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

LARGE AREA SILICON SHEET BY EFG

Program Manager: Juris P. Kalejs

Second Quarterly Report - Subcontract No. 954355

Covering Period: April 1, 1981 - June 30, 1981

Distribution Date: September 11, 1981

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ABSTRACT

The influence of parameters such as CO_2 concentration, gas flow patterns, quartz in the bulk melt, melt doping level and growth speed on ribbon properties has been examined for 10 cm wide ribbon. One of the more important findings is that ribbon quality is optimized for ambient CO_2 in argon concentrations in the range from 1000 to 5000 ppm. Cell performance degrades at CO_2 concentrations above 5000 ppm and IR interstitial oxygen levels decrease. These experiments have been done primarily at a growth speed of 3.5 cm/minute.

Cartridge parameters influencing the ribbon thickness have been studied and thickness uniformity at 200 micrometers (8 mils) has been improved. Growth stability at the target speed of 4.0 cm/minute has also been improved significantly.

A successful demonstration of interface ambient control in Furnace 16 has been carried out. Ribbon characterization has shown that SPV diffusion lengths and cell performance have improved with CO_2 introduced into the multiple furnace environment.

The construction of a new multiple ribbon furnace for growth of four 10 cm wide ribbons is proceeding on schedule with assembly of a number of subsystems underway. This furnace is being built in-house at MTSEC's expense and is to be introduced into the JPL program in the last quarter of 1981.

A new 10 cm cartridge design has been completed, primarily to incorporate modifications in the post-growth temperature profile, and fabrication of components has been started.

TABLE OF CONTENTS

SECTION		PAGE
	ABSTRACT	iii
I	INTRODUCTION	1
II	HIGH-SPEED GROWTH AND QUALITY STUDIES	7
III	MULTIPLE RIBBON GROWTH AND AUTOMATIC CONTROLS	17
IV	CELL AND MATERIAL CHARACTERIZATION	27
	REFERENCES	45
	APPENDICES	47

LIST OF TABLES

TABLE		
I	Run Data for Growth of 10 cm Wide Ribbon in Furnace 17	9
II	Run Data for Ribbon Growth in Furnace 18	14
III	Summary of 10 cm Wide Ribbon Growth Runs in Furnace 16	19
VI	Summary of Averaged Solar Cell Data for Phosphine Processed Cells from Run 17-136	28
V	Summary of Averaged Solar Cell Data for Phosphine Processed Cells from Ribbon Grown with High CO_2 Levels	31
VI	Summary of Solar Cell Data for Multiple Ribbon Furnace Run 16-259	34
VII	Summary of Averaged Solar Cell Data for Phosphine Processed Cells from Run 17-162	36
VIII	Summary of Averaged Solar Cell Data for Phosphine Processed Cells from Run 17-147	38
IX	Summary of Averaged Solar Cell Data for Phosphine Processed Cells with Quartz in Melt	40
X	Summary of Averaged Solar Cell Data for CVD Processed Cells with Quartz in Melt	42

LIST OF FIGURES

FIGURE		
1	Diffusion length as a function of flow rate of 1% CO_2 in Ar gas mixture to growth cartridge in run 16-259	32

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 (approximately 50 cm^2) for ribbon grown in single-ribbon (cartridge) furnaces at speeds of the order of 2 cm/minute. Cell efficiencies averaged 9% for ribbon grown in the more complex multiple-ribbon furnace at speeds of up to 4 cm/minute.

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. In the past year, significant developments related to the work toward these goals 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 (approximately 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 on 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 (J.P. Kalejs)

A. Overview

Optimization studies have been carried out with the 10 cm cartridge in Furnace 17 to investigate factors influencing growth stability and material quality at growth speeds in the range from 3.5 to 4.0 cm/minute. Growth stability has been improved considerably at the target speed of 4.0 cm/minute. Results from growth with high (greater than 0.5%) CO_2 concentrations show that as-grown ribbon diffusion length and cell parameters degrade with increasing concentration above about 0.5% of CO_2. Interstitial oxygen levels coincidentally decrease to the order of the detection threshold of approximately 1×10^{16} atoms/cc.

Development of a 10 cm cartridge to grow without cold shoes has continued in Furnace 18. Full-width growth has been established at 2 cm/minute, and testing of design iterations is underway to find reproducible growth conditions and improve growth stability.

B. Experimental

1. Growth Stability

Optimization of growth conditions for 10 cm wide ribbon in Furnace 17 is proceeding through examination of the influence of design iterations in a number of cartridge components on growth stability in the range of growth speeds from 3.5 to 4.0 cm/minute. Among these are: details of the die end and bulb geometries, the cold shoe configuration, die shield gas distribution system, and end and face heater designs. In the process, ribbon thickness has also been monitored with the purpose of achieving uniform thickness profiles across the ribbon width at a target thickness of 200 micrometers (8 mils).

A summary of the experiments carried out this quarter in Furnace 17 is given in Table I. As a consequence of varying the face heater profile across the ribbon width, compensation for die top temperature gradients produced by other cartridge components has been possible, and acceptable thickness uniformity achieved while maintaining growth stability. Thickness variations were reduced to within the measurement error of 25 micrometers (approximately 1 mil) over the central 7 cm span of the ribbon for ribbons grown in the thickness range of 200 to 300 micrometers (8 to 12 mils). The ribbon thickness still increases considerably within the two centimeters closest to each ribbon edge. This is due partly to the cold shoe which increases in thickness there,

TABLE I

RUN DATA FOR GROWTH OF 10 cm WIDE RIBBON IN FURNACE 17

Run No.	Speed Range (cm/minute)	Gas Flow Conditions (ℓ/minute)		Comments
		Main Zone	Cartridge	
17-160	3.1 - 3.5	6, Ar	-	Study of thickness uniformity across ribbon width. Gas introduction system to cartridge malfunctioned.
17-161	3.3	6, Ar	-	Study of thickness uniformity continued. Gas system malfunction again.
17-162	3.1 - 3.3	6, Ar	0.5 - 1.5, 1% CO_2 in Ar	Test of new design of die top shield and gas introduction configuration. Additional examination of thickness profiles.
17-163	3.1 - 3.3	6, Ar	1 - 1.5, 1% CO_2 in Ar	Repeat of 17-162.
17-164	2.8	6, Ar	1, Ar	Growth of ribbon for seed material.
17-165	3.4 - 3.8	6, Ar	0 - 1, 1% CO_2 in Ar	Test of die design with reduced die tip cross section. Die top thermal balance poorer than with regular die.
17-166	3.2	6, Ar	2, Ar	Repeat of run 17-147 with quartz in melt and higher growth speed.
17-167	-	-	-	Growth attempted with modified belt puller. Poor growth conditions limited growth.
17-168	*	6, Ar	1, Ar	Poor growth conditions later traced to malfunction in electronics associated with puller speed control.
17-169	*	6, Ar	1, Ar	Growth conditions poor once again due to recurring electronics problems
17-170	*	6, Ar	1, Ar	Diagnostic run to examine cause for speed control malfunctions.
17-171	*	6, Ar	1, Ar	Diagnostic run to study puller control problems.
17-172	3.4	6, Ar	1, Ar	Puller speed control problem fixed in this run. Initial trial of rebuilt cartridge.
17-173	3.6 - 4.1	6, Ar	1, Ar	Good growth conditions at higher speeds.

*The puller speed control electronics were malfunctioning during these runs, and actual growth speeds are not known.

TABLE I (continued)

Run No.	Speed Range (cm/minute)	Gas Flow Conditions (ℓ/minute)		Comments
		Main Zone	Cartridge	
17-174	3.5 - 3.6	9, Ar	1.5, Ar	Growth in higher speed range with quartz in melt to compare to results of earlier run 17-147.
17-175	3.5	6, Ar	1 - 1.5, 1% CO_2 + 100 ppm O_2 in Ar	Higher speed run with controlled meniscus ambient gases for quality studies.
17-176	3.5 - 3.8	6, Ar	0.3, Ar + 1% CO_2 + 100 ppm O_2	Experiment for quality evaluation with high CO_2 levels and high growth speeds.
17-177	2.5 - 3.5	6	0 - 1, Ar + 1% CO_2 + 100 ppm O_2	Test of non-bulbous end die design. Growth not as stable as with bulbous end.
17-178	3.5	6	0 - 1, Ar + 1% CO_2 + 100 ppm O_2	Repeat of 17-176.
17-179	3.5 - 4.1	6	0 - 1, Ar + 1% CO_2 + 100 ppm O_2	Test of shorter end heaters. Good stability at higher speeds of 4.0-4.1 cm/minute.
17-180	2.6 - 3.1	6	0	Preliminary trial of new cold shoe design. Poor growth conditions.
17-181	3.5	6	0 - 1, Ar + 1% CO_2 + 100 ppm O_2	Repeat of 17-176 with melt doping level of 4 Ω-cm.

and partly to the end and face heater balance that is being maintained to create a temperature gradient favorable to ribbon edge position control. This work has demonstrated that the flexibility to tailor the die top isotherms to reduce thickness nonuniformity is available without a sacrifice in growth stability. Further work in this area will be needed after the design of other cartridge components which also influence die top isotherms is fixed.

The improvement in ribbon thickness uniformity has also carried over to producing more reliable growth conditions and stability near the target speed of 4.0 cm/minute. In a number of runs, growth at this speed has been sustained over periods of the order of 30 minutes. At this point, it is not clear what are the chief causes of growth termination, or freezes. Additional flexibility in operator control over the ribbon edge position is still desirable, while another important factor is ribbon nonflatness. The latter produces perturbations of interface position relative to the die top, and clearly affects the length of the period of growth that can be achieved between freezes. Future work in this area will examine ways to improve ribbon flatness by testing different designs of puller belt and rollers, by defining tolerances in cartridge and puller needed to minimize misalignments, and by use of modified cold shoe and linear cooling plate geometries (see also Section IIIC).

Another area of investigation in connection with growth stability studies is the influence of the interface gas distribution system. Gas velocities and flow patterns can have an impact on stability and heat transfer in the critical area of the ribbon edges. A modified die shield was introduced in runs 17-162 to -170, designed to vary the velocity and flow pattern of the interface gases. No conclusions were drawn on its impact on growth conditions on the basis of this series of tests, and its use was discontinued when it was observed that SPV diffusion length changes with CO_2 were not occurring reproducibly.

The introduction of CO_2 and O_2 mixtures has been noted to influence growth stability in certain circumstances. It is conjectured that it is the nonuniformity in the gas concentrations, particularly for oxygen, that is responsible. It has been shown that oxygen by itself at higher concentrations results in unacceptable growth perturbations because of generation of SiO_2 on the meniscus surface, thus producing changes in local heat transfer conditions. Greater care in ensuring gas uniformity has made the use of CO_2 and O_2 possible at higher growth speeds than before. For example, in the series of runs from 17-175 to -181 where high CO_2 concentrations were used, growth at 3.5 cm/minute was not noticeably affected when the gas was introduced. Since the importance of gas flow conditions has now been demonstrated, more study of their influence is planned as part of future quality optimization studies.

A preliminary trial of a new design of cold shoe was made in run 17-180. The initial attempt used a continuous nickel tube, 0.32 cm (0.125 inch) in outer diameter, shaped to extend along each side of the growth interface. Growth with this arrangement was difficult. The tubes by themselves did not provide sufficient heat removal because of their much reduced surface area as compared to the conventional cold shoe. This demonstration illustrates that important functions of the cold shoe are to remove heat from cartridge components and shield the growth interface from an otherwise hotter radiating environment. The next stage in developing the new cold shoe design will be to increase the cold shoe surface area through attachments of shields to the tubes.

The experiments in Furnace 18 have been directed toward establishing good growth conditions in a 10 cm cartridge without cold shoes. They are summarized in Table II. Growth at 2 cm/minute has been reasonably reproducible, but nonuniformity in die top isotherms has limited flexibility in attempts to attain higher growth speeds. A number of cartridge component design changes have been introduced to improve the die top temperature balance: face heater profiling, shield placements, end heater and die design variations. These have not had the desired impact, and the search for additional factors is continuing.

A new two-piece die design has been introduced to attempt to improve on some areas of deficiency of the one-

TABLE II
RUN DATA FOR RIBBON GROWTH IN FURNACE 18

Run No.	Purpose
18-271	Repeat of run 18-271, with good growth conditions at 2 cm/minute.
18-272	Increased length end heaters used. Poor growth conditions due to hot die center.
18-273	Additional modification in end heater design. Growth conditions not noticeably improved over run 18-271.
18-274	Profiled face heater from Furnace 17 used. No improvement in growth conditions.
18-275	Additional face heater profiling. No significant changes in growth conditions.
18-276	Poor growth conditions with additional face heater modifications; limited growth due to SiO buildup in furnace.
18-277	Repeat of 18-276. Poor growth conditions resulting from additional face heater modifications.
18-278	Return to configuration of run 18-275. Growth at higher speeds moderately successful.
18-279	Support pins from die shields and tabs from cartridge floor removed to try to alleviate temperature imbalances. No improvement in growth conditions.
18-280	Test of new profiled face heaters resulted in center of die too cold.
18-281	Additional change in face heater profile made. No growth due to shield misalignment.
18-282	Repeat of 18-281, with new face heater profile and two-piece die. Growth conditions appeared reasonable in spite of die misalignment.
18-283	Good growth stability with two-piece die and profiled face heater. One and a half meters grown with improved ribbon thickness profile.

piece die in general use in 10 cm growth. Manufacture of the new die is simpler and the capillary channel can be made more reproducibly. The latter has been one of the problem areas in manufacture of the one-piece die because the saw cuts forming the capillary channel are internal to the die, and cannot be toleranced to the desired degree. Several trials of this die have shown it to be reliable and some improvement of die top temperature isotherms has also been noticed.

Experiments in Furnace 18 have not proceeded smoothly because of additional factors associated with furnace unreliability. Steps are being taken to correct some of these problems. A new control electronics cabinet with more up-to-date components is being constructed. The main zone heater and power studs have been redesigned to incorporate all graphite parts. The molybdenum posts presently in use have become increasingly unreliable, and appear to be the cause of a temperature gradient in the main zone which has been affecting the die top isotherms. The furnace water cooling system has also developed problems contributing to the general unreliability of the furnace, and plans are being made to rebuild it in the near future. These improvements are scheduled to be carried out in the next quarter.

2. <u>Material Quality</u>

Growth conditions for 10 cm wide ribbon have been extended to include higher concentrations of CO_2 and O_2 mixtures and faster growth speeds. Improvements in the gas distribution system have been a contributing factor to the attainment of more uniform and reproducible results. The latest series of ambient experiments (runs 17-176 to -181) has been carried out at a growth speed of 3.5 cm/minute (see Table I). Concentrations of CO_2 and O_2 up to 1% CO_2 and 100 ppm O_2 have been used without noticeable impact on growth stability at this speed.

Previous experiments indicated that material parameters and cell performance were still improving with increasing CO_2 concentrations in the range from 3000 to 5000 ppm. A series of runs was therefore made using 1% CO_2 + 100 ppm O_2 in argon. The results were disappointing, however, as it was found that the SPV diffusion length and cell parameters did not improve. Rather, a degradation of cell performance was in evidence at the highest concentration (see Section IV). Interstitial oxygen levels also dropped to the IR spectroscopy detection threshold (approximately 1×10^{16} atoms/cc) for the 1% CO_2 experiments. The evidence gathered to date of material quality dependence on CO_2 concentration shows that the best results for cell performance are obtained for CO_2 in the range from 1000 to 5000 ppm (at a main zone flow rate of 6 liters/minute). The interstitial oxygen levels also peak in this region, and on the low concentration side track the cell improvement observed with increasing CO_2 concentration. More details on this effect and other experiments related to quality are given in Section IV.

III. MULTIPLE RIBBON GROWTH AND AUTOMATIC CONTROLS (B.H. Mackintosh)

A. Overview

A reduced number of runs were carried out in Furnace 16 this past quarter. All were single-cartridge growth runs. Improvements in the automatic width control system have been tested in these experiments, and the video electronics has been debugged. Interface gas control has been successfully demonstrated in Furnace 16 with the die shield gas distribution system in use in Furnace 17, and significant improvements in SPV diffusion length and cell performance have been demonstrated.

Design work on an extended version of the 10 cm cartridge has been completed and fabrication of the cartridge parts has been started. This design extends the length of the afterheater and reduces the magnitude of the cartridge exit temperature drop. This large temperature drop has resulted in frequent seed breakage and a reduction in productivity in the 10 cm cartridge system.

Other design improvements have been incorporated into the new cartridge as part of the schedule of 10 cm cartridge design specification and updating for year-end trials in a new multiple furnace.

Parts for a new furnace for multiple growth of four 10 cm wide ribbons have begun to arrive. This furnace, designated No. 21, is to be built at Mobil Tyco's expense and introduced into the program in November, 1981. Assembly work on the main zone, cartridge and puller power supplies is now in progress.

B. Single-Cartridge Experiments

No multiple ribbon growth runs were made this quarter, as more effort is being directed toward construction of a new four 10 cm ribbon furnace. Seven single-cartridge runs were made, with emphasis on testing improvements in automatic width control system and in ambient control. The results are summarized in Table III.

Improvements in the automatic width control video components were tested, and control system signals monitored during growth in several runs. No spurious noise signals were detected, and it appears that they have been eliminated as a significant source of growth interruptions. The capabilities of automatic width control in helping to meet the long-term productivity goals could not be evaluated in these runs because of factors not related to the control system itself. High levels of SiC and ribbon binding due to non-

TABLE III

SUMMARY OF 10 cm WIDE RIBBON GROWTH RUNS IN FURNACE 16. ALL ARE SINGLE-CARTRIDGE RUNS.

Run No.	Purpose
16-257	Test of reworked automatic width control system. No obvious problems, but growth without freezes of more than one hour infrequent. Ribbon non-flatness and high SiC levels appear responsible.
16-258	Repeat of 16-257. Similar difficulties with ribbon contacting guide plates appear to limit growth duration without freezes.
16-259	First test of die shield gas distribution system in use in Furnace 17.
16-260	Operator training run.
16-261	Test of video remote window position control. Test of constricted growth slot configuration.
16-262	Repeat of ambient control experiment 16-259. Poor growth conditions due to high SiC level, malfunction in gas distribution system.
16-263	Repeat of 16-259. High SiC levels once again; poor growth conditions.

flatness were obvious causes of many freezes in these tests. The SiC appeared to be a result of a longer than usual period of furnace inactivity during which the insulation material of the furnace became thoroughly saturated with atmospheric water vapor. In run 16-261, linear cooling plates with a growth slot constriction close to the interface were tried out. Although this reduced ribbon contact with the upper part of the growth slot, seeding of ribbon was difficult initially because flat seeds were not available.

A successful demonstration of interface ambient control was carried out in run 16-259 using the die shield gas distribution system developed in Furnace 17. Growth conditions in the speed range from 3.3 to 3.5 cm/minute were good under a number of varying main zone and die shield gas flow conditions with CO_2 used as the test gas. Significant improvements in SPV diffusion length and cell performance also occurred. These are discussed in more detail in Section IV. Runs 16-262 and -263 were made repeating the ambient control conditions of this experiment. Data for these runs are not yet available.

C. Redesigned Cartridge to Remedy Seed-Ribbon Breakage Problem

During this reporting period, the design of a new version of the 10 cm cartridge was completed and construction of a prototype unit started. This effort is directed at the resolution of the seed breakage problem which has severely hindered operation of the multiple ribbon furnace since the conversion to 10 cm wide ribbon in mid-1979.

1. Review of Thermal Stress Problems of 10 cm Cartridge, and Design Modifications Made to Remedy Them

Shortly after the 10 cm cartridge was introduced in mid-1979, its tendency to stress and frequently break seed ribbons was identified as resulting from the abrupt transition in temperature between the upper end of the linear cooling plates and the header block. This general feature of the axial temperature profile was a design carryover from the earlier 5 and 7.5 cm cartridges. However, when the 10 cm cartridge was designed, the ending temperature of the linear cooling plates was made even higher (550°C instead of 350°C), in order than a lower gradient could be maintained in the region of the linear cooling plates, which were of roughly the same length as in the earlier cartridges.

The results of this temperature profile, which exposes the ribbon to wall temperatures varying from 70°C to 550°C at points separated by approximately 2 cm, are now well known. When a seed ribbon is introduced into the cartridge, its free end (which usually is scribed and hence contains microscopic mechanical damage) is subjected first to a compressive stress across the axis, and then when it has entered a few centimeters into the linear cooling plates, the region of compressive stress is away from the end, and the end becomes stressed with a tensile force across the axis. It is quite characteristic of the 10 cm cartridge to cause fractures in full-width seed ribbons when they have been inserted between 5 and 10 cm, as measured from the upper surface of the header block.

Once a seed ribbon has been found which does not break, and growth is successfully started, the linear cooling plates seem to work as intended in the 1100-550 degree temperature range, yielding ribbon with little residual stress.

The effects of the severe temperature gradient (and gradient changes) experienced by the ribbon in the region between 550 degrees and room temperature have been less troublesome when the ribbon is being grown out than during seeding, but have hindered stable growth nevertheless. Only rarely has ribbon spontaneously fractured during growth, and these occasions have been associated with growth of ribbon which is considerably thinner in the center than near the ends. This occurs most often during seeding transients. A more common manifestation of the high level of plane stress induced in the ribbon near the top of the linear cooling plates is the elastic buckling out of the plane of the ribbon, which evidence indicates is occurring in this region. This buckling occurs in two ways, namely abrupt snapping out-of-plane of ribbon which is nearly flat entering this region, and non-abrupt deformation in which buckles of small amplitude which have already formed in the hotter regions of the cartridge are temporarily accentuated while passing through this temperature transition. Evidence of the abrupt buckling behavior is seen as sudden shifts of the ribbon over the die top. The resultant "kinks" in the grown ribbon show that sometimes the entire ribbon translates front-to-back, and sometimes a twist occurs. The ribbon can sometimes be heard "snapping" (not breaking) as these shifts are observed.

There are two related manifestations of the non-abrupt elastic buckling. The first is a pattern of heavy scraping of ribbon (SiC particles are responsible for most of the visible signs of scraping) against the linear cooling plates near the upper end, despite the fact that the peak-to-peak thickness of the ribbon after being grown out is always less than the spacing between the walls at this point. Heavy scraping contact between the ribbon and these walls is also implied by the fluctuations which have been observed when the pull rate has been measured with a rubber wheel, driving an optical encoder, rolling on the ribbon. The puller operates with a good, steady "plateau" speed, but the ribbon occasionally slips and its instantaneous growth rate drops. The meniscus is seen to "bounce up and down;" and, of course, if the speed decreases too greatly, a freeze occurs. This binding of the ribbon within the linear cooling plates has been a primary cause of freezes of 10 cm ribbons being grown under automatic width control in Furnace 16 and has hindered efforts to further refine the control system.

The analysis of the temperature distribution in the cartridge and in the ribbon which is the cause of these problems has consisted of several sets of carefully executed conventional thermocouple profiles of the linear cooling plates, and one set of measurements of the temperature profile in a length of ribbon by means of a thermocouple bonded to it with ceramic cement. The temperature profile for the region below 550 degrees obtained by this latter method, when analyzed graphically, showed a peak value of the first derivative (dT/dZ) of $-210^{o}C/cm$, and a peak negative value of the second derivative (d^2T/dZ^2) of greater than $150^{o}C/cm^2$. The main criterion for the design of the new cartridge was to reduce the maximum first derivative by a factor of at least three. It is assumed that in a profile having the same general shape, but with a lower peak gradient, and having the regions of transition of gradient spread out farther, the peak values of the second derivative, which cause the plane stresses, will be reduced by a similar factor of three or greater.

The other criteria for the redesign of the cartridge were to change the temperature profile in the range between 1100 and 600 degrees as little as possible, to limit the increase in length of the cartridge to a dimension which would still be relatively easy to handle, and to not further complicate the unit.

In the redesigned cartridge, a length of 6.86 cm (2.70 inches) has been added to the lower section, and the effective length of the linear cooling plates has been increased by 9.14 cm (3.60 inches). The linear cooling plates are screwed directly to the aluminum header block, eliminating the Inconel "LCP Mounting Fin." The calculated peak level of gradient in the plates is $-75^{\circ}C/cm$, as compared with the value currently measured in the ribbon of $-210^{\circ}C/cm$. The ribbon will exit from the new linear cooling plates at less than $100^{\circ}C$.

The new "stretched" cartridge is designed to be nearly identical to the existing cartridge in the region surrounding the die top in order to exhibit similar meniscus shape, growth rate, and controllability behavior. The same heating elements, floors, and die shields are used. The dies are made with a slightly smaller width dimension, reduced by 0.40 cm (0.160 inch). The cold shoes are identical to the profiled version in use except that the width between thick end sections has been reduced to place the thick ends in the same locations relative to the die ends. Also, the afterheater is better shielded from the cold shoes.

The new cartridge is electrically interchangeable with the standard version, and mounts to the puller base plate in identical fashion. The lower section of this cartridge is 6.86 cm (2.70 inches) longer than the standard version, requiring a "spacer box" where it sits on the furnace top plate, or an elevated top plate; in addition, the height to which the lift mechanism raises the cartridge must be increased.

Completion of the cartridge is expected in early August. It was hoped earlier that it would be ready for trial in early July, before Furnace 16 had to be taken out of service. It now appears that the completion of remodeling work in the Furnace 16 lab in preparation for the new Furnace 21, and the time necessary to clean up the furnace and ready it for operation, will cause a delay of two to three additional weeks. A plan to perform the initial growth tests using Furnace 17 is therefore being considered.

D. Additional Design Modifications

A number of additional changes not directly related to the altered cooling profile are being incorporated into the new cartridge as part of a general updating of its design. These are:

(i) Increased diameter of die top heater feedbars designed to reduce the resistive load.

(ii) Enclosed afterheater element and modified insulation designed to reduce heat loss to cartridge walls and cold shoes.

(iii) Growth slot construction additions to the linear cooling plates to restrict ribbon motion and improve alignment.

IV. CELL AND MATERIAL CHARACTERIZATION

A. Cell Characterization (L.A. Ladd, J.F. Long, and J.P. Kalejs)

Processing of ribbon grown under different ambient gas species and concentrations, with quartz in the melt, with varying melt doping levels and with varying growth speeds has been carried out in a search for parameters that may aid in optimization of cell efficiencies for ribbon grown in the high-speed system. Material characterization and some cell results were given in an earlier report.[1] Here, a more complete presentation of a processing matrix involving these variables is discussed under a number of headings.

1. CO_2 Concentration Effects

Preliminary data on the effects of introducing various concentrations of a gas mixture consisting of 1% CO_2 + 100 ppm O_2 in argon into the cartridge were obtained last quarter. This data suggested that higher levels of CO_2 + O_2 in the cartridge gas leads to higher levels of interstitial oxygen in the ribbon and better cell performance. This effect can be seen in the data for run 17-136 shown in Table IV. Although the diffusion length peaks for a gas mixture

TABLE IV

SUMMARY OF AVERAGED SOLAR CELL DATA FOR PHOSPHINE PROCESSED CELLS FROM RIBBON GROWTH RUN NO. 17-136, WITH CO_2 PLUS O_2 IN AMBIENT; GROWTH SPEED 3.1 cm/minute, $\overline{\rho}$ = 6 Ω-cm. ELH LIGHT, 100 mW/cm², 28°C, NO AR COATING.

Growth Segment	Cartridge Ambient	L_D (μm)	$[O]_i$ (10^{16} cm^{-3})	No. of Pieces	Cell Parameters			
					J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
1A	Ar	19	-	6	15.8	0.472	0.681	5.1
2B	0.14% CO_2 + 14 ppm O_2	45	16	11	18.6	0.519	0.717	6.9
2C	0.23% CO_2 + $\leq$3 ppm O_2	34	28	12	19.2	0.524	0.746	7.5
2D	0.07% CO_2 + 7 ppm O_2	27	4	11	13.7	0.493	0.728	4.9

containing 0.14% CO_2 + 14 ppm O_2, the cell performance keeps increasing up to the highest value of CO_2 + O_2 used in this run. Note also that the interstitial oxygen level increases with CO_2 + O_2 concentrations up to the highest values used in this run (0.23% CO_2 + 23 ppm O_2).

Based on this evidence, a number of experiments involving the use of higher concentrations of CO_2 + O_2 in the cartridge gas were undertaken in order to determine the effect on the interstitial oxygen concentration in the ribbon and on cell performance. The effect of the increased concentration of CO_2 + O_2 on interstitial oxygen was very marked. For levels of CO_2 + O_2 of 0.5% CO_2 + 50 ppm O_2 and above, only trace amounts of interstitial oxygen were detected by low temperature infrared spectroscopy. The detection limit depends on the ribbon thickness and for 250 to 300 micrometer thick ribbon it is approximately 1 x 10^{16} at/cm^{-3}. Clearly a [illegible] of processes are taking place at the meniscus and the growth interface which in combination determine the transport of oxygen into and out of the ribbon. A gas mixture which has 1% CO_2 plus 250 ppm O_2 will be used next to examine what effect changing the ratio of carbon to oxygen in the cartridge gas will have on the oxygen transport properties. Assuming that it is silicon carbide which forms on the meniscus surface and retards the transport of oxygen into the melt at higher CO_2 levels, the use of a different mixture may retard SiC film growth and allow higher levels of oxygen to be introduced into the ribbon. The available data also suggest that a 1% CO_2 gas mixture does not produce as much oxygen in the ribbon as the 1% CO_2 + 100 ppm O_2 gas mixture.

The effects on the performance of phosphine processed cells of using up to 1% CO_2 + 100 ppm O_2 in the cartridge of Machine 17 are shown in Table V. The best cell performance was obtained for the runs with less than this amount of CO_2 + O_2. These also were the runs which had the highest values of interstitial oxygen. It is also to be noted, however, that no exact correlation has been obtained so far between CO_2 + O_2 levels, interstitial oxygen levels, and cell performance. For a given CO_2 + O_2 level, a range of interstitial oxygen levels is obtained, and for a given interstitial oxygen level, a range of cell parameters is obtained. This suggests that all aspects of growth conditions and cell processing parameters are not yet well controlled.

Figure 1 shows the effects on the diffusion length of varying the flow rate of a 1% CO_2 gas mixture in the multiple ribbon Furnace 16. This run (16-259) was a single-cartridge run with the two other cartridge openings containing non-operating cartridges. As can be seen from the figure, increasing the flow rate of 1% CO_2 to the cartridge did have a pronounced effect on the diffusion length of the ribbon. The diffusion lengths obtained at intermediate flow rates are among the best that have been achieved to date with Furnace

TABLE V

SUMMARY OF AVERAGED SOLAR CELL DATA FOR PHOSPHINE PROCESSED CELLS FROM RIBBON GROWN WITH HIGH CO_2 LEVELS. GROWTH SPEED 3.5 cm/minute, $\bar{\rho}$ = 1 Ω-cm. ELH LIGHT, 100 mW/cm², 28°C, NO AR COATING.

Run No.	Ambient	[O] (10^{16} cm^{-3})	L_D (μm)	No. of Pieces	Cell Parameters J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
17-175	0.3% CO_2 + 30 ppm O_2	7	36	7	16.8	0.522	0.728	6.4
-176	1.0% CO_2 + 100 ppm O_2	-	38	10	15.7	0.508	0.686	5.5
-177	1.0% CO_2 + 100 ppm O_2	-	35	10	15.9	0.519	0.615	5.1
-178	0.5% CO_2 + 50 ppm O_2	trace?	35	6	17.6	0.525	0.730	6.8
-178	1.0% CO_2 + 100 ppm O_2	trace?	34	4	17.1	0.519	0.623	5.5

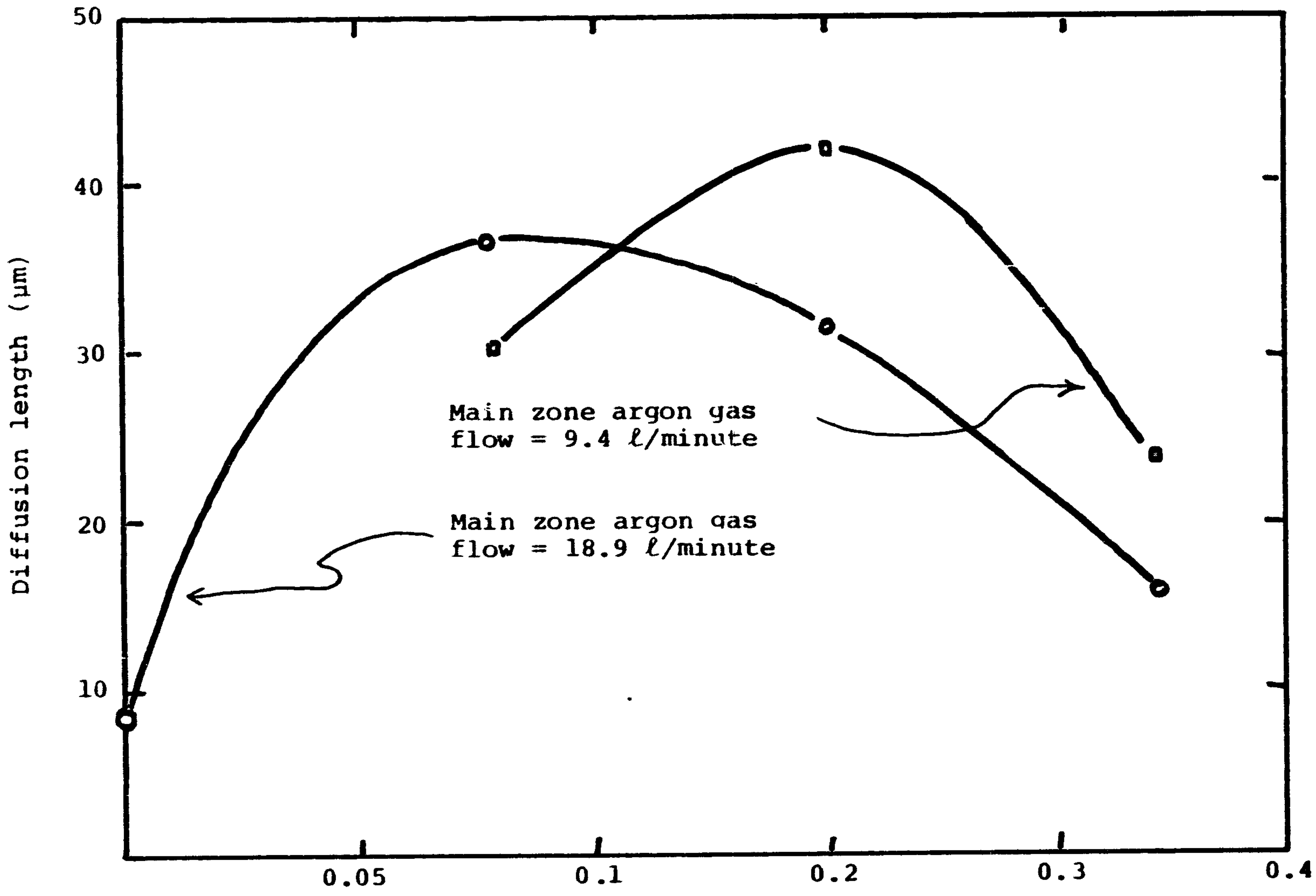

Figure 1. Diffusion length as a function of flow rate of 1% CO_2 in argon gas mixture to growth cartridge in run 16-259.

16. In analyzing this result, we can assume that the actual concentration of CO_2 near the meniscus will be proportional to the flow rate of CO_2 in the cartridge. The cartridge CO_2 gas mixture will be diluted by the main zone gas flow which also passes through the cartridge but the dilution will decrease with increasing CO_2 mixture flow rate. The flow rates of the cartridge gas were quite low in comparison with the main zone flow rates; thus, there may be a substantial dilution factor at the lower flow rates, while for the higher cartridge gas rates the full 1% mixture may penetrate to the interface. Although the data cannot be directly compared as far as concentration levels are concerned, the results essentially reproduce those from Furnace 17 which show lower SPV diffusion lengths as the CO_2 concentration approaches 1%.

Table VI shows the data for cells processed from run 16-259. The data again parallels that obtained from Machine 17 in that as the CO_2 concentration (flow rate) is increased, the cell performance improves and then declines. The difference is that the peak performance is obtained for a ribbon segment in which the interstitial oxygen level is below the detection limit of approximately 1×10^{16} cm^{-3}. We can thus say, synthesizing the data from Machines 16 and 17, that the best cell performance for phosphine processed cells seems to be achieved for an interstitial oxygen level of between just below the detection threshold (approximately 10^{16} at/cc) and 3×10^{17} cm^{-3}. Further work will have to be done to pin this

TABLE VI

SUMMARY OF SOLAR CELL DATA FOR MULTIPLE RIBBON FURNACE RUN NO. 16-259.

GROWTH SPEED 3.5 cm/minute, ρ = 1.0 Ω-cm, 6.25 cm^2 CELL AREA.

MAIN ZONE ARGON PURGE RATE 9.8 ℓ/minute.

PH_3 PROCESSED; ELH LIGHT, 100 mW/cm^2, 28°C, NO AR COATING.

SINGLE-CARTRIDGE OPERATION

Ambient Gas in Cartridge	L_D (μm)	O_i (10^{16} cm^{-3})	No. of Pieces	Cell Parameters J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
0 ℓ/minute, Ar†	8	*	7	11.0	0.469	0.753	3.9
0.07 ℓ/minute, 1% CO_2 in Ar	30	*	3	15.9	0.511	0.751	6.1
0.15 ℓ/minute, 1% CO_2 in Ar	42	N.D.	7	16.9	0.516	0.699	6.1
0.23 ℓ/minute, 1% CO_2 in Ar	24	6.8	7	16.1	0.513	0.658	5.4

†Main zone = 18.9 ℓ/minute, Ar.

*Not measured.

N.D. = Not detected.

down more exactly.

The study of the effects of CO_2 ambient gas concentration on ribbon quality was continued in run 17-162 using a new design of gas distribution system. This was changed to increase gas velocities and redirect the gas more directly toward the meniscus region. The cell data obtained in this case are shown in Table VII. No trends in cell parameters with ambient CO_2 concentration are evident in this case. The lower value of efficiency for segment 1D is attributed to an "end-of-run" effect such as is described in more detail in the next section. Infrared spectroscopy measurements showed no interstitial oxygen in the ribbon. It appears that the new gas distribution system did not have a positive impact for reasons not known at present. The results signify that the ribbon properties are sensitively dependent on the gas flow rates and flow patterns.

2. Crucible Melt Quartz Effect

Quartz has been used to introduce oxygen in the bulk melt contained in a graphite crucible to examine this as an alternate method of getting oxygen into the ribbon. Some inconsistency in cell results has been noticed in the process of doing this, and it has been suggested that either the quartz quality and/or dissolution process (rate and surface-to-volume ratio for quartz surface in contact with melt) have not been constant. A systematic analysis of one run, 17-147, was made to examine possible sources of nonuniformities in

TABLE VII

SUMMARY OF AVERAGED SOLAR CELL DATA FOR PHOSPHINE PROCESSED CELLS FROM RIBBON GROWTH RUN NO. 17-162, WITH CO_2 IN AMBIENT; GROWTH SPEED 3.5 cm/minute, $\overline{\rho}$ = 1.2 Ω-cm. ELH LIGHT, 100 mW/cm², 28°C, NO AR COATING.

Growth Segment	Cartridge Ambient	L_D (μm)	No. of Pieces	Cell Parameters			
				J_{sc} (mA/cm²)	V_{oc} (V)	FF	η (%)
1A	Ar	50	7	15.6	0.503	0.762	6.0
1B	0.09% CO_2	39	8	15.4	0.498	0.752	5.8
1C	0.23% CO_2	33	8	15.7	0.509	0.747	5.9
1D	0.37% CO_2	30	5	14.3	0.497	0.711	5.0

ribbon properties associated with run length, and the results are given in Table VIII. Ribbon from the beginning (segment 1A), middle (segments 1B and 1C) and end (last 0.6 meters, segment D) of the growth, totalling about 4 meters, was processed while keeping the identification of the samples with respect to location within the run. The last segment, 1D, obviously has produced the worst performing ribbon. Two causes are possible for this, both associated with the increase in surface-to-volume ratio of the silicon melt. In the conventional growth mode in Furnace 17, an initial silicon charge of about 250 to 280 grams is emptied entirely. In the last stages of growth, therefore, there will be an increase in the concentration of impurities dissolved in the bulk melt from the quartz if the melt volume is shrinking while the surface of quartz covered by the melt stays approximately constant and the quartz dissolution rate is constant. This would result mainly in a lower diffusion length and short circuit current. Also, the melt surface usually contains significant amounts of SiC, which also is entrained in the capillaries and ribbon with a greater frequency as the melt volume decreases at the end of the run. This would result mainly in the lower fill factors and open circuit voltages such as was observed here and in run 17-162, discussed in the last section.

Ribbon from the same run, segments 17-147-1B and -1C, was also processed as two different lots at two different

TABLE VIII

SUMMARY OF AVERAGED SOLAR CELL DATA FOR PHOSPHINE PROCESSED CELLS FROM RIBBON GROWTH RUN NO. 17-147, WITH QUARTZ IN THE MELT. GROWTH SPEED 2.5 cm/minute, $\bar{\rho}$ = 1.5 Ω-cm. ELH LIGHT, 100 mW/cm^2, 28^{o}C, NO AR COATING.

Process Batch	Growth Segment	L_D (μm)	No. of Pieces	Cell Parameters: J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
I	1A	38	8	18.7	0.542	0.721	7.3
	1B	-	8	17.1	0.536	0.737	6.8
	1C	-	8	17.2	0.535	0.748	6.9
II	1D	44	8	15.2	0.504	0.734	5.6
III	1B & 1C	-	12	18.9	0.531	0.745	7.5

times. The spread in efficiency values from 6.8 to 7.5 shown in Table VIII (or apprcximately 10% spread) reflects the uncertainty due to processing condition variations, measurement errors, or ribbon inhomogeneities. These variations have made it difficult to predict trends on the basis of only a few samples and has necessitated repetition of experiments. At the same time, the difference between results for segments 1B and 1C and segment 1D done in separate batches, as seen from Table VIII, appears to be outside of the range of uncertainty attributable to processing and measurement nonuniformity.

A further result of interest is the high as-grown material diffusion length for segment 1D, which does not reflect the poor cell performance for this segment. The reasons for this are not related to the oxygen level in this case because the material has about the same level of interstitial oxygen throughout.

Table IX summarizes the test data of three runs which were grown with quartz in the melt and processed with phosphine diffusions. All were doped to 1 ohm-cm. Run 17-166 had a piece of crucible quartz and run 17-174 had a piece of Suprasil quartz pinned underneath the melt. The averaged data for run 17-147 which also had a piece of crucible quartz pinned under the melt is also included in this table. As can be seen, there is a large variation in the interstitial oxygen level and in the cell performance. At this point, the

TABLE IX

SUMMARY OF AVERAGED SOLAR CELL DATA FOR PHOSPHINE PROCESSED CELLS FROM RUNS WITH QUARTZ IN THE MELT. ELH LIGHT, 100 mW/cm^2, 28°C, NO AR COATING; ρ = 1 Ω-cm.

Run No.	Growth Segment	Cartridge Gas Flow (ℓ/minute Ar)	Growth Speed (cm/minute)	O_i (10^{16} cm^{-3})	L_D (μm)	No. of Pieces	Cell Parameters J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
17-147[1]	1B & 1C	1	2.5	15	41	28	18.1	0.534	0.744	7.1
17-166[1]	1B	1.5	3.5	N.D.	22	9	12.5	0.498	0.744	4.6
17-174[2]	1A	2	3.5	60	39	10	14.5	0.495	0.690	4.9

[1] Quartz from standard GE crucibles.

[2] Suprasil-W rod.

reason for the variation in properties is not known. As mentioned above, we speculate that variations in the dissolution rate of the quartz and the impurity level in the quartz may account for the differences. There were also differences in the growth speed and main zone and cartridge argon purge rates for the three runs. Any of these could be a factor in determining both interstitial oxygen level and cell performance. The samples with the highest and lowest values of interstitial oxygen have the poorest performance and the samples with an intermediate interstitial oxygen level have the best performance. This is consistent with the results obtained in the last section that cell performance tracks with interstitial oxygen levels up to an intermediate level of interstitial oxygen and further suggests that for high levels of interstitial oxygen the cell performance falls off again. This assumes, of course, that the poor performance was not due to contamination of the melt from the quartz or to some other cause, such as speed or ambient conditions, unrelated to interstitial oxygen level.

Ribbon grown with quartz in the melt was also processed with a CVD diffusion. The results for two runs are given in Table X. In both cases, the CVD and PH_3 processes give cell parameters that are within the reproducibility expected for the material. These findings for ribbon grown with cold shoes are similar to those reported for ribbon grown without cold shoes at speeds of the order of 2 cm/minute.

TABLE X

SUMMARY OF AVERAGED SOLAR CELL DATA FOR CVD PROCESSED CELLS FROM TWO RUNS WITH QUARTZ IN THE MELT. ELH LIGHT, 100 mW/cm^2, 28°C, NO AR COATING.

Run No.	Growth Segment	Ambient	Growth Speed (cm/minute)	L_D (μm)	No. of Pieces	Cell Parameters			
						J_{sc} (mA/cm^2)	V_{oc} (V)	FF	η (%)
17-138	1B	0.07% CO_2	3.2	24	10	18.4	0.514	0.735	7.0
17-147	1A	Ar	2.5	38	6	18.1	0.537	0.695	6.8
	1B			-	5	17.5	0.542	0.717	6.8
	1C			-	4	16.9	0.541	0.657	6.0

3. Resistivity

Several melt doping levels have been used in the experiments described above. These have been mainly 1 and 4 ohm-cm. No identifiable trends of cell parameters with resistivity are indicated on the basis of the available data, and more work in this area is needed.

4. Growth Speed

It was noted in connection with runs 17-143 to -147 that growth speed decreases from the 3 to 3.5 cm/minute range in earlier runs to 2.5 cm/minute in these runs did not lead to better material quality, while generally producing a more inconsistent response of ribbon properties to ambient CO_2 concentration changes. Growth conditions are generally poorer at the lower speeds because the system is optimized to grow at 3.5 cm/minute and above. One result is thicker ribbon, which itself may be responsible for poorer quality because growth occurs with a lower meniscus and the interface closer to the die top. With the decrease in meniscus height, the inconsistency of response of properties to ambient changes may also be explained because the surface area for mass transfer between the gas and meniscus melt is smaller. Quality effects in the speed range from 3.5 to 4.0 cm/minute are of primary concern at this point, and future work will concentrate more in optimization studies in this region.

REFERENCES

1. J.P. Kalejs, et al., "Large Area Silicon Sheet by EFG," First Quarterly Report, DOE/JPL 954355/81-17, January 1, 1981 - March 31, 1981.

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APPENDICES

1. Updated Program Plan

An updated program plan went into effect on March 1, 1981.

2. Man Hours and Costs

Previous cumulative man hours were 111,691 and cost plus fixed fee was $4,079,257. Man hours for the second quarter of 1981 are 4,570 and cost plus fixed fee is $203,510. Therefore, total cumulative man hours and cost plus fixed fee are 116,261 and $4,282,767, 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.