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LOW ANGLE SILICON SHEET GROWTH

Large Area Silicon Sheet Task
Low Cost Solar Array Project

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Final Technical Report

JPL Contract No. 955378

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1.0 SUMMARY

This report describes the results of a program to demonstrate the feasibility of a low angle ("horizontal") silicon ribbon growth process. Twenty-six experimental runs were performed during the period from May 9 to September 9, 1979 and from December 1, 1979 to January 31, 1981. Ribbons were grown at pull rates from 5 to 68cm/min. Ribbon lengths up to 74cm were grown while widths varied from 5 to 25mm. Thicknesses varied from 0.6 to 2.5mm, with typical values of about 1mm.

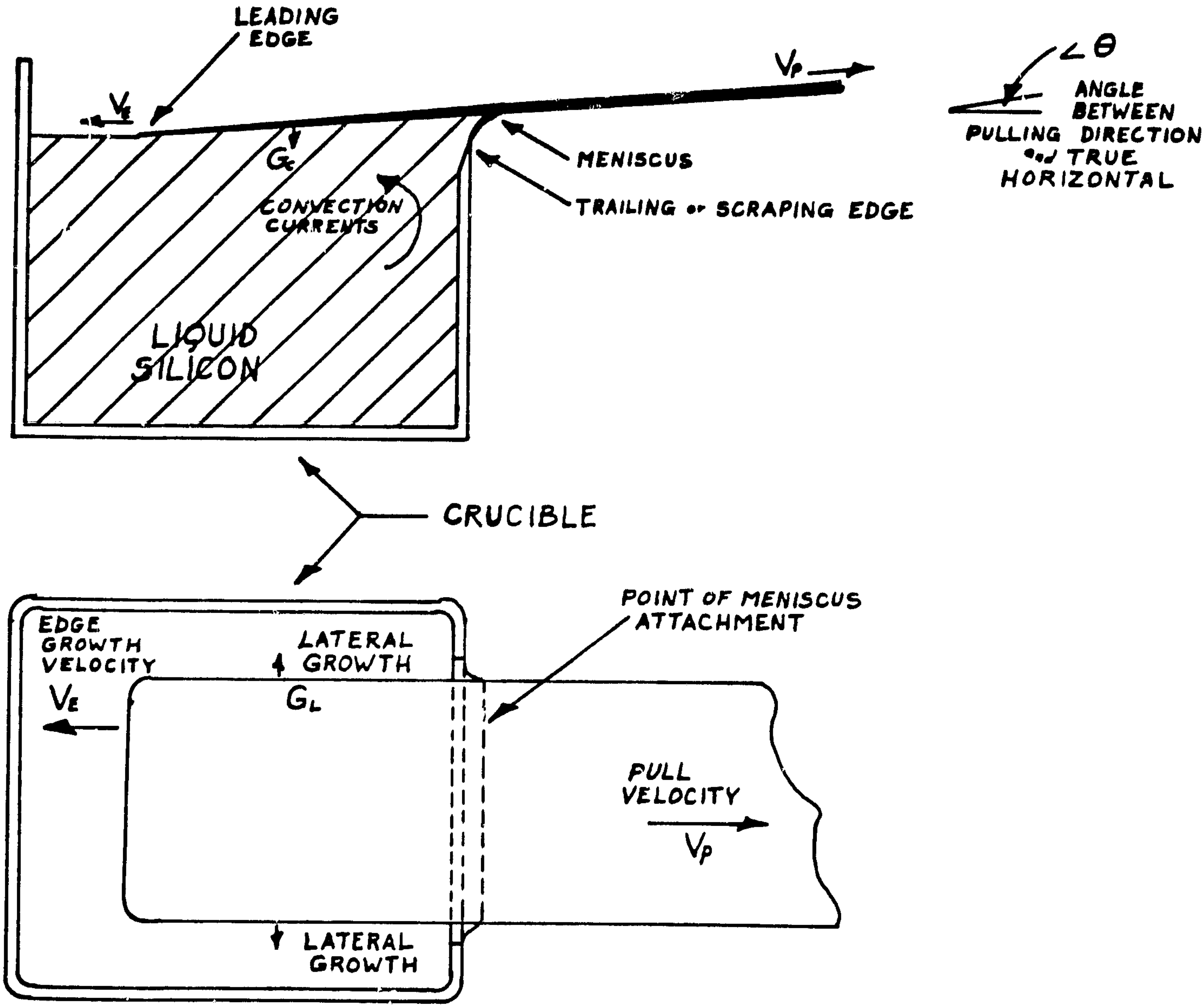
2.0 INTRODUCTION

2.1 Objectives

This effort is a feasibility demonstration of a novel approach to silicon ribbon (sheet) growth. Process features such as ribbon width control, leading edge control, and "scraper" function are to be experimentally tested. The behavior of shallow silicon melts and melt replenishment concepts are also to be studied.

2.2 Technical Approach

The crystal growth technique being tested under this contract is a variation of horizontal growth. The basic process consists simply of controlled freezing of a thin layer on the surface of the melt and pulling of the solidified material more or less parallel to the surface to some point of detachment of the solid from the melt. Figure 1 illustrates this process schematically. Thermal fields in the melt are produced by suitable means to promote growth at the leading edge while pulling in the opposite direction. Thus the leading edge becomes a constantly renewed seed for further growth in the thickness direction, at a rate G_c, as the ribbon is moved across the melt surface. A key feature of horizontal growth, which provides the high growth rates reported, is that the heat of fusion is lost by conduction through the thickness of the ribbon and subsequent radiation from the upper surface of the solid ribbon. Similarly, growth may



HORIZONTAL CRYSTAL GROWTH

SCHEMATIC DIAGRAM

FIGURE - 1

occur perpendicular to the pull axis as indicated by G_L in the plan view of Figure 1. Not only must these various growth rates be controlled, but the detachment of the meniscus from the ribbon's lower surface must also be controlled, and the instabilities generated by thermal convection currents in a deep melt must be eliminated.

Figure 1 is representative of the arrangement employed by Japanese workers who reported successful horizontal growth of silicon ribbons in the mid-1970's.[1] Despite the difficulties inherent to this particular approach to horizontal growth, they reported results indicative of the potential gains in productivity realizable with the basic technique. They reported single crystal ribbon growth at rates of 30 to 40cm/min, and maximum rates of 85cm/min, which produced dendritic ribbon. Values of mobility of majority carriers and lifetime were also reported equal to those typical of Czochralski materials.

EMC's approach to controlled horizontal (low angle) sheet growth is illustrated in Figures 2 and 3. Gradients controlling the various growth rates are established in the melt by thermal impedances within the growth trough. The meniscus is detached from the bottom surface of the ribbon by the "scraper" which provides a false crucible lip whose temperature can be effectively maintained above the melting point. The elevation of the scraper above the melt level raises the meniscus to provide increased stability to the ribbon edges. The shallow melt inhibits any

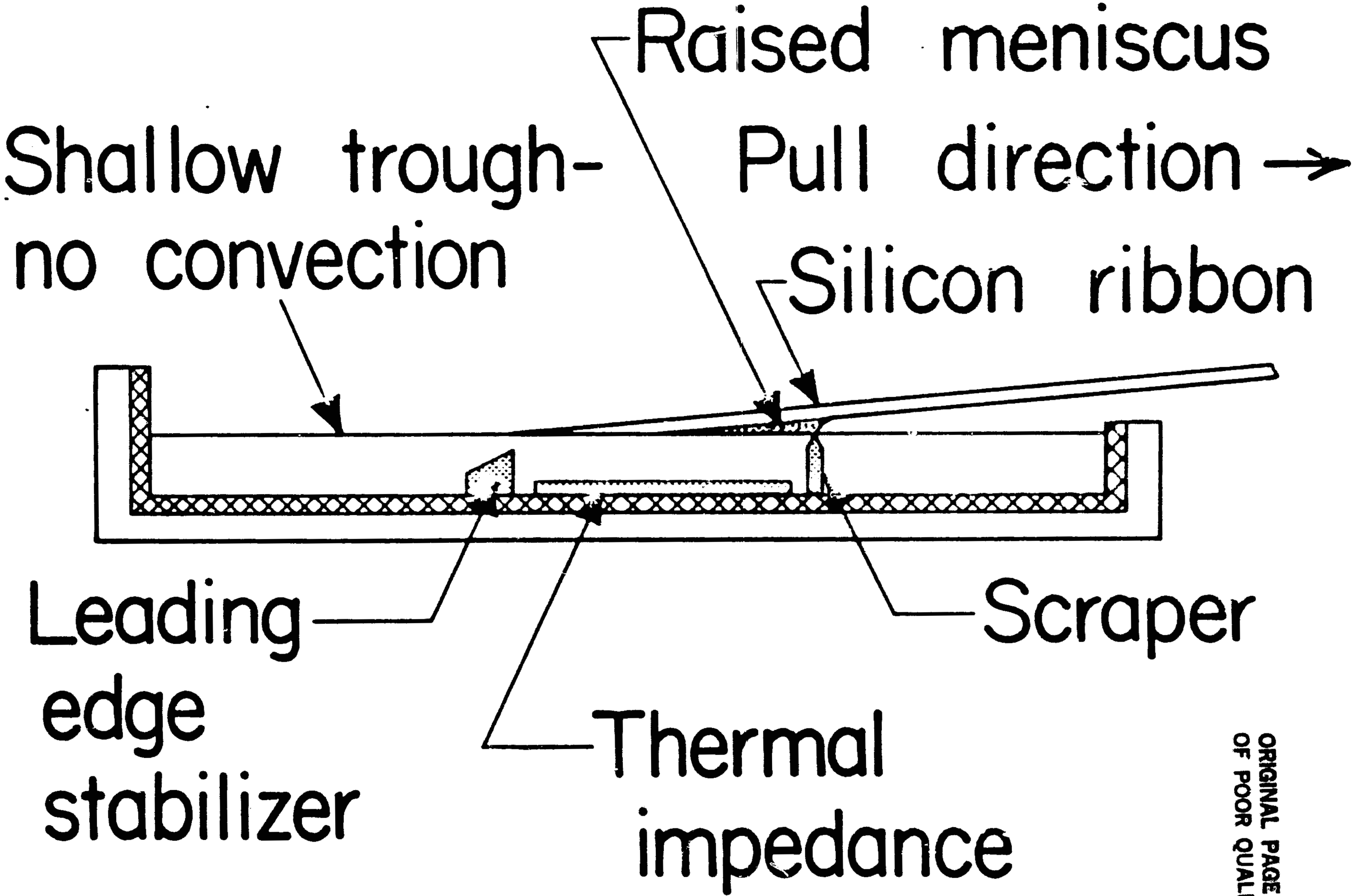


Figure 2

Schematic Diagram of EMC Approach
To Horizontal Ribbon Growth

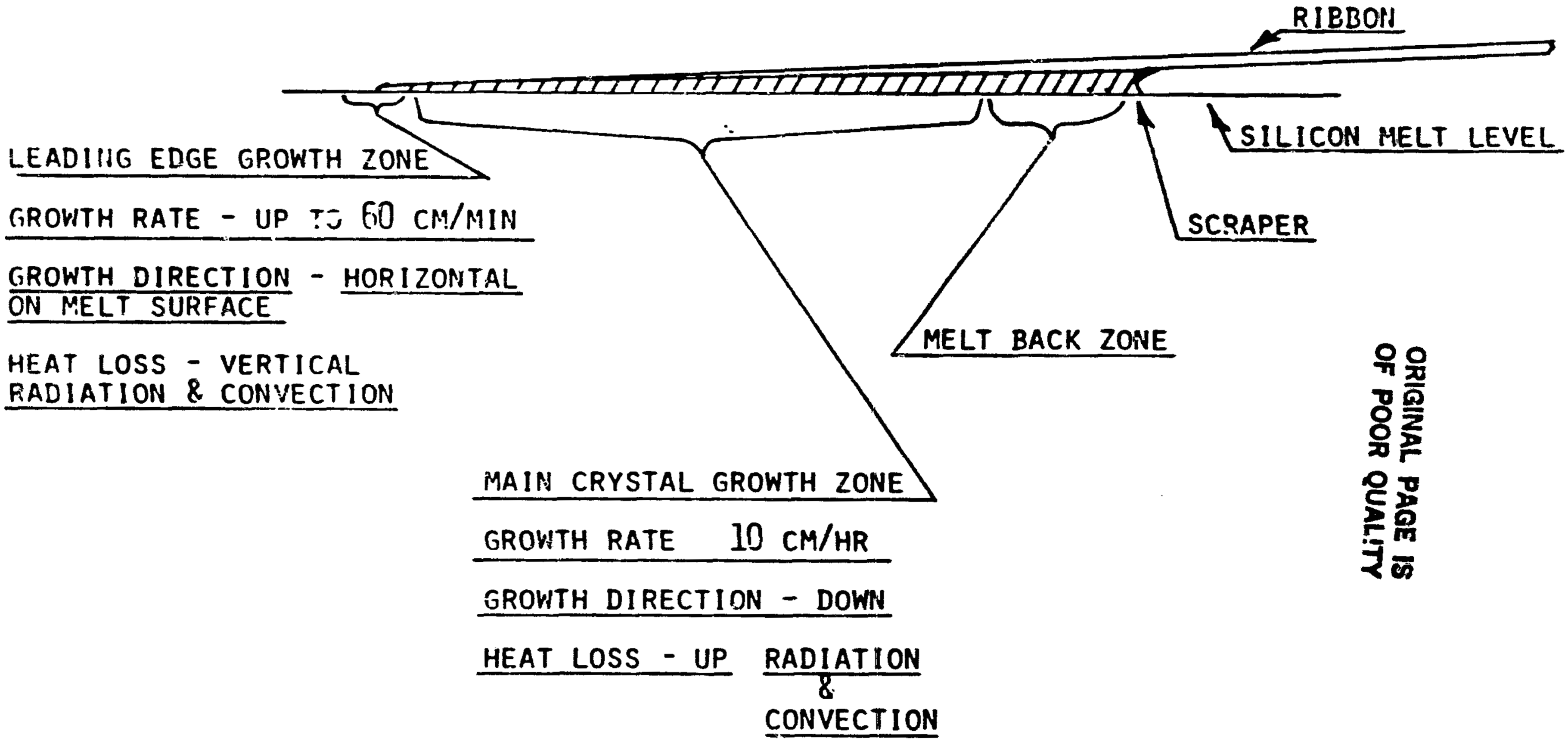


Figure 3

Horizontal Crystal Growth
Schematic Diagram

substantial convective flows which would otherwise destabilize the growth processes. Ribbon thickness is a function of the bulk growth rate, the linear pull rate and the growth zone length. Clearly, practical application of the method requires maintenance of the melt level and replenishment of the melt.

3.0 TECHNICAL DISCUSSION

3.1 Experimental Approach

3.1.1 Heater Design

The basic premise of our approach to low angle ribbon growth is the manipulation of a uniform heat flow to the crucible in order to produce the thermal fields in the melt required for controlled ribbon growth. Thus the heating arrangements are required to produce a uniform temperature over a substantial area.

The initial approach to this problem placed the shallow, rectangular quartz crucible on a thick-walled (0.5 inch) graphite support box. This box was supported from below, and allowed vertical motion of the crucible through the heater. The rectangular heater surrounded the support box on four sides and consisted of graphite rod heating elements connected at corner posts, and at each end to power feed studs angled below the horizontal to allow access to the surface of the melt. We found, however, in early experiments that this heater and support arrangement did not supply a sufficiently uniform heat flow to the crucible. It was then clear that a straightforward, large-area heater, supplying heat solely from below the growth system, would provide a more reproducible and logical heat flow situation. A heater element consisting of multiple thin

Grafoil^R sheets was designed and tested. The crucible/trough was then supported $\sim$0.4in above the heater, on a 0.25in thick graphite plate. Pyrometric measurements on such a plate indicated temperature uniformity of $\pm 5^{\circ}C$ over the area occupied by the crucible. Heavy (0.5in thick graphite) crucible sidewall supports on top of the support plate tended to distort this uniform temperature distribution somewhat. These sidewalls were gradually reduced in size until finally the crucible was being positioned on the support plate by four 0.5 x 0.25 inch studs. The evolution of the "supporting" sidewalls to mere locating pins was accompanied by improvements in shielding and insulation of the exposed crucible walls to maintain low enough temperatures that no distortion of the quartz would occur.

The trough support plate was supported from three individually adjustable (by thumb wheels on threads) rods reaching down through the furnace cover plate. This arrangement permitted modest vertical excursions of the set-up about two axes in the plane of the melt surface. The ability to adjust the attitude of the growth system appeared desirable from early runs. Our subsequent experience was that this capability is of no significant utility.

3.1.2 Crucible Design

Initial crucible design was based on a quartz bottom plate which was machined (ground) to incorporate the subsurface thermal impedance as an integral part of the one-piece bottom plate. Sidewalls were also made of flat stock, ground to provide rabbeted joints, and grooved to hold the stabilizer bars and scraper.

Two attempts were made to use the crucibles as mechanical assemblies, held by the graphite retaining walls. This was not successful, so subsequent crucibles were welded along the corners and edges while held by a heated graphite fixture.

The early crucibles employed a "Lincoln Log" approach to locating the leading edge, lateral edges and scraper with respect to each other. These pieces interlocked and the leading edge stabilizer and scraper extended to fit vertical grooves in the sidewalls. While this arrangement was precise and stable, our experience over the first several runs revealed a persistent problem with dewetting of the melt from the extension of the scraper and leading edge stabilizer. This would tend to cause freezing from the exposed quartz. The design evolved through attempts to decrease the heights of these locating extensions, to their eventual replacement by short, thin tabs which could be welded to the crucible bottom. This approach eliminated the problem of dewetting at these areas.

Some experiments were done with graphite leading edge stabilizers, scraper and plateau to examine the behavior of very shallow melt depths ($\leq$1mm) which could only be achieved by use of a material wetted by silicon.

3.1.3 "Cold Shoe" Design

After preliminary growth experiments, described in Section 3.2.1, a gas cooling block, or "cold shoe" was designed and built, in order to test the influence on growth of additional heat transfer from the seed and ribbon interface. The initial shoe was a 25mm x 25mm x 8mm OFHC copper block in the shape of a truncated wedge atop a square base, inverted, in use. The block was supplied with cooling water by two coaxial stainless steel tubes, and with gas by two stainless steel tubes (5mm ID x 6mm OD) attached to the narrow faces of the block. The gas fed from the tubes into a drilled hole (5mm) which acted as a plenum to a slot approximately 1mm wide sawn into the blunt tip of the wedge. This arrangement produced a stream of helium directed essentially perpendicularly onto the surface of the seed crystal.

3.1.4 Melt Level Control Concepts

Control and maintenance of the melt depth are important aspects of ribbon growth by the LASS process. Melt depth is a significant determinant of the temperature field experienced by the solid-liquid interface. We do not have any experimental indications yet of the sensitivity of the process to variation in the melt depth. However, we expect that control of the melt level on the order of plus or minus 0.010 inches (0.25mm) will probably be adequate. This is approximately a 2 - 3 percent variation.

The basic requirement for a melt replenishment and level control system is that the system must maintain the melt level constant. In so doing it must adjust for varying ribbon growth rates, intermittent replenishment and variations in replenishment volume. This means that the system must be able to sense <u>and</u> adjust the melt level both negatively and positively.

A variety of melt level sensing means are available including optical, mechanical and electrical. We are considering the use of a simple, counter-balanced floating quartz cup as the primary sensing element. Its position would be transmitted via a rod extending out of the system where an optical or electro-magnetic sensor would detect the vertical movement of the rod and provide an electrical control signal. Actual control of the melt level would be provided by a large quartz cup held on a rod driven up or down by a motor and headscrew in response to the signal from the floating cup sensor. The large displacer cup would drive down to compensate for lowering of the melt level as silicon is removed by a growing ribbon. In a system with intermittent silicon replenishment the displacer would be drawn upwards to maintain a constant melt level.

3.1.5 Melt Replenishment Concepts

A 10cm wide ribbon, 0.4mm thick, growing at 40cm/min will remove 16cm^3 of solid silicon from the system each minute. This will cause the melt level to decrease by 0.66mm/min, assuming a growth crucible 15cm square. Thus, to maintain a constant melt level, replenishment material must be applied at a rate of 37gms/minute for each ribbon, in the case of a multiple growth system. This rate of replenishment with solid material will require an energy input of approximately 2Kw/hr/ribbon, to provide the replenished material as liquid.

The choices in form of replenishment material, solid rod, chunk material, or possibly shot, dictate to some extent the replenishment system configuration which might adequately supply the necessary energy. Chunk and shot are probably best fed to a pre-melting crucible, separate from the growth crucible to provide isolation of splashing effects and oxide scums. The problem in such systems is to be able to regulate the liquid flow out of the pre-melter[2]. Replenishment by solid rod (as from the Seimens process) could be into a separate crucible[2], or into the main crucible as demonstrated by Mobil Tyco[2]. The lower volume throughput of the EFG process (typically, 0.9cm^3/min/ribbon at 3cm/min for 10cm ribbon, 0.3mm thick) makes the problem somewhat easier.

3.2 Experimental Results

3.2.1 Initial Experiments

A tabulation of experimental runs is presented in Appendix A. The reader is referred to this Table for details of the experiments. Discussion of the results is divided into two sections; the first deals with initial experiments to test the behavior of shallow melts and the functioning of various thermal control structures in the melt. While the second section discusses experiments which employed additional cooling of the ribbon growth. The components which are discussed herein are illustrated in the accompanying Figure 4.

The early experiments were intended to test an initial approach to the setup geometry and assembly, as well as the heater and crucible support arrangement described in Section 3.1.1. The "assembled" crucibles leaked, so that from the third run onward, the crucibles were welded together and have proved satisfactory. The runs from #3 through #6 demonstrated a number of aspects of the design which were modified. With reference to Figure 4, the early "Lincoln Log" type scraper and leading edge stabilizers extended to the crucible sidewalls, and the lateral stabilizers were located by notches in the bottom of the

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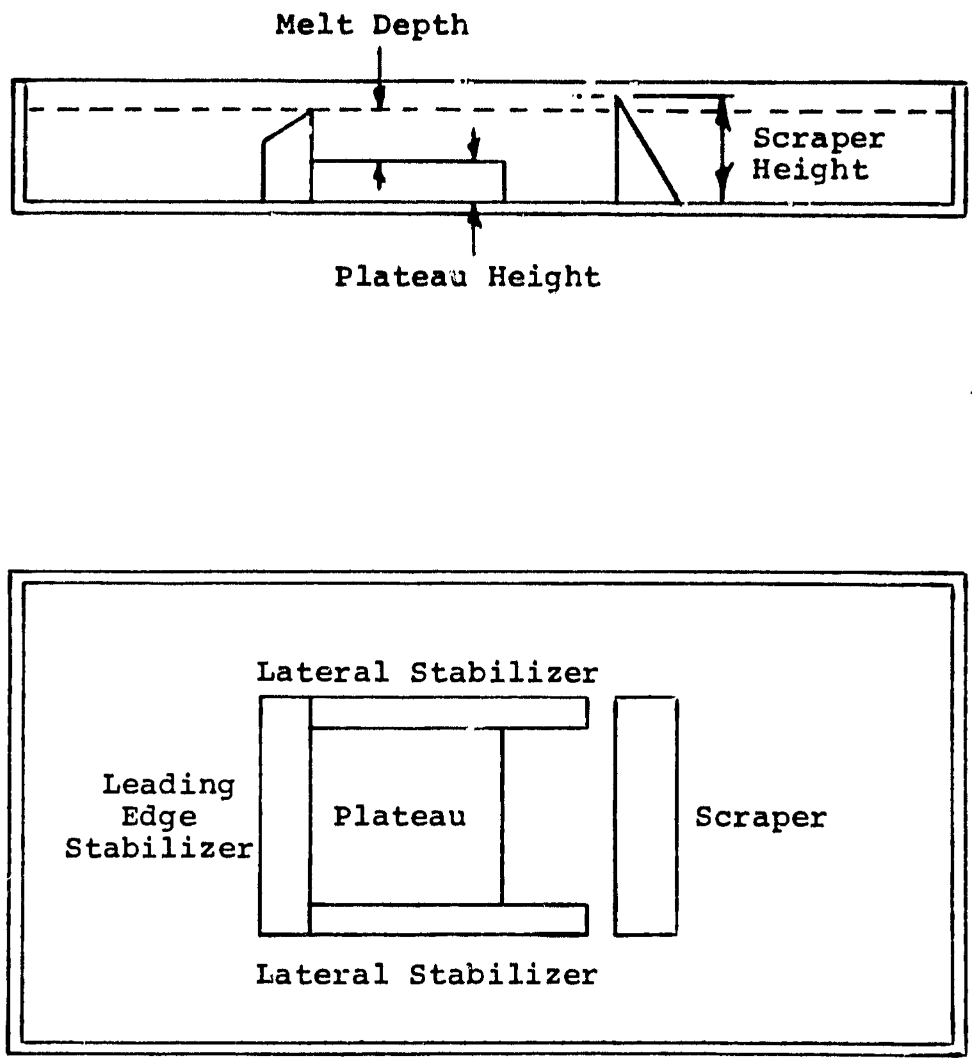

Figure 4

Schematic Drawing of Growth Crucible With Nomenclature transverse bars. Lack of wetting over the extensions of the scraper and leading edge at total melt depths of about 8mm led to their reduction to simple tabs. We also observed that wetting of the scraper on its vertical face was more complete and reproducible. This was tested in run #6 and much more uniform filling of the growth zone was obtained. Thus the scraper orientation is now as shown in Figure 4, with the sloping face of the scraper facing in the pull direction.

The next several runs (7-15) tested a variety of melt depths and subsurface component sizes. Numerous modifications were made to the shielding above the crucible. The aim of these experiments was to provide a thermal distribution in which a temperature close to, or at, the melting point would be provided across the leading edge region, a slightly higher temperature would exist over the growth area up to the scraper, and the surrounding melt would be sufficiently above the melting point to preclude spontaneous nucleation of solid Si. To provide these conditions, the heavy crucible support walls were progressively reduced in thickness; the heater configuration was radically modified in run #11. Plateau areas were enlarged, and additional thermal resistance was provided by placing insulating material under the crucible (runs 13 and 14) in the plateau region. The overall result of these many changes was insufficient. We were able to produce silicon melts of depths from a fraction of a millimeter to several millimeters, and to seed growth across the surface of the melt in these areas. We could not control the direction of growth from the seed in these experiments, nor could we maintain a sufficient temperature difference below the surrounding areas of the melt surface to prevent nucleation and freezing-out from various exposed quartz surfaces, typically the scraper or rear wall/leading edge stabilizer, all of which provided exposed quartz surfaces within the melt. Growth from a seed in these experiments was as likely to proceed toward the scraper or side walls as toward the leading edge. Growth rates, when pulling was initiated, appeared to be very slow, only a few centimeters per minute.

However, the primary difficulty encountered in these experiments was that of maintaining the peripheral areas of the growth crucible above the melting point, while producing an area at the "leading edge" of the growth zone close to the melting point. Only with the addition of extra cooling of the seed crystal could controlled growth be obtained without uncontrolled freezing from the walls of the crucible, or the scraper. Results of these experiments are described in the next section.

3.2.2 Experiments Employing Additional Cooling

A simple "cold shoe" was fabricated as described in

Section 3.1.3. The purpose of the shoe was to provide an area of cooling at the leading edge of the seed, and to initiate and maintain ribbon growth by manipulating pull speed, melt temperature, shoe position, and gas flow.

Initially an attempt was made in run 18 to provide additional cooling to the seed and crystal interface. A water-cooled stainless steel tube (6mm O.D.), bent in a squared-off U-shape with a length of about 2.5cm parallel to the melt, was inserted from the top of the furnace and positioned within a few millimeters of the interface. Seeding and growth attempts indicated little positive effect of this crude device. In run 20, after modifying the cold shoe to improve the impingement of the He stream on the leading edge area, the first directed ribbon growth was achieved. One of the ribbons grown then is shown in Figure 5 (1-101-20-2).

In subsequent runs (21 to 26) use of the cold shoe made it possible to grow ribbon with good reproducibility; however, freezing-out from the scraper and rear wall of the crucible was a persistent problem, terminating ribbon pulls more often than not. In the last few runs ribbons were grown in 75mm by 120mm crucibles without any substructure other than a scraper, and from larger crucibles. These runs seemed to demonstrate the dominant influence of the cold shoe on the growth process.

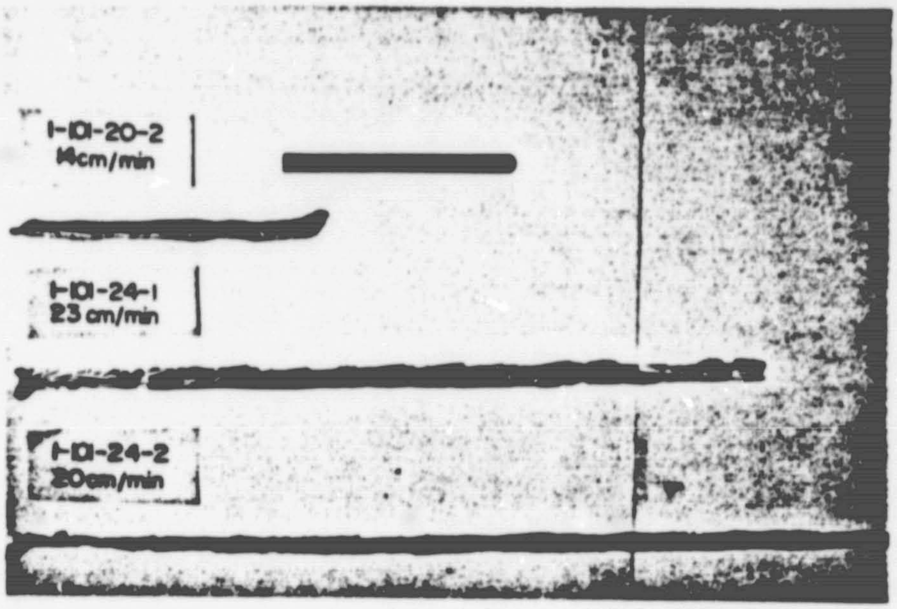

Figure 5

Silicon Ribbons Grown by the Low Angle Silicon Sheet Growth Process at Rates of 14 to 25cm/min.

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5.0 REFERENCES

1. T. Koyanagi, Proc. 12th IEEE Photovoltaic Specialists Conference, Baton Rouge, LA, 1976, p. 627.

2. D. Walters, Continuous Liquid Feed Czochralski Growth, Quarterly Report #8. JPL Contract 954886, October 1979.

3. F. Wald, et al, Quarterly Reports on JPL Contract 954355, 1977 - 1978.

4. J. Locher, et al, Quarterly Report #2 on SERI Contract XW-1-1303-1, April 1982

6.0 APPENDICES

6.1 Appendix A

Tabulation of Experimental Runs

Date	Run	Purpose	Setup	Results
May 30	1-101-1	Test quartz crucible assembly with graphite support box and "post-and-rail" heater.	"Lincoln log" substructures rabetted crucible walls.	Melt leaked from crucible during melting and filling.
June 7	1-101-2	Test Mo shims to hold crucible walls together.	" "	Apparent leaking led to shutdown. No actual leaking post-mortem.
June 12	1-101-3	Test crucible with welded joints and filling/wetting behavior.	Same substructures as previous runs.	Achieved filling of all but two areas in crucible. Lost melt by wicking into insulation. Welded joints success full.
July 6	1-101-4	Test filling/wetting behavior and seeding/growth.	Welded crucible 0.325in. scraper, 0.125in. plateau.	Filled crucible successfully. Modified shielding to provide more uniform temperatures. Flooding to insulation terminated run.
July 10	1-101-5	Test modified shielding.	Same as Run 4.	Freezing from scraper to seed. Wetting difficulties at scraper/lateral stabilizer joints.
July 13	1-101-6	Test reversed scraper and shielding of scraper.	0.325in. scraper; 0.157in. plateau; 0.260in. lateral stabilizers.	Scraper function improved. No seeded growth achieved. Freezing from scraper and rear wall.

Date	Run	Purpose	Setup	Results
July 16	1-101-7	Test leading edge stabilizer.	Same scraper and stabilizers .125in. plateau, leading edge stabilizer 0.380in.	Freezing from leading edge stabilizer when attempting to seed.
July 24	1-101-8	Test deeper melt, shielding modifications, and influence of removing impedances.	Scraper 0.46in., lateral stabilizers 0.360in.. No leading edge or plateau.	Freezing from scraper and rear wall. Power supply shorted ending run.
July 25	1-101-9	Test graphite leading edge and plateau.	Scraper 0.47in., plateau 0.250in., leading edge 0.390in., lateral stabilizers 0.400in.	Melt over plateau seemed cooler, still troubled with freezing from scraper and walls.
July 30	1-101-10	Test of higher plateau/leading edge (shallower melt depth).	Same scraper and lateral stabilizers. Plateau 0.420in., leading edge 0.460in.	No improvement in freezing-out of sites outside growth area.
August 3	1-101-11	Test new heater and support design with greater melt depths over graphite substructures.	Scraper 0.440in., lateral stabilizers 0.40in., plateau 0.420in., leading edge .440in.	Still freezing from scraper. Support broke, spilling Si, ending run.
August 7	1-101-12	Repeat of Run-11 with insulation under crucible at plateau.	Same component dimensions as previous run.	Freezing to scraper during seeding attempts. Crucible cracked ending run.
August 8	1-101-13	Test enlarged graphite plateau area. Two plateaus back-to-back.	Same as previous runs. No lateral stabilizers.	Grafoil under plateau region. Growth from seed laterally and towards scraper. Freezing out still from scraper.
August 10	1-101-14	Repeat test of double plateau with greater melt depths.	Plateaus 0.40in., leading edge 0.44in., scraper and lateral stabilizers 0.44in.	Moly and 1/8in. graphite felt under double plateau. Some short growth obtained; 3-5cm x 1cm. Arcing of heater connections terminated run.

Appendix A Cont'd.

Date	Run	Purpose	Setup	Results
August 15	1-101-15	Test of stepped height plateau with deeper overall melt.	Scraper 0.520in., lateral stabilizers 0.44in., plateau 0.44in. front, 0.46in. rear.	Freezing out from scraper and rear wall still interferes with growth.
August 17	1-1-1-16	Test of shallow melt in all-graphite set-up.	Scraper 0.17in., plateau 0.11in., leading edge 0.145in. Planned melt depth 0.16-0.17in.	Crucible support broke spilling Si, ending run.
August 20	1-101-17	Repeat of previous run.	Same as run 16.	Silicon wetted across top of crucible wall, attacked Mo support strap ending run.
	1-101-18	Test of graphite components held in quartz crucible and of water-cooled chill tube.	Same as run 16.	Freezing from scraper and side walls. No effect of chill tube.
	1-101-19	Test of graphite crucible with grooved wall to prevent spill-over, and water-cooled gas cold shoe.	Scraper 0.325in., double plateau 0.275in., leading edge 0.30in. No lateral stabilizers.	Freezing from walls and rear. Growth from seed with He cooling, freezing down to plateau.
August 31	1-101-20	Test of cold shoe with graphite crucible/set-up.	Scraper 0.280in., plateau 0.20in., leading edge 0.210in., no lateral stabilizers.	Ribbons grown with He cooling at ∿14cm/min 12 and 19cm/long. Spill-over at front wall ended run.
Sept. 6	1-101-21	Test of graphite set-up with He cold shoe.	Scraper 0.325in., plateau 0.150in., leading edge 0.175in., no lateral stabilizers.	Ribbons grown at 23 and up to 60cm/min 9 and 18cm long.
Sept. 12	1-101-22	Test of cold shoe.	Same as run 21	Ribbons grown at speeds of 20cm/min with He cooling. Lengths of 6, 10, 13cm, widths 7-25mm.

Appendix A Cont'd.

Date	Run	Purpose	Setup	Results
Nov. 27	1-101-23	Test of quartz crucible and cold shoe.	Scraper 0.34in., plateau 0.125in. No lateral stabilizers.	Grew four ribbons >60cm long z 2cm wide at speeds of 15-25cm/min.
Dec. 4	1-101-24	Test of quartz crucible without subsurface components.	Scraper 0.34in. No other components.	Grew ribbons 50 and 73cm long, ∿12 and 20cm/min. Cold shoe pushed Mo shield into melt ending run.
Jan. 25	1-101-25	Test of 4"x6" crucible with substructures.	Scraper 0.34in., plateau 0.125in., lateral stabilizers 0.175in.	Ribbons 20-40cm long grown at speeds 25-30cm/min. Problems of freezing to scraper or from back wall of crucible.
	1-101-26	Repeat of previous run.	All dimensions same, except lateral stabilizers 0.20in.	Very difficult to grow more than 15-20cm due to freezing.

6.2 Appendix B

Economic Projections

The following page is an IPEG calculation of add-on cost for Si sheet production by the LASS process which was presented at the 15th LSA Project Integration Meeting, April 1980.

A more recent analysis[4], March 1982, using cost data from current equipment, indicates an add-on cost of $\$12.38/m^2$. Table 6.2.1 presents the base case results of this analysis. The base case data are given in Table 6.2.2.

LSA PROJECT

LARGE AREA SILICON SHEET

COST PROJECTIONS ($ 1980) SAMICS/IPEG

ASSUMPTIONS: 5 CM WIDTH X 25 CM/ MIN - 10 RIBBONS/MACHINE/OPERATOR - 3 SHIFT/6 DAY WEEK - LABOR RATE: $8/HR - OVERHEAD: 100% - SET UP COSTS: $50,000/YR - UTILITIES: $75,000/YR - DEPRECIATION: $40,000/YR (5 YR. S.L.)

PROJECTION

PRODUCTIVITY: $10 \times 5 \times 25 \times 60 \times 7200 \times .75 \times 10^{-4} =$ 40,500 M^2/YR.

PRODUCTION COST: $280,000 (NO POLY COST)

ADD-ON COST: $6.90/$M^2$

TOTAL SHEET COST: $18.70/$M^2$ ($10/KG;15 MIL)

Table 6.2.1

Price Estimation Results Using Base Case Data

Production Rate

Production Quantity per run (m^2)	349.92
Production Quantity per year (m^2)	34,992.00

Cost Parameters and Coefficients

Parameter	Quantity	Coefficient
Equipment ($)	100,000	$C_1 =$ 0.57
Area (ft^2)	150	$C_2 =$ 109.00
Direct Labor ($)	34,309	$C_3 =$ 2.8
Materials ($)	183,775	$C_4 =$ 1.2
Utilities ($)	36,000	$C_5 = C_4$

Required Annual Revenue

Annual Manufacturing Cost (AMC) ($/year)	433,145

Add-On Price Estimate and Its Breakdown

Parameter	$/$m^2$	%
Equipment	1.629	13.2
Area	.467	3.8
Direct Labor	2.745	22.2
Materials	6.302	50.9
Utilities	1.235	10.0
Total Price	12.378	100.00

Table 6.2.2

Base Case Input Data for the Add-On Price Estimation Using IPEG

Production

Ribbon Width (cm)	10
Growth Rate (cm/min)	40
Run Length (hrs)	60
Number of Ribbon per Furnace	3
Furnaces per Production Unit	1
Furnaces per Operator	2
Process Yield	.9
Duty Cycle	.9
Runs per Year	100

Equipment

Furnace ($ each)	100,000
Equipment Lifetime (yrs)	7

Area

Area for One Furnace Unit (ft^2)	150

Direct Labor

Fringe Benefits Included	No
Labor Pay Rate ($/hr)	7.0
Number of Furnaces Per Operator	2

Table 6.2.2 (cont.)

Materials

Furnace Insulation ($/furnace)	775
Insulation Lifetime (runs)	20
Electrode Parts	1,500
Electrode Parts Lifetime	10
Heating Elements ($/furnace)	100
Heating Elements Lifetime (runs)	10
Crucibles ($ furnace)	400
Crucible Lifetime (runs)	1
Argon ($/100ft^3)	5.4
Argon Flow Rate (ft^3/hr)	150
Helium ($/100ft^3)	10.8
Helium Flow Rate (ft^3/hr)	100
Melt-In Crucible ($/furnace)	60
Melt-In Crucible Lifetime (runs)	1
Holding Fixture	450
Holding Fixture Lifetime (runs)	10

Utilities

Furnace Power Consumption (KwH/furnace)	60
Electric Power Rate	0.10