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LARGE AREA SILICON SHEET BY EFG

Second Quarterly Progress Report, January 1, 1976—March 31, 1976

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March 15, 1976

Work performed under contract No. NAS7-100

Mobil Tyco Solar Energy Corporation
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ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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by

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ABSTRACT

The thin/fast growth system (JPL No. 2) is modified and operational. An orientation study was run on this system prior to the changeover and thermal geometry experiments have been run since. (110) [211] seed orientation appears to be the optimum. Solar cells have been fabricated from the ribbon material and tested. The wide ribbon system (JPL No. 1) is being designed. Some parts are ordered. The theoretical analysis of heat flow in EFG silicon growth is expanded.

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INTRODUCTION

The objectives of this program are to produce at high speed, thin, wide sheets of silicon of the quality needed to fabricate 10% efficient solar cells. This is part of a total Mobil Tyco Solar Energy Corporation objective to produce silicon ribbon for solar cells at a cost which will allow their wide scale use for generation of electric power. The edge-defined, film-fed growth process was developed for the commercial production of continuous shaped single crystals of sapphire from the melt and was applied to growth of silicon ribbon for solar cells partly under NSF Grant G137067X via Harvard University, JPL Contract 953365, and under NSF Grant G143873. The basic feasibility of the application of EFG to the growth of silicon ribbon has been proven and the theoretical base for extending the technique to the efficient production of single crystal silicon sheet has been established.

The two major tasks in this program to produce large area silicon sheet by EFG are: the growth of thin ribbon at high speeds in existing apparatus; and the design and construction of a system for the growth of wide ribbons. These two tasks will be combined toward the end of the program to achieve the ultimate program goal. Concurrent with the development of the growth process and ribbon growth apparatus will be material and solar cell characterization programs and theoretical studies of various aspects of EFG silicon sheet growth including melt replenishment, the effect of h_{eff} and aspects of heat flow in the EFG silicon growth system.

During this second Quarterly reporting period, work has progressed along several fronts. Orientation and preliminary growth speed experiments have been carried out and are reported here. The results of initial theoretical analyses and material characterization also follow.

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TECHNICAL DISCUSSION

Apparatus

One major task of this program is to grow thin ($0.004'' \pm 0.001''$) silicon ribbon at high growth rates (7.5 cm/min). This task is to be performed using approximately half-time of a Mobil Tyco owned resistance heated system modified to current state-of-EFG-art specifications. Ribbon growth runs 14-134 through 14-156 (Tables I and II First Quarterly Progress Report and Tables II and III of this report) were made while new top heater, pedestal, and power supply subassemblies were being engineered and built. Installation of all the new subassemblies was accomplished in this reporting period with very little downtime.

The final configuration of the furnace chamber is virtually indistinguishable from growth station No. 15 previously described and illustrated.¹ The puller is a ball screw driven fixed-stroke machine capable of pulling ribbon at up to five inches per minute. A Copy of Design and a Standard Operating Procedure were delivered to JPL during this reporting period. Experimental growth runs have been made on the system since the changeover. In its present form the furnace is comprised of a vertical cylindrical graphite crucible heating element insulated with concentric carbon and graphite felt heat shields. Die heaters, or trimmers, include ribbon face heaters in parallel and end heaters controlled independent of one another. Moly and grafoil shields and a graphite fiberform package complete the top insulation. Thermal gradients between the two zones are being measured now. Real control of ribbon spreading and left-right balance is achieved with the end heaters. A molybdenum heat shield package to replace the high surface area graphite insulation elements is being planned in order to help reduce impurities in the system and improve thermal efficiency.

The current status of this furnace (designated JPL No. 2 under this program) is fully modified as planned and fully operational. In addition to the moly shield pack engineering plans being considered for the furnace include an all metal tie-in to the house high-purity argon system, a bellows above the furnace to better control gas flows, and relocation of the hot zone to improve the view of the operation.

The JPL No. 2 furnace, above, does not have the capacity for growing 2 in. and 3 in. wide ribbon - a goal of this program. The system designated JPL No. 1 will have that capability. This new system is currently being designed. Parts already specified are being ordered (Table I); the power supply for the crucible heating element has arrived. The system will be made up of a puller frame weldment supporting a 6 ft dovetail slide, a pulling head mounted on a ball screw driven slide saddle, and a furnace chamber mounted on another slide saddle.

A commitment has been made to horizontal furnace element geometry² so that the rectangular horizontal and vertical thermal sections approximate the geometry of the product, rather than obfuscate experimental results as in the case of the vertical cylindrical geometry systems used to date. Otherwise, no commitment as to hot zone design specifics or materials has been made. All planning has been aimed at a system flexible enough to accommodate as wide a variety of sub-assemblies and experimental variables as possible - including continuous melt replenishment.

July 1, 1976 is the turn-on milestone date for JPL No. 1. At that time it will be a system with the capacity to produce 5 ft long, 3 in. wide ribbon at speeds up to 3 in./min. By that time, new thin/fast technology from JPL No. 2 should be contributing to JPL No. 1 system or experiment planning.

Orientation Studies

In order to determine the effects of seed orientation on the orientation and defect structure of the ribbons, a number of ribbons were grown using seeds of different orientations. There has also been some indication that the seed orientation is related to the morphology of the defects generated in EFG crystals.³

Typical defects occurring in EFG ribbons include linear parallel boundaries, intersecting boundaries and SiC inclusions. Among these, it has been established that the SiC particles and intersecting boundaries are detrimental with respect to their influence on electrical behavior and solar cell characteristics.^{3, 4, 5} The linear boundaries are largely quiescent in that > 10% efficiency solar cells can be fabricated

Table I. JPL No. 1 Item Procurement Schedule

Item	Cost Anticipated/ Expenditure	Date Ordered	Date Promised	Date Received	Service Requirement	Comments
Power supply 25 kW 3 Φ , 208, 220, 440 V	2500/2500	1/12/76	6 wks 2/23	3/5/76	Recirculated water, 25 kW, wiring.	Top heater power supply will draw an additional 8.5 kW. Furnace chamber will require water also.
Puller frame weldment	1400/1500	3/8/76	3-4 wks 4/5		Rigging and assembly.	
Puller slide	4800/5500	3/8/76	10 wks 5/17		Rigging and assembly.	
Puller motor, controller, power supply.	1150/1160	Wk of 3/8	10 wks 5/17		Wiring, circuit design.	
Furnace chamber	3500/				Recirculating water.	
Vacuum pump.	600 0.700/				200 V	
Shield pack Mo/Mo-Ta	1000/					
Top heater power supply	800/				Wiring circuit design.	8.5 kW voltage not spec.
Graphite parts (crucible holder, heat shields, heating elements, etc.)	300/				Design, Mach. Shop.	For running with and without cartridge or womb.
Controllers	2 at 400/				Wiring circuit design.	
Mo parts	600/				Engineering, Mach. Shop	
Cartridge parts 3"/2"	1100/				Engineering, Mach. Shop	

in ribbons containing linear boundaries. Consequently the factors during growth that promote the formation of linear boundaries rather than intersecting boundaries are of interest.

The sustained growth of long ribbons has been observed to result in an essentially uniform structure consisting of linear parallel boundaries largely parallel to the ribbon edges. These linear boundaries have been determined to be predominantly composed of twins. The attainment of a structure consisting essentially of parallel twins parallel to the ribbon edges suggests that following the establishment of steady-state growth conditions the defect structure of the ribbons essentially reaches a steady-state equilibrium configuration.

Orientation experiments involving variously oriented seeds have been conducted with a view towards determining whether an "equilibrium defect" structure develops in EFG ribbons and if so what the effects of seed orientation would be on the establishment of such a structure.

Table III lists the seed orientations used as part of the investigation. Seeds with (111) orientations were chosen because (111) planes are the preferred twin planes in diamond cubic materials. It has been reported that EFG silicon ribbons have a preferred surface orientation of (110)^{5,6}. (110) is a low index, high-symmetry plane. Hence, (110) seeds with different growth directions were chosen. A ribbon seed was also chosen for comparison with the (110) [211] seed.

Ribbon growth runs Nos. 14-148 through 14-156 comprised the growth orientation study - a total of 32 growth attempts were made (Tables II and III). The results are characterized below. For the orientation study, the growth process was standardized as nearly as possible. The furnace arrangement included an active one-piece ribbon face heater (this, prior to the state-of-the-art conversion described earlier) as well as the crucible main heater. A flat molybdenum heat shield sat on insulating stand-offs on the face heater. A two-piece graphite die with a 12 in. radius of curvature was used for all these orientation runs. Growth was from a 3 in. diameter quartz crucible contained in a graphite shell. Growth run results from 14-153 and -154 are anomalous with reference to ribbon thickness and growth rate (these two dimensions are interdependent). On run 14-153 a modified top heater had replaced one lost the previous run. Run 14-154 was replaced with a new standard top heating element which was shorted to the heat shield throughout the run. For run 14-155 a debugged standard element was used and ribbon thickness and growth rates returned to normal.

Table II. Growth Runs

Run No.	Date	Purpose	Die	Insulation	Comments
14-148	1/6/76	To establish a gradient for orientation experiments.	C-10015-14	Molybdenum radiation shield fiberform insulation.	Thermal system suitable for orientation studies.
14-149	1/7/76	Growth orientation Study No. 1.	C-10015-14		Seed: (111) [110]
14-150	1/9/76	Growth orientation Study No. 2.	C-10015-14		Seed: (110) [211]
14-151	1/13/76	Growth orientation Study No. 3.	C-10015-14		Seed: (110) [110]
14-152	1/14/76	Growth orientation Study No. 4.	C-10015-14		Seed: (100) [110]
14-153	1/15/76	Growth orientation Study No. 5.	C-10015-14		Seed: (111) [110]
14-154	1/19/76	Growth orientation Study No. 6.	C-10015-14		Seed: (111) [211]
14-155	1/21/76	Growth orientation Study No. 7.	C-10015-14		Seed: (110) [111]
14-156	1/23/76	Growth orientation Study No. 8.	C-10015-14	Fiberform	Seed: As-grown ribbon with many parallel twins.
14-157	2/6/76	To test the remodeled growth stations.	C-10166-6		Thermocouple failure; manual control used.
14-158	2/10/76	To test remodeled station with more power to end heater.	C-10166-6		Silicon failed to melt completely.
14-159	2/12/76	To introduce better insulation.	C-10166-6		Lower zone 140° setting required to maintain melt
14-160	2/18/76	To further improve the insulation.	C-10166-6		Molybdenum shield warped and shorted out top heaters.
14-161	2/20/76	To grow from a one-piece die with improved insulations.	C-10436-2		2.9 in./min grown for 1 inch.

Table II (continued)

Run No.	Date	Purpose	Die	Insulation	Comments
14-162	2/24/76	Fast growth from a one-piece die.	C-10436-2	Flat Mo shield over face heaters, graphite ring over heating element fiberform package.	Meniscus changes from smiling at seeding to frowning at 1.5 in/min.
14-163	3/1/76	To evaluate fast growth from a one-piece die with Baffle Shield (Dwg. No. B-10745).	C-10436-2	Same as above with the substitution of a Mo Baffle Shield over face heater.	Baffle shield makes full width growth easier.
14-164	3/3/76	Fast growth from a one-piece die with Baffle Shield.	C-10436-2	Same as previous experiment.	Die cracked while heating up before filling.
14-165	3/5/76	Fast growth from a one-piece die.	C-10436-2	Fiberform and Baffle shield.	Thermal profile measurements made.
14-166	3/10/76	Fast growth from a two-piece die.	C-10166-6	Fiberform and flat moly shield.	Thermal profile measurements made.

Table III. Growth Run Results

Ribbon No.	Seed	Length (in.)	Avg. Width (in.)	Thickness (mils)	Max. Growth Rate (in./min)	Comments
14-148-1	(111) [110]	7	1	7	80 sec/in.	Two freezes.
-2	(111) [110]	15	1	7	0.7	Stable growth.
-3	(111) [110]	18	1	7	0.7	Thicker at ends.
14-149-1	(111) [110]	9	1	7	0.8	
-2	(111) [110]	21	1	7	0.9	
-3	(111) [110]	22	1	7	0.8	Very stable when growing full width.
14-150-1	(110) [211]	18	1	7	0.8	
-2	(110) [211]	25	1	7	0.75	
-3	(110) [211]	6	1	7	0.75	Parallel twins and oblique 30° twins.
14-151-1	(110) [110]	4	0.5	-	-	Twinning places at 37°.
-2	(110) [110]	24	1	7	1.15	Low angle twins.
-3	(110) [110]	1	1	7	-	Froze at 1 inch.
-4	(110) [110]	32	1	7	0.9	Many parallel twins.
14-152-1	(100) [110]	4	1	7	0.8	60° twins from seed.
-2	(100) [110]	24	1	7	0.8	Unstable growth.
-3	(100) [110]	1	1	7	0.8	Froze on sight.
-4	(100) [110]	13	0.9	7	0.6	No parallel twins.
14-153-1	(111) [110]	5	0.75	17	1.15	Many carbides.
-2	(111) [110]	14	0.75	17	0.8	35° twin from seed.
-3	(111) [110]	3	0.5	-	0.5	High spot in meniscus.
-4	(110) [110]	14	0.75	15	0.6	More stable growth but growth structure unchanged.

Table III (continued)

Ribbon No.	Seed	Length (in.)	Avg. Width (in.)	Thickness (mils)	Max. Growth Rate (in./min)	Comments
14-154-1	(111) [211]	1	0.5	12-15	-	
-2	(111) [211]	1-1/2	0.5	15	-	Poor thermal balance caused by shorting of radiation shield to top heater.
-3	(111) [211]	14	0.8	15	0.75	
-4	(111) [211]	3	0.8	15	-	
14-155-1	(110) [111]	6	0.8	7	1.0	70°> twins from seed.
-2	(110) [111]	24	1.0	7	0.9	70°> twins from seed.
-3	(110) [111]	7	1.0	7	0.9	No parallel twins.
14-156-1	(111) [110]	9	1	7	0.95	37°> twins.
-2	As-grown ribbon.	18	1	7	0.8	Parallel twins.
-3	(111) [110]	6	1	7	1.0	Parallel twins.
-4	(111) [110]	27	1	7	0.8	Parallel twins off by a few degrees.
14-157-1	(111) [110]	2	0.5	13	1.1	
-2	(111) [110]	24	0.5	12	0.8	Gradually spreading.
14-158-1	(111) [110]	1	< 1	12	up to 1	Froze in center.
-2	(111) [110]	1	< 1	12	up to 1	
14-159-1	(111) [110]	1	< 1	12	1.1	
-2	(111) [110]	3	< 1	12	0.8	
-3	(111) [110]	20	< 1	12	1.4	
-4	(111) [110]	6	< 1	12	0.6	
-5	(111) [110]	1	< 1	12	0.8	

Table III (continued)

Ribbon No.	Seed	Length (in.)	Avg. Width (in.)	Thickness (mils)	Max. Growth Rate (in./min)	Comments
14-160-1	(111) [110]	1	< 1	10	1.1	Spreading condition achieved at 1.5"/min.
-2	(111) [110]	1	< 1	10	1.1	
-3	(111) [110]	1	< 1	10	1.2	
-4	(111) [110]	1	< 1	10	2.0	
14-161-1	(111) [110]	14	< 1	15	2	} Meniscus smiling at first, then as speed increases shape changes to a frown.
-2	(111) [110]	21	< 1	15	1.5	
-3	(111) [110]	21	< 1	15	1.6	
-4	(111) [110]	9	< 1	15	1.6	
-5	(110) [211]	19	< 1	15	1.6	
14-162-1	(110) [211]	18	3/4	12	1.5	} -3 splintered on cutting highly strained. Ripples very pronounced.
-2	(110) [211]	15	3/4	13	2.9	
-3	(110) [111]	25	1	10	2.0	
-4	(110) [111]	5	1	10	1.6	
14-163-1	(111) [211]	5	1/2	17	32 sec/in.	Ripples not as frequent.
-2	(111) [211]	3	1/2	14		
-3	(111) [211]	12	1/4			
-4	(111) [211]	30	1	11		
-5	(111) [211]	40	1	12	35 sec/in.	
14-164-1	(111) [211]	8	1/2	14	1.5	} Flood caused by cracked die changed thermal conditions.
-2	(111) [211]	5	1/2	17	1.5	
-3	(111) [211]	4	1/2	14	1.5	
-4	(111) [211]	9	1/2	17	1.5	

Table III (continued)

Ribbon No.	Seed	Length (in.)	Avg. Width (in.)	Thickness (mils)	Max. Growth Rate (in./min)	Comments
14-165-1	(111) [211]	20	0.5	17	1.6	Spread to full width at 22 inches. Spread to full width at 12 inches. Best condition where die is lowest.
-2	(111) [211]	14	0.5	17	1.6	
-3	(111) [211]	45	1	13	1.5	
-4	(111) [211]	36	1	13	2.0	
14-166-1	(111) [110]	6	0.8	12	1.6	
-2	(111) [110]	5	0.3	12	1.8	
-3	(111) [110]	3	0.4	12	1.7	
-4	(111) [110]	2	0.6	12	1.7	

Some observations on the growth of these orientation study ribbons: the defect and surface structure of the ribbons are best where the ribbon is growing stably, at full width, and at ~ 0.8 in./min; (110) [111] growth is very active right after seeding and the structure breaks down dramatically; (111) [110], (111) [211], (100) [110] liquid-solid interfaces are unstable and display facetting; (110) [211] single crystal seeds most commonly lead to growth with twins parallel to the growth direction and a smooth interface; the unstable 'orientation seeking' period between initiation of growth and steady-state growth is greatly reduced when seeded from EFG ribbons effectively (110) [211] containing twins parallel to each other and to the growth direction (the equilibrium defect structure described below).

A silicon crystal with a known surface orientation, growing along a given crystallographic direction shows first order twin traces on the ribbon surface such as those presented in Fig. 1; Table IV lists the ribbon surface orientations and growth directions following twinning on the appropriate (111) planes for the selected seed orientations. Such twin traces have been seen in that part of the ribbon which is proximal to the seed. However, interactions between primary twins leads to higher order twin generation, with a rapid breakdown of the simple matrix-twin relationships. The crystal achieves the "equilibrium" state wherein a majority of the linear boundaries lie parallel to each other, and parallel to the direction of crystal growth.

In order to establish the "equilibrium" orientation, X-ray Laue patterns were obtained at points on the ribbon one foot and two feet from the seed. The orientations were determined across the ribbon width at eight equidistant points at each of these regions. As an example, Figs. 2(a) and 2(b) show two X-ray patterns taken two feet from the seed in ribbons 14-149-3 (seed orientation: (111) [110]) and 14-154-3 (seed orientation: (111) [211]). From patterns such as those in Fig. 2, it was deduced that the predominant orientation of EFG silicon ribbon approximates to (110) [211]. Fig. 3(a) is a selected area electron channeling pattern (SAECP) from a region exhibiting the equilibrium structure and is compared to a similar pattern from the (110) [211] seed in Fig. 3(b). Note the similarities in the diffraction patterns. This preferred orientation was observed in all the ribbons after about one foot of growth, irrespective of the seed orientation. Fig. 4 shows the orientation at the end of 2 feet of growth in sections of various seed orientations. Note the clustering of ribbon orientations near the (110) pole. The position at which the equilibrium structure is established was found to be dependent on the seed orientation.

Fig. 1. Schematic of principal twin systems in silicon relative to frequently used seed and growth orientations. (After Leipold, et al. Proc. IEEE Photovoltaic Spec. Conf. Rec. 11th, 1975.

Table IV. Expected Ribbon Surface Orientations and Growth Directions after Twinning.

<u>Seed Orientation</u>	<u>Twinning Plane</u>	<u>Orientation after Twinning</u>	
(110) [1 $\bar{1}$ 1]	(111)	($\bar{1}\bar{1}$ 4)	[1 $\bar{5}$ 1]
	($\bar{1}\bar{1}$ 1)	(110)	[1 $\bar{1}$ 1]
	($\bar{1}\bar{1}\bar{1}$)	($\bar{1}\bar{1}$ 4)	[5 $\bar{1}$ 1]
	(1 $\bar{1}\bar{1}$)	(110)	[1 $\bar{1}$ 5]
(100) [011]	(111)	(1 $\bar{2}$ 2)	[4 $\bar{1}$ 1]
	($\bar{1}\bar{1}$ 1)	(12 $\bar{2}$)	[011]
	($\bar{1}\bar{1}\bar{1}$)	(1 $\bar{2}$ 2)	[011]
	($\bar{1}$ 11)	(122)	[4 $\bar{1}$ 1]
(110) [1 $\bar{1}$ 2]	(111)	($\bar{1}\bar{1}$ 4)	[7 $\bar{1}$ 2]
	($\bar{1}\bar{1}$ 1)	(110)	[1 $\bar{1}$ 2]
	($\bar{1}\bar{1}\bar{1}$)	($\bar{1}\bar{1}$ 4)	[172]
	($\bar{1}$ 11)	(110)	[55 $\bar{2}$]
(110) [1 $\bar{1}$ 0]	(111)	($\bar{1}\bar{1}$ 4)	[1 $\bar{1}$ 0]
	($\bar{1}\bar{1}$ 1)	(110)	[1 $\bar{1}$ 4]
	($\bar{1}\bar{1}\bar{1}$)	($\bar{1}\bar{1}$ 4)	[1 $\bar{1}$ 0]
	(1 $\bar{1}\bar{1}$)	(110)	[1 $\bar{1}$ 4]
(111) [1 $\bar{1}$ 0]	(111)	($\bar{1}\bar{1}\bar{1}$)	[1 $\bar{1}$ 0]
	($\bar{1}\bar{1}$ 1)	(151)	[1 $\bar{1}$ 4]
	($\bar{1}\bar{1}\bar{1}$)	(115)	[1 $\bar{1}$ 0]
	(1 $\bar{1}\bar{1}$)	(511)	[1 $\bar{1}$ 4]
(111) [2 $\bar{1}$ 1]	(111)	($\bar{1}\bar{1}\bar{1}$)	[2 $\bar{1}$ 1]
	($\bar{1}\bar{1}\bar{1}$)	(151)	[2 $\bar{1}$ 7]
	($\bar{1}\bar{1}$ 1)	(115)	[27 $\bar{1}$]
	($\bar{1}$ 11)	(511)	[25 $\bar{5}$]

(a)

(b)

Fig. 2. Orientation of ribbon two feet from the seed: (a) seed orientation: $(111) [110]$; (b) seed orientation: $(111) [211]$. The predominant orientation is close to $(110) [211]$, irrespective of seed orientation.

(a)

(b)

Fig. 3. Selected area channeling patterns from (a) the equilibrium structure and (b) (110) [211] seed. Note the similarity in the two patterns, suggesting that the equilibrium structure is close to a (110) [211] orientation.

Figs. 5 and 6 show regions of ribbons near the seed for the (111) [110] and (111) [211] seed orientations, respectively. These show predominantly first order twin generation. However, the stable defect configuration is not achieved in these cases till much later in the growth process. In contrast, the stable orientation forms immediately after seeding when either the (110) [211] seed or a ribbon seed is used. Figs. 7 and 8 show ribbon starts using such orientation seeds.

In order to further examine the twinning orientation possible, a region close to the seed was chosen since the initial twin traces should correspond to first order twins making comparisons with theoretical predictions feasible. The seed orientation was (111) [110]. Figure 9 is an example of such a region. It was found that the orientation of regions 1 and 2 was (111), while region 3 had a (511) orientation. Thus region 3 is a first order twin obtained after twinning on the (111) planes. Regions 1 and 2 have the same orientation. This can be due to (1) both regions being related in a matrix-twin relation, or (2) the linear boundary separating the two areas may be composed of multiple twins. A high magnification photograph of the twinned region does not clearly reveal the multiple-twin nature of the boundary. A detailed examination of the nature of the linear boundaries is under investigation at present.

Solar Cell Data

1 cm × 2 cm solar cells were fabricated in a number of ribbons grown by using different seed orientations. Since in all cases the regions selected to fabricate the cells were well removed from the seed-crystal interface, the defect structure was similar in all the cells. The structure observed was the above mentioned "equilibrium defect" structure consisting of parallel linear boundaries. The cells were fabricated with standard phosphorus diffusion techniques and aluminum-silver metallization. Table V shows typical data. The performance limits observed are related to the relatively high densities of lifetime reducing impurities in the ribbons. With better die purification procedures and improved insulation materials in the resistance heated furnace, an improvement in the performance of the cells should result and approach the characteristics of those ribbons grown from RF heated systems (> 10% AM1 efficiency). Consequently the solar cell data from current ribbons are only of interest to the extent that they provide a continuing indication of the chemical purity of the ribbons. The chemical purity may be related to other than the obvious factors such as die purification procedures and the "chemistry" of

Fig. 5. First order twin generation in ribbon next to seed (orientation of seed: $(111) [110]$). This seed orientation gives rise to intersecting boundaries initially. However, twin reactions lead to the generation of parallel twins and the equilibrium structure, as the crystal grows. Arrow indicates seed-crystal interface. Magnification: 7.5 X.

Fig. 6. First order twin generation in ribbon next to seed (orientation of seed: $(111) [211]$). Arrows indicate seed-crystal interface. Note that there are no parallel boundaries, parallel to the crystal growth direction. Parallel boundaries form as crystal grows, and the equilibrium structure is eventually attained. Note also the generation of slip bands in both ribbon and seed. Magnification: 8 X.

Fig. 7. Generation of parallel twin boundaries next to the $(110) [211]$ seed. Arrows indicate seed-crystal interface. The $(110) [211]$ seed orientation gives rise to parallel twins immediately after seeding. The few intersecting boundaries seen transform, as a result of twin interactions, into the equilibrium structure as the ribbon grows. Magnification: 6.5 X.

Fig. 8. Generation of parallel twin boundaries next to the ribbon seed. Arrows indicate the seed-crystal interface. As can be seen, the linear boundaries in the seed propagate into the growing ribbon. Observe some of the intersecting boundaries generating parallel twins. The structure very quickly attains the equilibrium orientation in this case. Magnification: 6.5 X.

(1-2)

(3)

Fig. 9. Region of ribbon near the seed. Seed orientation was $(111) [110]$. Primary twinning on (111) planes can be seen. Regions 1 and 2 have the same orientation, i.e., (111) , while region 3 has the (511) orientation. These orientations conform to those expected for the given seed orientation.

Table V. Summary of Solar Cell* Properties

Ribbon No.	Cell No.	Area (cm ²)	V _{oc} (V)	I _{sc} /A (mA/cm ²)	Fill Factor	I _{Leak} (μA)	P _{max} /A (mW/cm ²)
14-149-2	4209	Assume 2.35	0.547	17.0	0.77	50	7.2
14-149-3	4210	↓	0.510	13.5	0.60	600	4.2
14-154-3	4211		0.525	15.2	0.71	50	5.7
14-154-3	4212		0.525	14.2	0.73	40	5.4
14-149-2	4450		0.517	14.7	0.61	400	4.6
14-150-2	4452		0.536	15.3	0.69	100	5.7
14-150-2	4453		0.528	14.9	0.68	150	5.4
14-153-2	4455		0.502	11.4	0.72	175	4.1
14-155-2	4456		0.492	12.8	0.54	800	3.4
14-156-1	4457		0.534	15.8	0.69	200	5.8
14-156-2	4458		0.513	13.8	0.68	40	4.8
14-156-2	4459		0.534	15.9	0.72	30	6.0
14-156-4	4460		0.529	15.7	0.68	50	5.7

*Cells not AR coated.

furnace components such as the insulation material. Other factors of interest would be the effects, if any, of growth speed on impurity incorporation, and the effects, if any, of ribbon width on impurity distribution.

Thin and Fast Growth

Theoretical Thin and Fast Ribbon Growth by EFG

Among the major objectives of this program are the growth of uniformly thin ribbons and the achievement of fast growth rates. This section summarizes the current theoretical understanding of thin and fast ribbon growth, outlines the way in which experimental approaches are expected to be guided by theory, and identifies the problem areas where further theoretical work will be carried out during this program.

The Growth of Thin Ribbons

The theory of crystal shape control in EFG has received considerable attention during a recently concluded NSF/RANN program.^{7,8} It has been found that the ribbon thickness is related to the die and meniscus geometries by an expression of the form:

$$t = f(t_d, s, h_{eff}, \phi_o), \quad (1)$$

where t_d is the die thickness, s is the height of the meniscus or growth film, h_{eff} is the effective height of the growth interface above the liquid level in the crucible, and ϕ_o is the angle between the growth direction and the tangent to the meniscus at the solid-liquid interface ($\phi_o = 11^\circ$ for the steady-state cross-sectional growth of silicon crystals).⁸ The functional relationship f is known;^{6,7} therefore, the ribbon thickness can be readily calculated from the other parameters which can be experimentally controlled and measured. Good quantitative agreement has been found⁹ between the experimental and theoretical values of the ribbon thickness.

The complete form of Eq. (1) predicts that the ribbon thickness decreases with decreasing t_d or with increasing s and h_{eff} ; the growth of thin ribbons can therefore be accomplished by: (i) using a thin edge-defining die dimension; (ii) growing with a high meniscus; and/or (iii) using a growth setup with a high value of h_{eff} .

Another prediction of EFG theory involves the stability of the ribbon dimensions as a function of the controllable process parameters t_d , s and h_{eff} . The theory¹⁰ shows that the side of the ribbon (i.e., the thickness dimension) is inherently stable in EFG; the stability can be quantitatively expressed by the value of the derivative $|dt/ds|$. Clearly, a lesser dependence of the ribbon thickness on changes in the meniscus height (say, caused by thermal fluctuations) implies more stable edge-defined growth. The analysis shows that $|dt/ds|$ decreases (i.e., stability increases) with decreasing meniscus height and with decreasing h_{eff} . The uniformity of the ribbon thickness along the width of the ribbon is, of course, also influenced by the value of $|dt/ds|$. Considerations of the width dimension of the ribbon have also been made; the geometrical stability here depends on the relationship between the radius of curvature of the ribbon at the ribbon edges and the effective edge-defining die radius. Again, the width stability increases with decreasing meniscus height and with decreasing h_{eff} .

The experimental approaches to thin ribbon growth are thus clearly guided by the theory; the controllable empirical parameters are t_d , s and h_{eff} . Further theoretical work in this area will involve a complete quantitative description of the geometrical relationship in Eq. (1) for the range of experimental variables of interest in thin ribbon growth, and a detailed examination of the effects of s and h_{eff} on the stability of the ribbon shape. The geometrical relationship and the stability concepts will be extensively investigated by appropriate ribbon growth experiments.

Fast Ribbon Growth

The rate of ribbon growth is limited by the rate at which latent heat which is generated at the growth interface by solidification can be removed by conduction through the ribbon and by radiation from the ribbon surface. The requirement of steady-state heat balance at the growth interface is that the rate of heat removal by conduction-radiation through the ribbon be equal to the sum of the latent heat term and the rate at which heat arrives at the interface by conduction through the meniscus film. This leads to an expression relating the ribbon thickness to the gradient in the liquid and the growth rate:^{6,7}

$$G = k_l^{-1} [\alpha f(\beta) t^{-1/2} - L V_g] , \quad (2)$$

where

$$\beta = T_m/T_o \text{ and } f(\beta) = [1 - \beta^{-4} (1 + 4 \ln \beta)]^{1/2} . \quad (3)$$

In the above expressions, V_g is the growth velocity, L is the latent heat of fusion per unit volume of solid, k_l is the thermal conductivity of liquid silicon, G is the temperature gradient in the liquid ahead of the growth interface, T_m is the melting temperature of silicon, and T_o is the effective temperature of the radiating environment. Also, $\alpha = (\sigma \epsilon k_m T_m^5)^{1/2}$, where σ is the Stefan-Boltzmann constant, ϵ is the emissivity of the ribbon surface and k_m is the thermal conductivity of solid silicon at the melting temperature.

The highest growth rate under the above conditions is therefore achieved when the environment temperature is 0°K (i.e., $f(\beta) = 1$). The maximum growth rate for a given ribbon thickness then corresponds to the condition where the gradient in the liquid is zero; therefore, we have

$$(V_g)_{\max} = L^{-1} (\sigma \epsilon k_m T_m^5)^{1/2} t^{-1/2} . \quad (4)$$

The direction of the experimental effort to achieve fast ribbon growth rates is therefore: (i) to minimize the gradient in the meniscus film; (ii) to design the shields/afterheaters to prevent, as much as possible, the radiation of heat to the ribbon from the components beneath the die top (i.e., to approach the $T_o = 0^\circ\text{K}$ condition); and (iii) to grow thin ribbons. The latter point is worth further consideration at this juncture. Equation (4) or its equivalents in other melt growth techniques is often interpreted in the literature to suggest a unique relationship between growth rate and the appropriate crystal dimension; therefore, in ribbon EFG, the faster the growth rate, the smaller should be the ribbon thickness. This is totally erroneous, however. The ribbon thickness in EFG is determined solely by the geometrical condition in Eq. (1); the growth rate for that ribbon thickness then obtains from the heat balance condition in Eq. (2) for the given gradient in the liquid. Therefore, the same ribbon thickness can be grown, in principle, over a wide range (0 to $(V_g)_{\max}$ in Eq. (4)) of growth rates, and attempts to correlate V_g and t should take into account the possible variations of the gradient G in the liquid. Similar conclusions to the above can be made to other melt growth techniques. A more useful way to interpret Eq. (4) is that it represents the maximum ribbon thickness for a desired growth rate.

Recent theoretical work in this area (which will be pursued on this program) involves the determination of the temperature profile in the ribbon as a function of the radiating and cooling environments. Most of this work is related to the study of thermal stresses in the ribbons, as discussed in the next section, but it is also relevant insofar as the ribbon growth rate is concerned. The analysis assumes that the heat flow is one-dimensional, i.e., the temperature profile along the vertical (or crystal pulling) axis is calculated. A numerical analysis program has been developed which can, at the present time, calculate the steady-state thermal profile in the ribbon under the following conditions: (i) an arbitrary afterheater geometry; (ii) an arbitrary temperature distribution in the afterheater; (iii) convective heat transport in the ribbon; (iv) radiative and/or convective cooling at the ribbon surface; (v) thermal conductivity constant or proportional to $1/T$; (vi) arbitrary emissivities of the radiating surfaces; and (vii) arbitrary, constant temperature at the cold end of the ribbon. The temperature profile is printed in graphical form by the computer.

One significant refinement to the earlier theory^{6,7} which gave Eqs. (2) to (4) has been the inclusion of convective heat transport (i.e., the specific heat carried by the moving ribbon) into the numerical calculation of the steady-state temperature profile. The effect of this term on the thermal profile turns out to be quite important, as illustrated in Fig. 10. Figure 10(a) shows the vertical temperature of distribution in a stationary ($V_g = 0$) ribbon 4 cm long, 0.025 cm (~ 10 mils) thick for radiation into a 0°K environment, whereas Fig. 10(b) is the steady-state profile for a ribbon growing at 0.117 cm/sec (~ 2.76 in./min). The latter rate corresponds to the condition where the gradient in the liquid is approximately zero, and is therefore the maximum ribbon growth rate for a 0.025 cm thick ribbon. Equation (4) which neglects the convective heat transport predicts, on the other hand, that $(V_g)_{\max} = 0.146$ cm/sec. Table VI lists the values of $(V_g)_{\max}$ obtained from Eq. (4) and the corrected values including the convective transport as a function of ribbon thickness; it is seen that the former predictions are approximately 20% too large.

In summary, therefore, the experimental approach to fast ribbon growth is well understood; the highest growth rate in a passive system (i.e., one without active cooling, such as by gas convection) can be achieved by a radiating environment which, in essence, is at 0°K. Meaningful calculations of the vertical temperature profile in the ribbon for a variety of radiating and cooling conditions must perforce involve numerical analysis on the computer; theoretical work is well underway in

Fig. 10. Calculated vertical temperature profiles in a 0.025 cm thick, 4 cm long ribbon for (a) $V_g = 0$ and (b) $V_g = 0.117 \text{ cm/sec}$. The convective heat transport is included in the calculations. Radiation into a 0°K environment is assumed; the temperature at the far end of the ribbon is fixed to 400°K . The arrow denotes the position of the growth interface on the printout.

Table VI. Maximum Ribbon Growth Rate as a Function of Ribbon Thickness

t (cm)	$(V_g)_{\max}$, cm/sec (using Eq. (4))	$(V_g)_{\max}$, cm/sec (corrected value)*
0.01	0.231	0.179
0.02	0.163	0.130
0.025	0.146	0.117
0.03	0.133	0.107
0.04	0.116	0.094
0.05	0.103	0.084

*The corrected value is obtained by numerical solution of the steady-state temperature profile; the model includes the convective heat transport (i.e., specific heat) by the moving ribbon. Radiation into a 0°K environment is assumed.

this area. Experimental measurements of the thermal profile under actual ribbon growth conditions will be carried out to compare with the theoretical predictions. The theory will then be used to guide the empirical development of the ribbon environment to achieve fast growth rates and tolerable residual stresses in the grown ribbons.

Thermal Stresses in EFG Ribbons

Prior experience during the growth of ribbons at rates in excess of 5 cm/min (using the appropriate "cold" radiative environment) has indicated the existence of large residual stresses in the grown ribbons. In addition to its effects on the ribbon structure (dislocations, etc.), the stresses cannot be tolerated if they result in the ribbon splitting or shattering during the subsequent process of cutting for solar cell blanks. The residual stresses undoubtedly result from the plastic deformation which the ribbons undergo at the high temperatures. The plastic deformation, in turn, is caused by the thermal stresses which arise as a result of non-uniform temperature distributions in the solid; if the thermal stresses exceed the yield stress of the material at the given temperature, plastic flow will result.

In an attempt to obtain a quantitative assessment of the effects of various temperature distributions on the residual stress in the ribbon, we have carried out a theoretical analysis¹¹ of the thermal stress in the ribbon prior to the beginning of this program. A principal prediction of the theory is that if the steady-state temperature varies linearly with a set of rectangular Cartesian space coordinates, then all the stress components are identically zero throughout the solid (provided that surface tractions, body forces and displacement discontinuities are absent). Conversely, this is the only temperature distribution for which all the stress components are identically zero. It can be readily argued, however, that a linear vertical temperature distribution cannot be obtained (in view of the $1/T$ dependence of the thermal conductivity) in ribbon EFG for the usual case where the ribbon surface is cooled by radiation or convection. Even if the ribbon surface were perfectly insulated, a linear temperature distribution could not result.

The theoretical analysis¹¹ considers the case of a thin ribbon (hence the plane stress solution is applicable) with arbitrary temperature distributions along the length and across the width of the ribbon. The solution is obtained either in analytic form (for simple forms of the temperature distribution) or by numerical analysis on the computer. For the usual case of interest where the temperature profile is a function of the vertical coordinate x only, the variation of the stress components σ_{xy} , σ_{yy} and σ_{xx} with the variable y along the ribbon width is shown in Fig. 11. The normal stress component in the longitudinal direction (σ_{xx}) is seen to lead to a compressive stress in the outer portions of the ribbon, whereas the center of the ribbon is in tension. The magnitudes of the stress components as a function of the vertical coordinate depend on the functional form $T(x)$ of the temperature profile. For $T(x)$ which is appropriate for radiation into a 0°K environment (see Fig. 10(a)), the calculated thermal stress is found to be far in excess of the yield stress of silicon at the high temperatures under consideration. It is therefore not surprising that excessive residual stresses are encountered in ribbons which are grown under this condition.

The theory further predicts that the thermal stress components should be proportional to (width) ^{n} , where $n \cong 2$ to 4. Experimental results qualitatively confirm a strong dependence of the residual stress on ribbon width.

A frequent mode of deformation of the ribbons has been observed to be a low temperature macroscopic buckling along the longitudinal axis which results in a rippled surface; the amplitude of the ripples is typically ~ 0.4 cm and the wavelength

Fig. 11. The variation of the stress components σ_{xy} , σ_{yy} and σ_{xx} with the coordinate y across the ribbon width w . The stresses are normalized with respect to the maximum in each case.

is ~ 4 cm. From the variation of the stress components in Fig. 11, it can be readily argued that this buckling is consistent with the plastic deformation which occurs as a result of the thermal stress exceeding the yield stress at the high temperatures. Some of the ribbons grown during this period (e.g., Fig. 12) have also been observed to undergo this buckling, as reported in the next section.

In summary, it is anticipated that thermal stress will be a problem area of continuing importance during the present program in view of the fast ribbon growth rates and large ribbon widths sought. The theoretical framework for analyzing thermal stresses in EFG ribbons has been well established; the theoretical and experimental results of the thermal profile calculations described in the preceding section will be continually combined with the stress analysis to predict the residual stress in the grown ribbons. Appropriate experimental measures of the stress will, of course, also be carried out and the results will be compared with the theory. As stated above, the aim of the theory is to guide the empirical development of the proper ribbon cooling environments to achieve the optimum combination of fast growth rate and low residual stress in the ribbons.

Experimental

On completion of the orientation runs 14-148 through 14-156, the station was modified as described under 'Apparatus' above. Runs -157 through -166, have been efforts to establish a controlled thermal environment around the die top conducive to growth at a reasonable speed (Table II). The results of runs 157 through 161 are shown in Table III.

On runs 161 and 162 some material was grown at terminal speeds approaching the contract goal of 3 in./min. That is, the seed was dipped, growth was initiated even as the die temperature was being reduced, growth speed was continuously manually increased so that increasing heat of fusion would compensate for the automatically diminished top heater output, and then the ribbon froze to the die. The top speed the puller achieved just before the freeze was 2.9 in./min. That was certainly not an equilibrium growth rate for the system and obviously the run ended when the die top temperature dropped below the melting point of silicon. A new heat shield (Fig. 13) with a very narrow orifice to increase the gradient between melt and die top was fabricated. Runs 163 through 166 have been efforts to understand the relationship between the observed growth process and the system thermal geometry. A procedure for thermal profiling above the die top at the conclusion of each run has

Fig. 12. Portion of ribbon No. 14-162-3 showing ripples on ribbon surface (growth conditions are described in Table III).

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	TOLERANCES	SURFACES ✓ MICRO-INCHES	CHK		
	ANGLES $\pm .005$ FRACTIONS $\pm$ 2 PLACE $\pm$ 3 PLACE $\pm$		APPD		
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FINISH	MATERIAL		APPLICABLE DOCUMENTS		
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Fig. 13. Baffle Shield

just been established. The results and analysis will be reported during the next period.

Problems

No problems in meeting program goals have been encountered. Some program milestones have been missed by, in some cases, as much as 4 to 6 weeks. All elements of the program should be back on schedule by the middle of April.

Plans

Thin ribbon die designs will be tested and evaluated. Experimental and theoretical evidence will guide thermal modifier-trimmer design for faster low-stress ribbons. Material and solar cell characterization will continue. Efforts will be made to clean up the chamber.

Planning and building of JPL No. 1 will continue. Growth from (110) [211] seeds will become a standard operating procedure.

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CONCLUSIONS AND RECOMMENDATIONS

The results are propitious. Although the anticipated problem of frozen-in stress is real, high growth rates are feasible. It has been established that an equilibrium defect structure which does not significantly degrade solar cell performance develops in ribbons after sustained growth irrespective of the seed orientation used, and that this structure can be realized at seeding if a (110) [112] orientation seed or a ribbon containing the equilibrium structure is used for seeding.

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APPENDICES

1. An Updated Program Plan

Table VII. Note the extension of 2_1 and 2_2 to June 1 and February 1, respectively.

2. Man Hours and Costs

Previous man hours were 875 and cost plus fixed fee was \$30,129.45. Man hours for January plus February were 915 and cost plus fee was \$29,577.88. Man hours and cost for March are estimated to be 502 and \$15,975.00 respectively. Therefore, second quarter man hours are estimated to be 1417 and cost plus fee to be \$45,552.88. Cumulative two quarter man hours and costs plus fixed fee are estimated to be 2292 and \$75,182.33 respectively.

3. Engineering Drawings and Sketches Generated during the Reporting Period

Figure 13.

4. Summary of Characterization Data Generated during the Reporting Period

Significant information generated on the characteristics of ribbons includes (a) verification of the existence of an equilibrium defect structure consisting of parallel twins parallel to the edges of the (110) [211] ribbon; (b) determination that the orientation of the seed does not influence the attainment of the equilibrium structure or orientation; (c) the use of a (110) [112] seed or of a ribbon seed exhibiting the equilibrium structure and orientation leads to the establishment of the equilibrium structure and orientation at the seed crystal interface. Other orientations require sustained growth before the equilibrium structure is reached.

5. Action Items Required by JPL

None.

6. New Technology

No reportable items of new technology.

There is no cumulative new technology.

Table VII. Updated Program Plan

TASKS	1975		1976						1977											
	No.	Dec.	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	Apr.		
Machine No. 1 (wide ribbon)																				
1. Design and develop Machine No. 1																				
2. Test and use No. 1 for 2" ribbon																				
3. Develop 2" ribbon																				
4. Develop 3" ribbon																				
5. Improve machine purity (optional)																				
Machine No. 2 (thin, fast)																				
1. Design and test thin ribbon dies																				
2. Seeding and orientation experiment																				
3. Improve machine purity (optional)																				
4. Thermal geometry control																				
5. Study meniscus effect																				
6. Combine 1 through 5 above																				
7. Optimize thin ribbon growth.																				
Material and Solar Cell Characterization																				
Theoretical Thin and Wide Ribbon Study																				

Standard Operation Procedure for Mach. No. 2, Mach. No. 2 Modified.

Design Review of Mach. No. 2

Mach. No. 1 Completed, Standard Operating Procedure for Mach. No. 1

Design Review of Mach. No. 1

Achieve 2" Wide Ribbon

Achieve 0.004" Thickness Ribbon

Achieve 2"/min Growth Rate

Melt Replenishment Paper Study

Achieve 3" Wide Ribbon

Optimize Orientation

Achieve 3"/min Growth Rate

3" Wide 0.004" Thick Ribbons

MILESTONES*