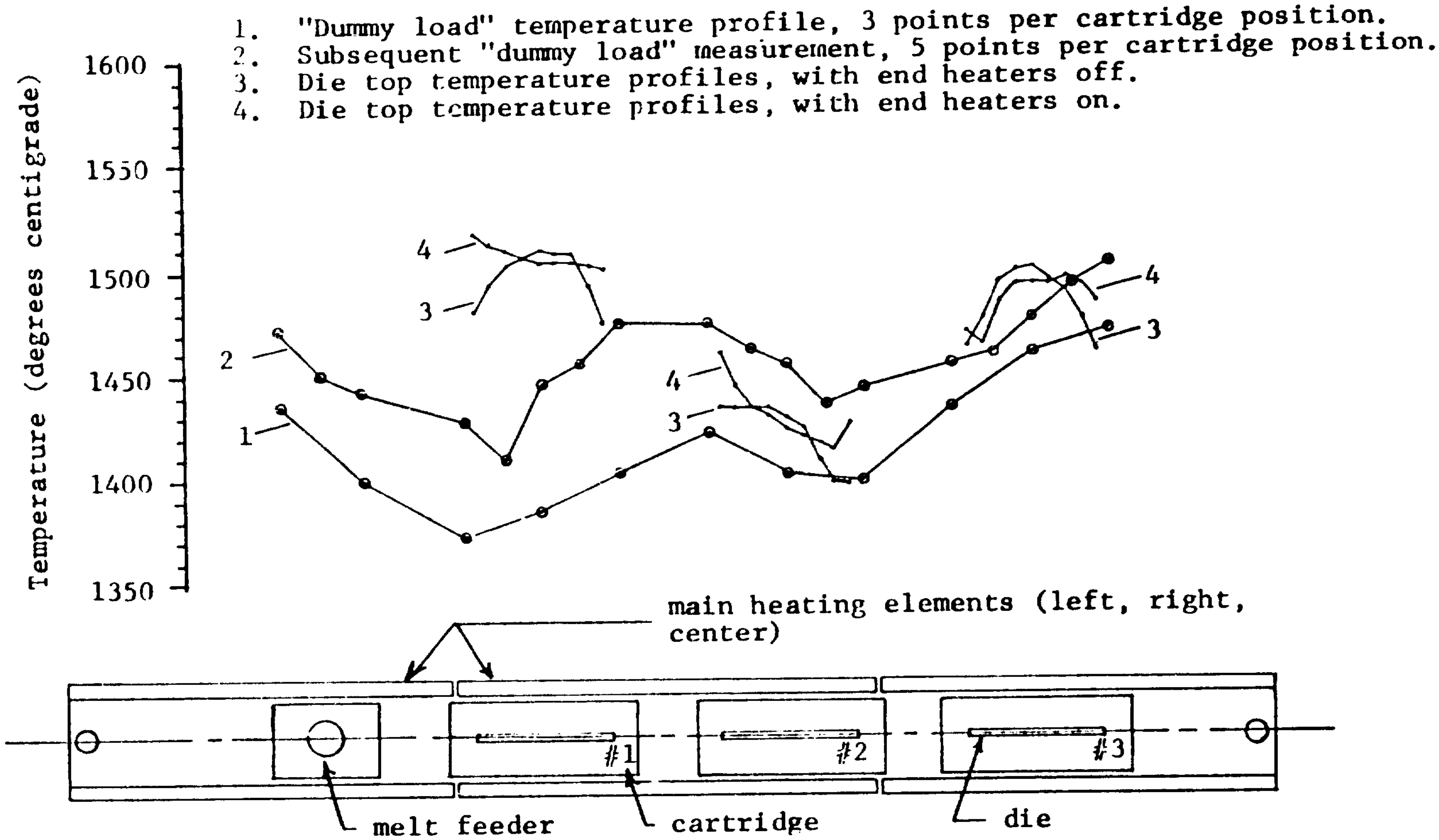

FIGURE 1
Temperature measurements for main heating elements used in run 243. See Table III and text.

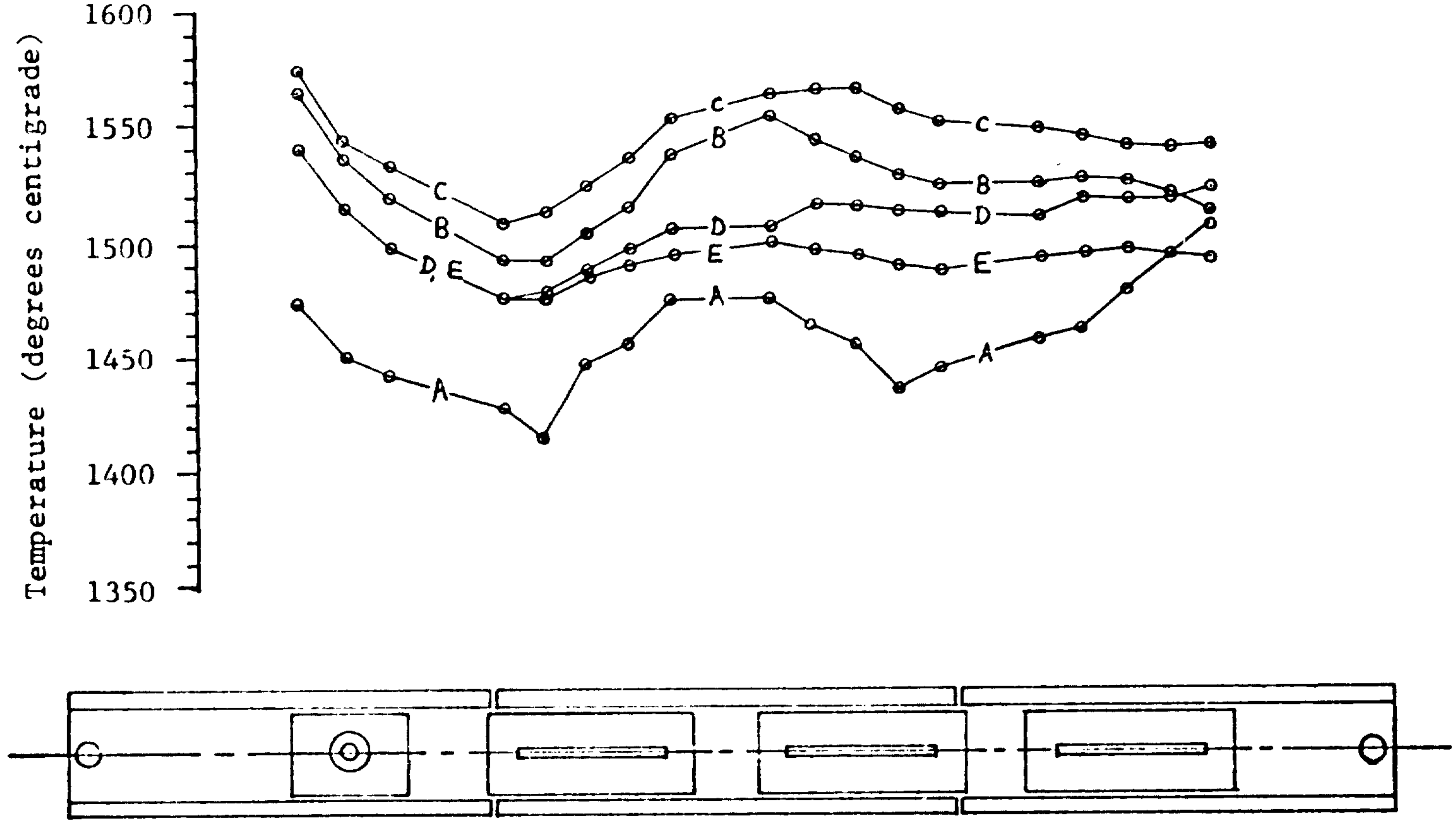

FIGURE 2
Temperature profiles for combinations of main heating elements listed in Table III.

TABLE III

MAIN HEATING ELEMENTS INSTALLED IN FURNACE 3A IN FOURTH QUARTER

Run Nos.	Profile in Figure 3	Heating Elements: Designation/Resistance (Milliohms)					
		Left		Center		Right	
243	A	L	24.86	X	20.90	ZZ	22.11
	B	L	24.86	X Mod 1	21.35	ZZ Mod 1	25.7*
244-245	C	L	24.86	X Mod 2	21.20	ZZ Mod 2	25.50
246	-	J	24.63	N Mod	21.8*	ZZ Mod 2	25.50
247-248	D	J	24.63	P Mod	21.45	ZZ Mod 2	25.50
249	-	J	24.63	CC	20.53	BB	25.41
250	E	J	24.63	CC	20.53	ZZ Mod 2	25.50

*Estimated.

heaters can be remachined in various ways, e.g., by selectively thinning to locally increase power dissipation, or by inserting pipe-threaded plugs to shunt portions of the conductive path. The average temperature of the end heating elements can be adjusted relative to the average temperature of the center element by thinning the appropriate one all along its length. The significant changes made to the heaters listed in Table III to progressively improve the profiles (as shown in Figure 2) are as follows:

A to B: Heater "X" was thinned at the first and last segments to reduce the distinct profile dip at these locations. Heater "ZZ" was progressively thinned from its center to its left end, to partially cancel the gradient designed into it by slot location.

B to C: Heater "X mod 1" was thinned all along its length to reduce its average temperature, and plugged to flatten its central hot spot. Heater "ZZ mod 1" was selectively thinned a bit more and plugged to reduce the temperature in one spot.

C to D: No intentional change was made but heaters "L" and "X mod 2" were broken by shattering of the crucible following run 245, and were replaced by others machined to the same dimensions, but differing slightly in resistivity of the graphite. Replacement heater "N mod" suffered the same fate, and was replaced by dimensionally identical "P mod."

Following the measurement of profile D, the drawings from which the original heaters were made were changed by relocation of the slots which define the conductive path to replicate that profile, which had been achieved by machining away portions of the outer surfaces of the original heaters. Elements "CC" and "BB" were made from the new drawings. As profile E on Figure 2 shows, center heater "CC" yielded a profile which is quite uniform. However, redesigned heater BB (profile not shown) had a considerable temperature drop-off toward the right end, which was quite evident in run 249 where cartridges 1 and 2 operated well but cartridge 3 did not. One more iteration should be sufficient to arrive at a design of the end heater having the optimum profile "built in" by varying the pitch of the conductive bars.

The temperature and resistance measurements made in investigating this problem have fairly precisely defined the optimum resistances of the heaters for proper end-region to center-region balance and for matching to the power supply.* Because the resistivity of graphite stock is somewhat variable, we presume that it will be necessary to include, in the fabrication process for heaters, a preliminary resistance measurement and a final "skimming" along each element's length, to adjust its resistance to the desired value.

The ultimate criterion for the suitability of a main furnace temperature profile is the growth performance of the cartridges. As discussed in the next section, the heater profiles D and E of Figure 1 were qualitatively "good" in this respect. In order to more quantitatively define the thermal environments of the three cartridges in terms of both average temperature, and lateral balance, we decided to measure and record the power levels of the die-region heaters in each cartridge in each future run. An electronic wattmeter specially adapted to this task was built and these measurements were made starting with run 248. The results of these measurements will be discussed in future reports.

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*Close examination of the data of Table III and Figure 2 reveals some anomalies, such as different temperatures in the right-hand third of D and E where the same heater was used. We suspect that the insulation package of the furnace does not close as tightly in some runs as in others, allowing radiation leaks.

C. Multiple Growth Runs

This is the first reporting period in which three cartridges and a full-length crucible were in place in all runs. A brief narrative of these runs follows, and data on throughput for those which produced significant quantities of ribbon are listed in Table IV.

Run 244: This run followed the major alteration of the main furnace temperature profile which resulted in full-width growth of ribbon from cartridge position No. 2 for the first time. The stability of this ribbon under automatic control was excellent in this and in subsequent runs. Cartridge 3 displayed a hot center and cold left side condition which prevented full-width growth.

Run 245: Shortly into this run, the die heater in cartridge 2 developed arcing and the furnace was shut down. However, prior to the shutdown, all three cartridges were observed to show a similar hot center condition in seeding attempts. A decision was made to change the profile of the face heater element to correct this.

Run 246: One cartridge was assembled with an experimental profiled face heater; another was set up with the cold shoes closer to the die than normal. The profiled face heater effectively corrected the hot center condition in the die of the cartridge in which it was installed. Observations were made of the melting patterns in the three dies, and wattage measurements were made (prior to construction of the special wattmeter instrument mentioned above).

Run 247: All three cartridges were set up with the profiled face heater elements. Cartridges 1 and 2 were growing at full width and cartridge 3 was being spread to full width also when the melt replenishment unit failed due to deterioration of an electrical insulator.

TABLE IV

RUN DATA FOR MULTIPLE 10 CM WIDE RIBBON GROWTH IN FURNACE 16 (MODEL 3A)

	Run Nos.:	244	247	248	249	250
Overall System:	Duration of growth period (meters)	279	150	317	310	595
	Length of ribbon produced (meters)*	12.2	7.9	18.9	8.2	33.0
	Time percentage of simultaneous three-ribbon growth	0	15	34	0	21
Cartridge No. 1:	Length of ribbon produced (meters)	4.1	3.8	6.0	3.2	9.6
	Time percentage of run period operating	44.8	80.0	56.7	31.2	48.1
	Average growth rate (cm/minute)	3.25	3.17	3.30	3.27	3.36
Cartridge No. 2:	Length of ribbon produced (meters)	7.6	3.0	8.9	5.0	15.6
	Time percentage of run period operating	93.5	62.0	98.7	47.0	39.7
	Average growth rate (cm/minute)	2.92	3.22	2.81	3.40	2.92
Cartridge No. 3:	Length of ribbon produced (meters)	0	1.5	4.0	0	7.8
	Time percentage of run period operating		38**	47.3		47.0
	Average growth rate (cm/minute)		2.6**	2.63		2.78

*All listed quantities are for full-width ribbon, except Cartridge No. 3 in run 247.

**Estimated data.

Run 248: The system was set up as in run 247. Cartridge No. 2 was the first to be set in operation, and it ran continuously for 317 minutes. Cartridge No. 1 grew ribbon during five periods between 20 and 70 minutes long. Difficulty was encountered with ribbon breakage which caused significant delays in restarting the growth. Cartridge No. 3 was not successfully started up until halfway through the run, because of seed-ribbon breakage. The run ended when the available stock of silicon charge rods was depleted.

Run 249: Center and right main heating elements of the new design discussed above were installed for this run to see if the ribbon growth performance correlated with the temperature distribution of the heaters themselves, which had been measured without the furnace insulation in place. This was the case; the temperature drop-off toward the end of the right heater caused a lateral gradient in cartridge 3 which prevented satisfactory growth.

Run 250: With the right main heater "ZZ mod 2" reinstalled, all three cartridges once again performed well. The primary effects detracting from the throughput of the overall system in this run were:

(a) Seed ribbon breakage: Approximately five hours of time were spent cumulatively during the day (which are not included in the figure "duration of total growth period" on Table IV) in inserting seed ribbons and removing those which cracked or failed to align properly with the die; many broken pieces were melted-in or retrieved. These difficulties have been described in previous reports. To minimize the likelihood that ribbon would spontaneously fracture after restarts following freezes, the operator chose to keep the growth rate somewhat lower and the ribbon somewhat thicker than the cartridges' configuration would otherwise allow.

(b) Interaction with melt replenishment unit: The ribbon in cartridge 1 froze twice at times which appeared to coincide with the insertion of a new, cold silicon rod into the replenishment unit. This possible interaction will be investigated in the near future. The existing melt feeder was operating at its limit to furnish silicon for the three relatively thick full-width ribbons simultaneously grown during portions of this run. Silicon rods were added at intervals of about 20 minutes.

(c) Inoperative automatic growth controls on Station 3. The automatic control equipment had been furnished several months earlier and was assumed to be fully completed and tested. However, the opportunity to use it did not present itself until runs 248 and 250. Following run 248, the operator commented that this unit did not seem to work as effectively as the other two. In run 250, control unit No. 3 was found to be completely inoperative due to wiring errors which could not be corrected without shutting the machine down. Hence, while cartridge No. 2 ran continuously for 407 minutes on automatic control, the growth in cartridge No. 3 ended five times due to freezes or abrupt loss of width. Another problem was observed with a portion of the electronics of cartridge 3 which may have been responsible for some incidents of loss of width in this run. A power-line voltage transient of unknown origin, occurring at regular intervals, was occasionally causing false triggering of the semiconductor device (triac) controlling power to the face heater. Whether or not the automatic width control system would have compensated for the momentary increases in face heater power is not known. Electrical phenomena of this type can now be monitored much more conveniently than in the past because of the existence of the electronic wattmeter, mentioned above, which has a recording capability.

D. Conclusion

Although the 1980 Technical Features Demonstration (TFD) goals for this system have not yet been met, the work of this quarter has eliminated the last remaining barrier to high-throughput multiple ribbon operation. There is one outstanding operational difficulty, which, although it does not absolutely prevent fulfillment of the TFD goals for throughput, will consistently degrade the system's performance and jeopardize the success of any given run, until it has been resolved by design modification of the growth cartridges. This is the thermal stress/seed-ribbon breakage problem.

There are also, in the furnace equipment as it currently exists, several reliability trouble spots which can be solved by design refinement when work is begun on the successor to this ribbon-growing system. The most serious of these are:

(1) Furnace water jacket and cooling system: The furnace chamber of this system, now five and one-half years old, is internally corroding, resulting in frequent blockages of cooling water and overheating. The water jacket will eventually rust through. The water cooling provisions of several other components of the system are inadequate as well.

(2) Melt replenishment unit: This device has proven somewhat unreliable when "pushed" to provide melt at a sufficient rate for three 10-cm ribbons. A new version, now in design in an internal program at Mobil Tyco, will be made available for incorporation into the successor to the current furnace.

(3) Cartridge power supplies: The triac power controllers for the cartridge heaters are the same units which were used to power the 5 cm cartridges. These are operated too near to their power ratings to ensure high reliability. The reliability of the cartridge heaters themselves would also

be improved by the utilization of controllers which incorporate electronic current limiting.

Despite our clear recognition of these shortcomings, we have decided not to invest time in repairing or rebuilding the existing furnace, as we anticipate starting construction of a new furnace for 10 cm wide ribbon early in 1981. Indeed, all these reliability problems have been addressed, and many other refinements have been made, in the design of the multiple ribbon furnaces which are operating in an internal program at Mobil Tyco. Major portions of the new JPL machine can be built from existing drawings, and while these portions are in procurement, engineering effort can be directed toward further improvement of the 10-cm cartridge, which is unique to the JPL program.

Our plan for the first quarter of 1981 thus includes the following items:

(1) Make additional multiple growth runs under essentially the same conditions as run 250. Further investigate the operation of the automatic control system and the thermal interactions between the main furnace, the melt feeder, and the cartridges. Develop operator technique and make minor modifications as necessary to attain higher growth rates from the cartridge configuration now in use.

(2) Design a lengthened version of the cartridge to reduce thermally induced stresses. Also include in the cartridge redesign a more effective seal for the ribbon exit slot so that gas flows through the cartridge and around the meniscus can be controlled. Evaluate these changes in ribbon growth experiments.

(3) Review the operation of the recently built, internally funded multiple ribbon furnaces for possible further design improvement; start procurement of major components of a new multiple ribbon furnace.

IV. CELL AND MATERIALS CHARACTERIZATION

A. Cell Characterization by C.T. Ho

1. Overview

Ambient growth experiments have been conducted in the 10 cm wide high-speed Machine 17 after the gas introduction system was redesigned. Solar cell evaluation for the ribbons showed that the previous experimental results demonstrated in Furnace 18 (JPL No. 1) were also reproducible in the high-speed furnace. However, when quartz is added to the silicon melt, the ambient gas effect tends to disappear.

In the multiple System 3A, simultaneous three-cartridge growth with automatic control (run 16-242) has produced solar cells with conversion efficiency ~8%. Meanwhile, the initial cell data for the ribbon grown from experimental Furnace 18 (JPL No. 1), after the conversion to 10 cm cartridges, also showed about 8% conversion efficiency.

2. High-Speed Furnace No. 17

A series of experiments involving oxygen introduction during growth has been conducted in the 10 cm wide, high-speed system. Runs 17-136 and 17-139 were two gas ambient runs with CO_2 gas being introduced into the growth system using a new die-top radiation shield configuration. Runs 17-137 and 17-138 were again grown without and with the CO_2 gas ambient condition, and quartz was added to the crucible (bulk) melt (see Section II). All runs were boron doped to an average resistivity of 5 Ω-cm. The ribbons were cut into two sizes, 2.5 x 5.5 cm^2 and 5.5 x 10 cm^2, and processed into solar cells by the standard PH_3 diffusion process. The photovoltaic output data under a simulated AM1 condition are tabulated in Table V.

TABLE V

SOLAR CELL DATA FOR 10 CM WIDE RIBBON GROWN IN FURNACE 17 WITH AND WITHOUT CO_2

100 mW/cm^2, ELH LIGHT, 28°C, NO AR

Run No.	Growth	Thickness (cm)	Speed (cm/minute)	Process	Average Resistivity (Ω-cm)	Cell Results			
						J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
17-136	CO_2 off CO_2 on	0.030	3.1	PH_3, 55 cm^2, no AR	5.9	15.8 18.5	0.472 0.511	0.681 0.706	5.08 6.66
17-139	CO_2 off CO_2 on	0.019 - 0.033	3.5	PH_3, 13 cm^2, no AR	5	15.5 19.0	0.470 0.513	0.734 0.753	5.35 7.33

TABLE VI

SOLAR CELL DATA FOR 10 CM WIDE RIBBON FROM MULTIPLE GROWTH RUNS IN FURNACE 16 (JPL NO. 3A)

100 mW/cm^2, ELH LIGHT, 28°C, NO AR

Run No.	Growth	Process	Cell Results			
			J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
16-242	3-cartridge multiple run {1, 2, 3}	CVD, 55 cm^2, no AR	16.6 15.9 15.8	0.474 0.488 0.487	0.677 0.704 0.679	5.32 5.48 5.23

TABLE VII

SOLAR CELL DATA FOR 10 CM WIDE RIBBON FROM FURNACE 18 (JPL NO. 1)

100 mW/cm^2, ELH LIGHT, 28°C, NO AR

Run No.	Growth	Process	Cell Results			
			J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
18-221	SOP	PH_3, 13 cm^2, no AR	15.6	0.498	0.749	5.80
18-223	quartz in melt		15.5	0.497	0.737	5.71

The results of the two gas ambient runs, 17-136 and 17-139, without the quartz doping essentially reproduce the previous experimental results of runs 18-191 and 18-199 from Furnace 18.

The improvement of the cell output (Table V) and the enhancement of bulk diffusion length under illumination shown in Figure 3 are consistent with that expected for the quartz-like behavior. However, in runs 17-137 and 17-138, oxygen introduction by adding the quartz chips in the silicon melt has not shown additional beneficial effects on the photovoltaic property. This ribbon is being characterized further.

3. Multiple Growth Furnace 16 (JPL No. 3A)

A three-cartridge multiple growth run, 16-242, was made with automatic width control during the growth. The boron melt doping level was about 10 Ω-cm. Ribbons grown from each cartridge were separated and processed into 5.5 x 10 cm^2 solar cells by the standard CVD diffusion process. As shown in Table VI, the results indicated that the automatic width-controlled growth yields virtually the same baseline result as the manual growth. At the present time, the baseline cell conversion efficiency for the multiple system is 8 to 9%.

4. Experimental Growth Furnace 18 (JPL No. 1)

A cell fabrication run was made to evaluate the ribbons grown from Furnace 18 since it had been changed over to a 10 cm cartridge system. The cell samples were chosen from two growth runs: standard baseline run 18-221, and with quartz added to the crucible melt, run 18-223. Both runs were boron doped to ≈5 Ω-cm. However, the grown ribbons showed severe warpage. In order to minimize the yield loss during cell fabrication, we cut the cell blanks into 2.5 x 10 cm^2 and processed them with the PH_3 diffusion method. The simulated AM1 data are listed in Table VII.

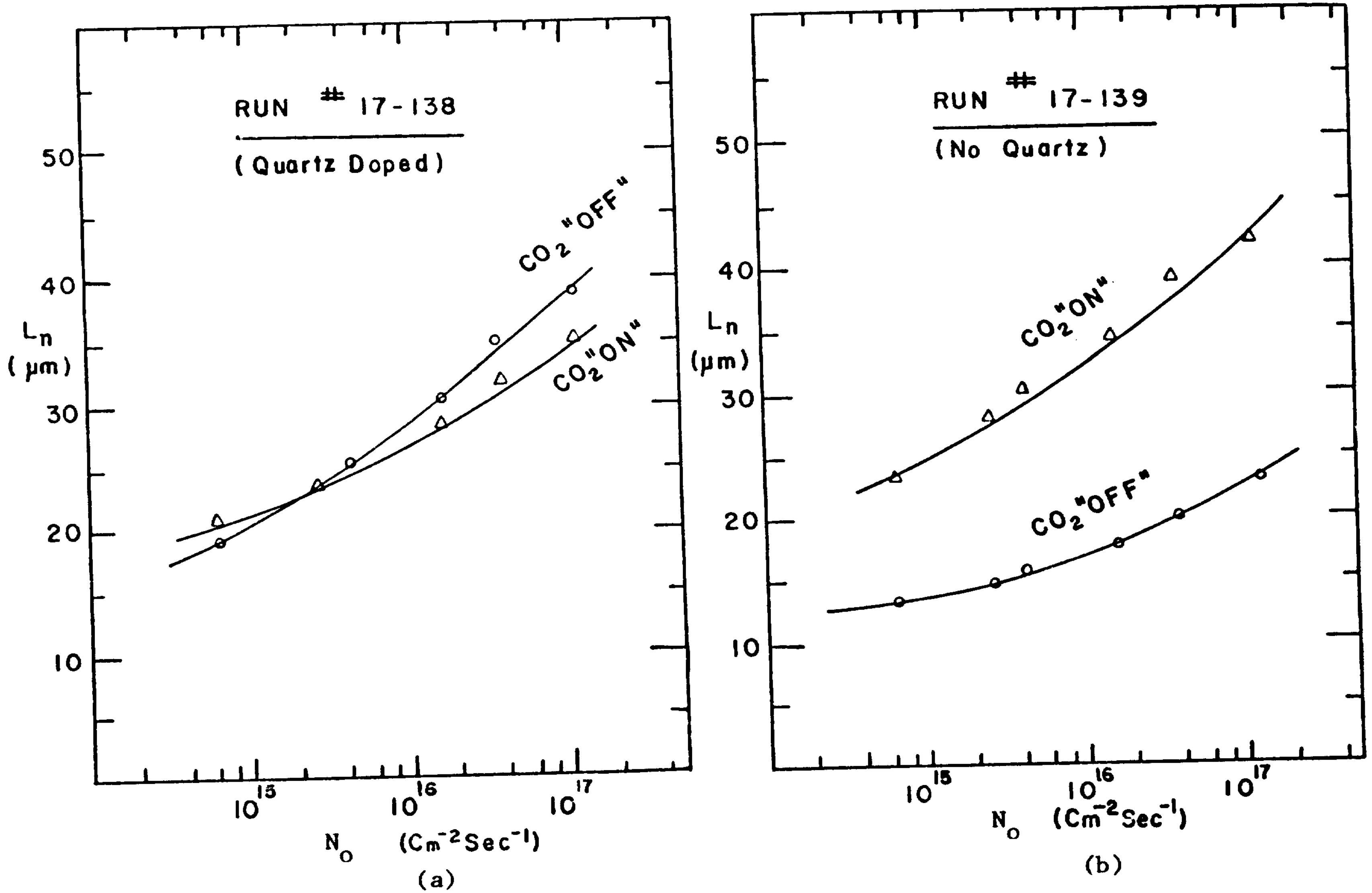

FIGURE 3

Light enhancement characteristics of ribbon grown with and without CO_2 from melt: (a) with quartz, and (b) without quartz added. Plot is of diffusion length (L_n) vs. photon density (N_o).

The small number of cell samples in Table VII reflect the problem encountered in the cell processing. But, from the limited data available here, the ribbon grown with quartz added to the melt did not show any discernible improvement in photovoltaic parameters when compared with the standard baseline run. Since both cell groups have low bulk diffusion length, it is not clear whether a high impurity density in the ribbon negates the effect of oxygen impurity introduced from the quartz during the growth.

B. Materials Characterization by J.F. Long

1. Overview

The bulk of the characterization work performed this quarter was carried out on ribbon grown in Furnace 17. These runs included control runs, furnace atmosphere studies with variable main zone flow rates, introduction of CO and CO_2 into the meniscus environment, and doping level experiments.

2. Furnace 17

The first run undertaken this quarter was a standard, 1 Ω-cm control run. The minority carrier diffusion length average of 30.9 μm reproduced previous baseline quality levels for comparison to ensuing runs.

Runs 17-132 and 17-133 were both attempts to gain a clearer understanding of the meniscus environment as a function of gas flow conditions. While the gas flow rates were varied, the gas composition at the cartridge gas outlet was monitored with a residual gas analyzer (RGA). This monitoring and subsequent data reduction and interpretation were performed by Dr. Jim Gregory of Mobil Tyco. Table VIII is a summary of the relevant data for these two runs. The major finding of this analysis was that a reduction of the main zone purge rate in both runs appears to cause a significant entrainment of room air

TABLE VIII
RESULTS OF RESIDUAL GAS ANALYZER (RGA) STUDY OF FURNACE 17 AMBIENT.

Run and Sample No.	Gas Flow Conditions	RGA Analysis		$\overline{L_D}$ (μm)
17-132-1	main zone Ar: 12 ℓ/minute cartridge Ar: none	N_2 : 7% O_2 : 1%	CO_2 and H_2O constant throughout	33.1
-2	main zone Ar: 9 ℓ/minute cartridge Ar: none	N_2 : 11% O_2 : 1.7%		30.6
-3	main zone Ar: 6 ℓ/minute cartridge Ar: none	N_2 : 20% O_2 : 3.5%		26.6
-4	main zone Ar: 6 ℓ/minute cartridge Ar: 1 ℓ/minute*	N_2 : 22% O_2 : 2.9%		30.6
17-133-1	main zone Ar: 12 ℓ/minute cartridge Ar: none	N_2 : 6.3% O_2 : 1.1% CO_2: 0.085%	H_2O constant	32.6
-2	main zone Ar: 9 ℓ/minute cartridge Ar: none	N_2 : 6.3% O_2 : 1.2% CO_2: 0.085%		31.0
-3	main zone Ar: 6 ℓ/minute cartridge Ar: none	N_2 : 18% O_2 : 2.8% CO_2: 0.1%		23.6
-4	main zone Ar: 6 ℓ/minute cartridge Ar: 1 ℓ/minute*	N_2 : 17% O_2 : 2.7% CO_2: 0.1%		19.2

*Left side only. RGA sampling done with right-side gas jet.

into the furnace, as evidenced by the increase in N_2 and O_2. Note that these two gases are detected in ratios which closely approximate their ratio in air. It is felt that the air was being introduced into the gas jet sampling tube from somewhere near the top of the furnace because of the low sampling rate used in the RGA analysis. Although this does not necessarily imply air entrainment when normal cartridge gas flow rates are utilized through this jet, it probably does indicate significant amounts of air in the furnace during reduced main zone growth. No clear trend was established in these two runs between flow rate and diffusion length.

All other runs undertaken this month were doped to a nominal melt resistivity of 4 Ω-cm (except 17-133, doped to 2 Ω-cm). Three runs in which no other departures from standard procedure occurred are tabulated below:

Sample No.	Nominal ρ (Ω-cm)	Measured ρ (Ω-cm)	Thickness (cm)	$\overline{L_D}$ (μm)
17-131-1B	4	5.4	0.030	46.9
17-135-1B	4	5.5	0.032	24.4
17-141-2D	4	7.3	0.019	36.2

In run 17-135 above, the standard Furnace 16 (3A) cartridge was employed. High SiC particle densities were noted on this ribbon, suggesting possible differences in the ambient or thermal environment of this cartridge. This may account for the lower diffusion lengths observed in this run.

A number of the runs this quarter involved the introduction of CO or CO_2 into the meniscus ambient. The results are contained in Table IX. Results for this entire series are mixed. Runs 134, 136, and 139 suggest improvement with gas introduction. Runs 137 and 138 show no improvement. A discussion of the effects observed in solar cells fabricated from this series of runs is given above.

TABLE IX

SPV DIFFUSION LENGTH RESULTS FOR GAS AMBIENT EXPERIMENTS WITH 10 CM WIDE RIBBON IN FURNACE 17

Run and Sample No.	Melt Doping ρ (Ω-cm)	Measured ρ (Ω-cm)	Gas Flow Conditions		Quartz Added	$\overline{L_D}$ (μm)
			Cartridge Gas	Flow Rate (ℓ/minute)		
17-134-1A	4	6.5	Ar	0.5	no	21.1
-3A		6.4	0.05% CO in Ar	0.5		44.6
-3C		5.7	0.1% CO in Ar	0.5		33.0
17-136-1A	4	6.3	Ar	1.0	no	18.9
-2B		6.0	0.2% CO_2 in Ar	1.3		45.3
-2C		5.0	0.3% CO_2 in Ar	1.5		34.4
-2D		6.2	0.1% CO_2 in Ar	1.1		27.5
17-137-1A	4	5.6	Ar	1.0	yes	36.5
-2A		3.6	0.2% CO_2 in Ar	1.3		35.3
-2B		3.2	0.3% CO_2 in Ar	1.5		30.4
17-138-1A	2	2.9	Ar	1.0	yes	24.2
-1B		2.3	0.1% CO_2 in Ar	1.2		24.5
17-139-1B	4	5.4	Ar	1.0	no	27.8
-2A		4.6	0.2% CO_2 in Ar	1.25		41.4

REFERENCE

1. F.V. Wald et al., "Large Area Silicon Sheet by EFG," Annual Progress Report, DOE/JPL 954355 (October 1, 1979 - September 30, 1980), February 1981.

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 100,661 and cost plus fixed fee was $3,657,319. Man hours for the fourth quarter of 1980 are 5,224 and cost plus fixed fee is $204,747. Therefore, total cumulative man hours and cost plus fixed are 105,885 and $3,862,066, respectively.

3. Engineering Drawings and Sketches Generated During the Reporting Period

None.

4. Summary of Characterization Data Generated During the Reporting Period

See Section IV.

5. Action Items Required by JPL

None.

6. New Technology

Any new items of technology will be separately reported pending possible patent action.

7. Other:

As-grown ribbon sent to JPL, fourth quarter 1980.

APPENDIX 7

As-grown ribbon shipped to JPL, fourth quarter 1980

Date Shipped	Run Number	Width (cm)	Total Length (cm)	Number of Pieces
11/24/80	18-170	5	112	13
	-171	5	130	22
	-173	5	122	14
	-174	5	152	18
11/26/80	16-195	10	18	1
	-205	10	115	3
	-206	10	110	2
	-211	10	52	1
	-223	10	91	4
12/22/80	16-212	10	24	1
	-214	10	139	4
	-217	10	376	11
	17-090	10	114	28
	-093	10	124	33
	-097	10	94	21
	-099	10	5	2
	18-177	5	41	8
	-183	5	20	4
	-186	5	10	2

NOTE: Information pertaining to each of these runs was included with each shipment. This information may also be found in prior reports.