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MASTER

WEB-DENDRITIC RIBBON GROWTH

USC Solar Report No. Q-3

Quarterly Report, April 1, 1976—June 30, 1976

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June 22, 1976

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South Carolina University
Columbia, South Carolina

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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Quarterly Report for Period 4-1-76 to 6-30-76

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JPL Contract No. <u>954344</u>

Contractor: University of South Carolina
College of Engineering
Columbia, S.C.   29208

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Technical Content Statement

Abstract

This is a report of the third quarter's work on the web-dendritic ribbon growth at the University of South Carolina. A brief description of the work initiated and carried out during this period to meet the program goals is given along with a copy of the Program Plan covering the entire period of the contract.

Man-Hours and Cost Totals

Previous		Current Quarter		Cumulative	
Man-hours	Cost	Man-hours	Cost	Man-hours	Cost
5137	$65,876	3123	$49,885	8260	$115,761

Summary of Results

The web growth investigation portion of this program was spent in growing a backlog of primitive dendrites and developing a number of dendritic seed crystals having a variety of twin plane spacings. These seeds are to be used in the determination of the optimum twin spacing for dendritic-web growth. Having determined the optimum twin spacing for the seed dendrites, the optimized seeds will be used in the experimental part of the investigation to determine the limitation on the growth width and pull rate of the dendritic-

web section.

Primitive dendrites containing 2,3,4 and 5 twin planes with twin plane spacings of 0.6 to 19.2 mm. have been grown. A supply of dendrite seed crystals have been grown from these primitive dendrites.

Computer programs have been developed that appear adequate for the thermal analysis of the dendritic-web growth. A preliminary two dimensional thermal model of the melt, crucible, susceptor and lid has been completed and numerical results obtained. In this model only radiation heat transfer was assumed for the lid. The temperature profiles obtained were thus lower than is found experimentally in the furnace. The model is currently being revised to include conduction and convection losses to the argon gas ambient.

The characterization experiments have all been tested and found adequate for characterization of the material grown in the dendritic-web furnace.

<u>Review by Task Element</u>

T.E. 1.2.1: Pull nominal web based on 1965 experience; establish measure-
          ments baseline

     In the last quarterly report (Q-2), progress in controlling the
center of thermal symmetry, broadening the lateral temperature gradient,
and improving the vertical temperature gradient in the melt was summarized.
The stability of the melt was evidenced by the fact that we have pulled
dendrites thirty feet long before being purposely terminated. Webs have been
grown up to 2 feet before termination. Dendrite pulls are longer because
we are growing materials for seeds. Webs are only grown long enough to get
the necessary observations and/or data.

     Sandblasting of the crucible was studied further and appears to have
eliminated the occasional problem of the melt staying off center. Sand-
blasting is now a part of the crucible preparation procedures.

     The installation of the x-y motion on the r.f. coil, mentioned in
the last report, to go with the already existing z motion was completed and
contributed to the control of the center of thermal symmetry.

     It was also pointed out again that the twin spacing for silicon
dendrite or web growth had never been optimized. Values of the twin spacing
that we were using were given, and the suggestion was made that the spacing
was too narrow. It was deemed in the best interests of the contract to spend
as much of this report period as necessary in growing primitive dendrites,
and from these, pulling long dendrites to be used for seeds for web growth.
The other part of the report period was used to gain information on parameters
involved in the three equations used in the thermal modeling discussed in T.E. 1.3.

     Primitive dendrites were grown by quickly dipping a seed of silicon

into the supercooled melt. Silicon grows rapidly on this "cold" seed, and must be pulled quickly from the melt. The resulting growth mass contains many "growth mistakes" in the form of twins. Only those twin spacings that sustain growth by the twin plane reentrant edge mechanism will result in primitive dendrites protruding from the growth mass. The growth mass with the few protruding primitive dendrites was usually too large to be pulled through the largest opening in our heat shield. A new heat shield for primitive dendritic growth was made as shown in Fig. 1.

The primitive dendrites were fractured from the growth mass and the twin spacing measured. A start has been made on pulling longer dendrites for seeds as web growth and in studying their behavior to find the optimum spacing.

The web-growth facility was out of operation for three weeks during this report period due to the necessity of fabricating and installing a closed distilled water cooling system for the r.f. generator.

T.E. 1.2.2: Vary pull rate; measure effects, etc.

Our initial range of pull rates covered pull rates of dendrites quite well but was at the top of the range desired at present for pulling webs. A smaller drive wheel was installed. The change to the smaller drive gave not only an improvement in the webs but also a better control over the button formation. Both webs and dendrites have been taken to pull-out by increasing the pull rate. No actual data on the effect of pull rate on dendrites or webs is given because the thermal conditions changed from pull to pull.

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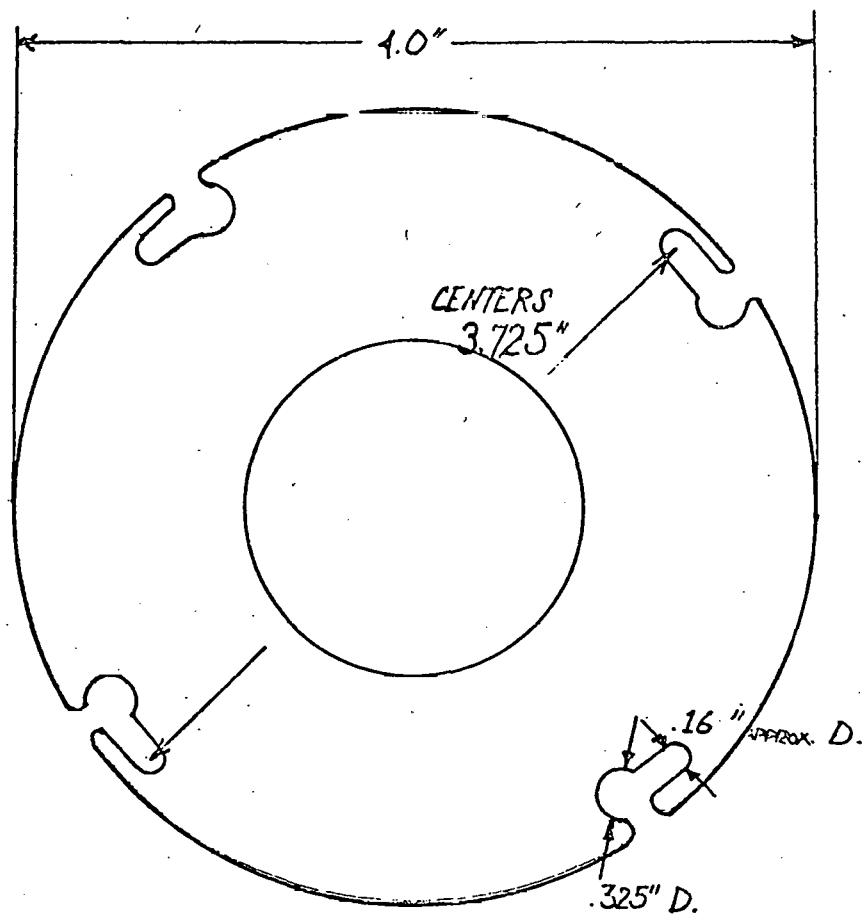
FIGURE 1. Heat Shield for Pulling Primitive Dendrites

T.E. 1.2.3: Vary r.f. heat transfer rate, etc.

As mentioned in 1.2.1, the center of thermal symmetry and the thermal conditions in the melt are such that the growth of two-dendrite webs can be sustained. The parameters now under study or that have been varied are: (a) T, temperature of the melt; (b) V, velocity of pull; (c) dT/dz, temperature gradient into the melt; and (d) H, total irradiation. These are found, either directly or implicitly, in equations 1, 2, and 3 given in the discussion under section T.E. 1.3.

The temperature of the melt, T, and the pull velocity, V, were taken up in previous reports, and the pull rate is discussed in 1.2.2. The temperature of the melt can be changed by adjustment of the r.f. input over a moderate range or by adjusting the height of the susceptor in the r.f. coil. The latter method, which must often be used, also changes the very important temperature gradient, dT/dz,

Our preliminary experiments for determining the contribution of other effects on dT/dz consisted of:

(a) Shape of susceptor. A susceptor was made that has the same diameter at the bottom as at the top, rather than tapered. This increase in diameter at the bottom decreased the dT/dz.

(b) The heat loss by conduction into the pedestal was reduced by putting an alumina spacer between the susceptor and the pedestal. This gave an even more dramatic decrease in dT/dz.

(c) A new heat shield was made from vacuum-deposited Mo which has a very shiny surface and thus a high reflectivity. The higher reflectivity increased H causing the temperature of the melt, T, to be increased and thus decreased dT/dz. Seeding was not possible with this shield even when the T was decreased by lowering the susceptor. Sandblasting was required to bring

back its old value.

(d) H and also T (and thus dT/dz) were varied by changing the opening in the heat shield as reported earlier. After heaters were placed on the heat shield to reduce the radiation losses from the web as shown in Fig.2. It also cuts down some on the radiation loss from the melt.

(e) A new dumbell shaped heat shield was made. The drawing for it is given in Figure 3. This was aimed at changing the radiation loss from the melt and in improving the dT/dz at the center of the melt.

4.0"

1.6"

.68"

.235"

2.26"

.02" MOLY SHEET

1.05" .6"
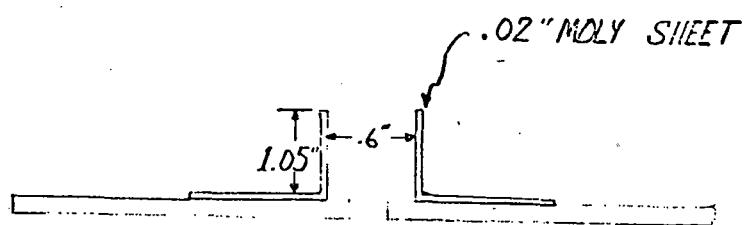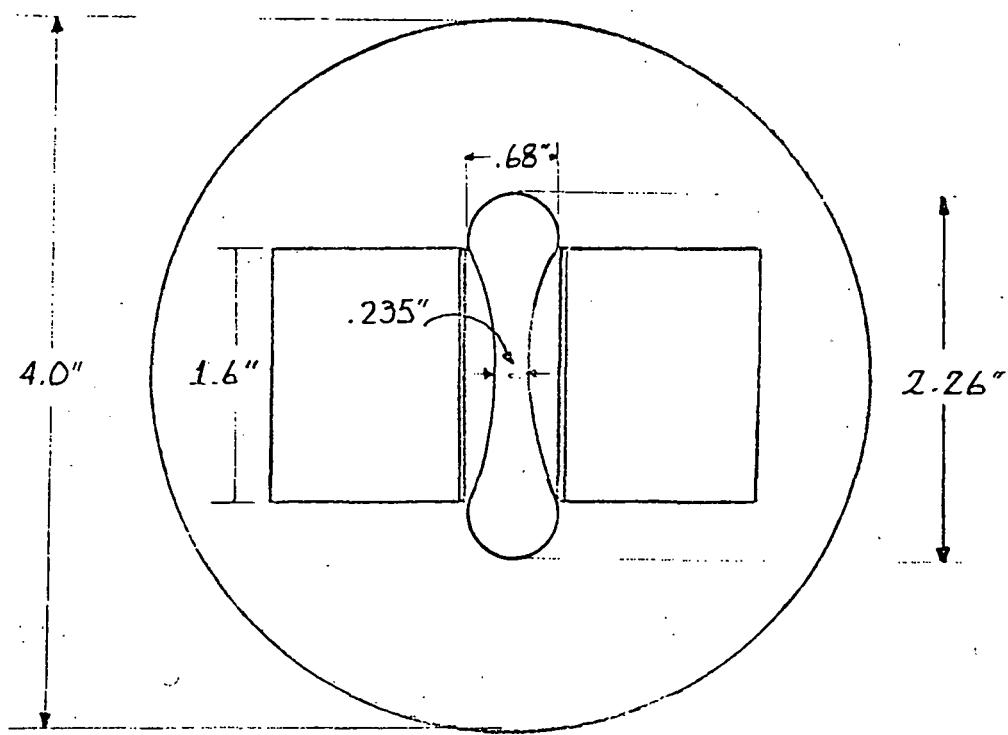
NOT TO SCALE

FIGURE 2.   Afterheaters

Figure 3

9
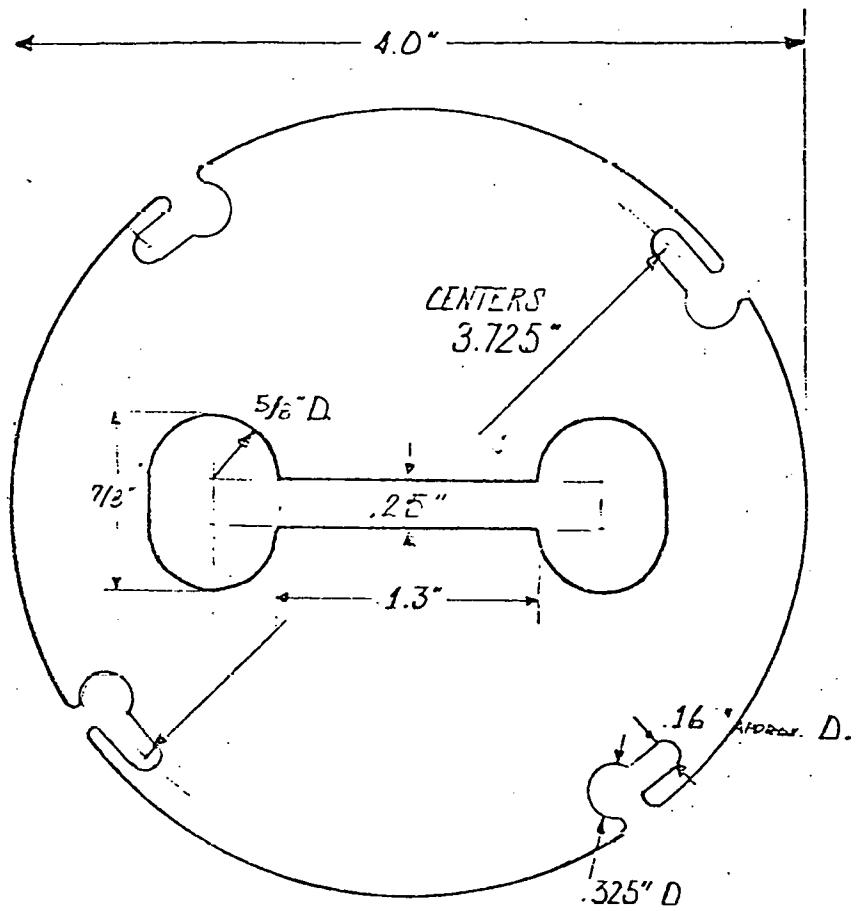
T. E. 1.3: Web-Growth Analysis

a. Web Thermal Analysis

The temperature distribution in the web and meniscus is being investigated with a one-dimensional heat transfer model. Figure 4 shows a cross-section of the web and meniscus. Temperature variations in the z-directions are expected to be large compared to variations across the width and thickness. Heat transfer occurs in the z-direction by thermal conduction and heat convection as a result of pulling the web. Heat exchange between the surface and surroundings is by thermal convection and radiation.

The differential equations for the temperature variation with elevation can be determined by an energy balance on a small elemental volume. The differential equations for the temperature variation in the web and meniscus are respectively,

$$\frac{d^2T}{dz^2} + \frac{\rho_s C_{ps} V}{K_s} \frac{dT}{dz} - \frac{hP}{K_s A_s} (T-T_a) - \frac{(W-\alpha H)}{K_s A_s} = 0 \qquad (1)$$

and

$$\frac{d^2T}{dz^2} + \frac{1}{A_m} \frac{dA_m}{dz} + \frac{\rho_s C_{pm} A_s V}{K_m A_m} \frac{dT}{dz} - \frac{hP}{K_m A_m} (T-T_a)$$

$$- \frac{(W-\alpha H)}{K_m A_M} = 0 \qquad (2)$$

where

A - cross-sectional area

c_p - specific heat

H - total irradiance of surface

h - convertive heat transfer coefficient

K - thermal conductivity

P - perimeter

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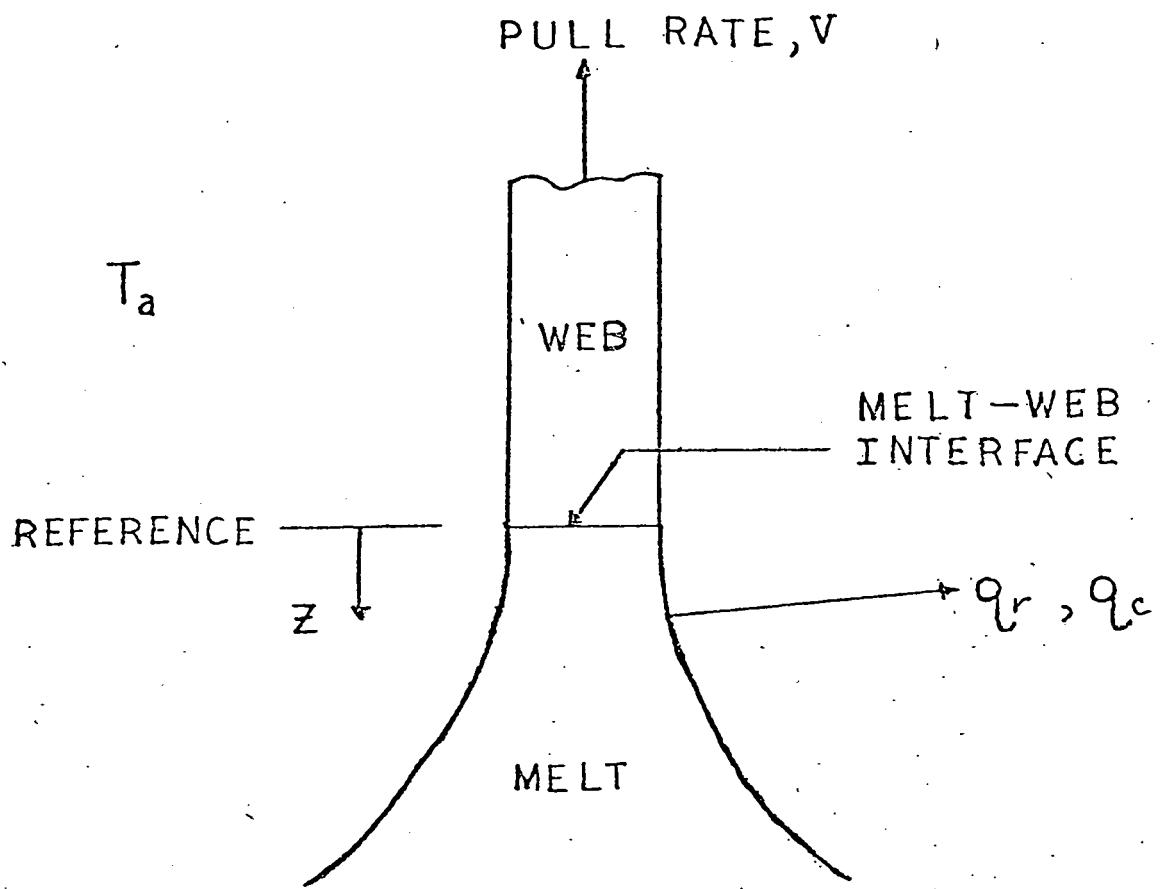
Figure 4. Cross-Section of Web and Meniscus at the Liquid-Solid
Interface

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T — temperature of web or meniscus

T_a — temperature of atmosphere

V — web pull velocity

W — emissive power of surface

z — distance from liquid -solid interface

α — thermal radiation absorptance of surface

ρ — density

Subscripts s and m indicate solid and melt, respectively.

A thermal balance at the liquid-solid interface yields the following interface condition,

$$-K_s A_s \frac{dT}{dz} \Big|_s + K_m A_m \frac{dT}{dz} \Big|_m + \rho_s A_s VL = 0 \tag{3}$$

where L is the latent heat of fusion of the silicon and $\frac{dT}{dz} \Big|_s$ and $\frac{dT}{dz} \Big|_m$ are respectively the temperature gradients in the web and melt at the interface. It is assumed in Equation (3) that the temperature of the solid and liquid are equal at the interface, that is, interface subcooling is negligible.

Because of the complexity of the governing differential equations given by Equation (1) through (3) an analytical solution is not possible. Therefore, numerical solutions will be obtained using the computer. The CSMP3 computer program is currently being investigated as a means for obtaining numerical solutions. Preliminary results have been encouraging and it is expected that numerical data will be forthcoming.

The greatest difficulty in obtaining accurate temperature calculations is predicting the irradiance, H, of the web and meniscus. The term is contained in Equation (1) and (3) and is the thermal radiation received primarily from the lid and silicon melt. The value of H is a function of z since the view factor from the lid and silicon melt varies with position. Initial calculations will be performed with very approximate estimates of H but we will continue to study this problem in order to improve the calculations.

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Thermal Model of the Melt, Crucible, Susceptor, and Lid

Figure 5 shows the nodal geometry for the thermal model presently being used to represent heat transfer in the melt, crucible, susceptor, and lid. Due to the cylindrical geometry of the system, the length of the nodes increases with radial distance from the central axis.

As a result of the thermal effect of the slot in the lid and the web, an azimuthal temperature variation in temperature exists which causes a three-dimensional temperature variation. The three dimensional nodal geometry is obtained by sectioning at increments of azimuthal angle. However, it is seen in Figure 6 that the symetry exists about diametrical lines along the slot and perpendicular to the slot. Consequently, thermal modeling of the 90° section is sufficient to determine the temperature field. The 90° section will be incremented into four "pie" sections of 22.5° increments.

Induction heating by the RF field is present primarily in the outer periphery of the susceptor. The nodes in this region have a thickness equal to the skin depth of approximately 0.059 cm and are seen in Figure 5. Heat generation also occurs in the outer periphery of the lid and is also included in the model. Internal heat generation is assumed uniform up to the skin depth and zero elsewhere.

Heat transfer through the quartz crucible is due to solid conduction and thermal radiation transmitted through the quartz. The quartz is transparent to a large fraction of the radiant energy emitted by the silicon and molybdenum surfaces.

The lid is heated by radiation heat transfer from the silicon melt and the crucible. The lid in turn radiates heat to the surroundings from the upper surface. Conduction and convection heat transfer to the

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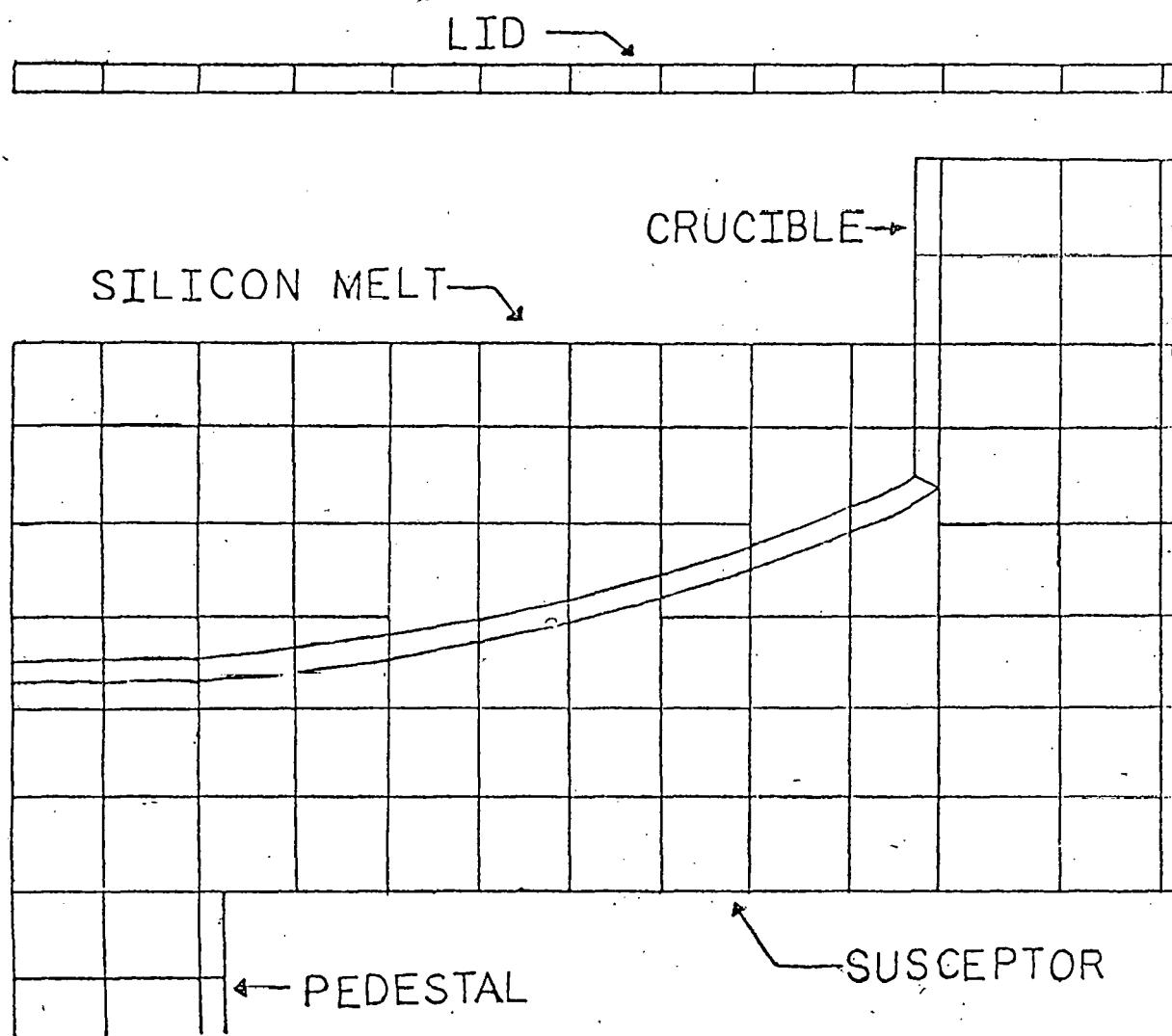
Figure 5.   Thermal Model Modal Geometry Melt Crucible, Susceptor, and Lid
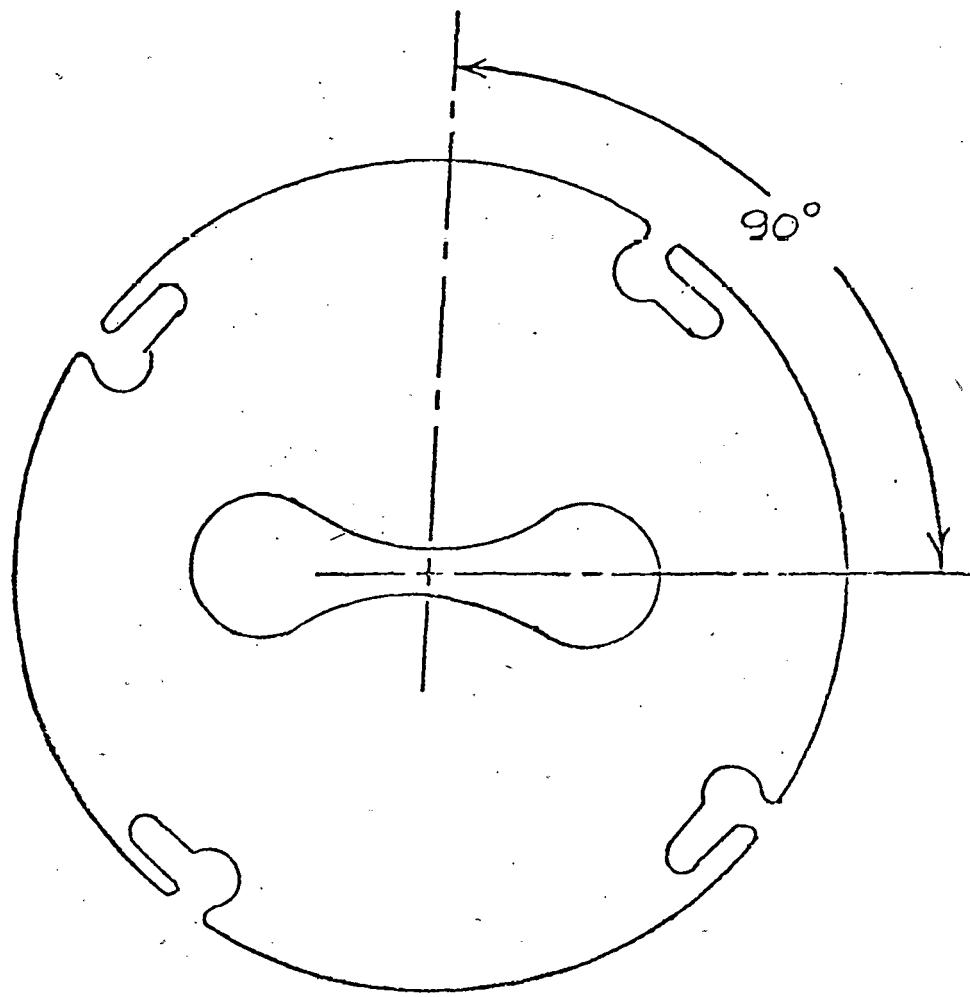
Figure 6. Temperature Field of Each 90° Sector is Identical
Due to Symmetry.

argon atmosphere also effects the lid temperature.

The LION-4 computer program is being used to calculate the temperature field. The internal heat generation rate is specified and from that the temperatures are calculated.

A preliminary two dimensional model consisting of a single section has been completed and numerical results have been obtained. Only radiation heat transfer was assumed for the lid. As a result, the temperatures are considerably lower than experimental results obtained in the furnace. The model is currently being revised to include conduction and convection in the argon. It appears that the two-dimensional model will be extremely useful in investigating the thermal characteristics of the system.

Although the azimuthal temperature variations are not determined by the model, many other characteristics can be studied with it. Computer run time is significantly less for the two-dimensional model, therefore, it is hoped that much of the thermal analysis will be performed with this model rather than the three-dimensional model which presently remains to be completed.

T.E. 2.2: Structural Characterization

This period was spent in gathering data on:

(a) dimensions of dendritic web growth;

(b) twin spacing in grown dendritic web; .

(c) number of twins in primitive dendrites;

(d) twin spacing in primitive dendrites. .

Representative date of the above is given in the section of this report on Summary of Characterization Data.

T.E. 2.3: Electrical characterization; web bulk silicon

Data was collected during this report period on the resistivity and charge carrier type of the dendritic web samples grown. In all cases the samples were p-type ranging in resistivity from 2 to over 100 ohm-cms.

Representative data of the above is given under the section on Summary of Characterization Data.

Work also has been carried out to develop a technique for applying test patterns on narrow web (less than 5 mm) in order to electrically characterize these materials. Conventional techniques used for wafers have been found inappropriate due to geometric difficulties with the small size samples. Two techniques for applying appropriate metallization patterns on the web are being pursued. The first involves the development of a system for projecting the test pattern images directly onto the web surface without direct contact of the test pattern mask onto the surface of the web. The second involves the development of a flexible acetate mask that can be applied directly to the web surface by means of an adhesive layer. Neither of these techniques has been developed to the extent that they can be tested as yet.

T.E. 2.4: Electrical characterization; web solar cells

       With the exception of setting up a standard solar cell facility, work in this area was held in abeyance pending the completion of the program to grow seed dendrites for the determination of the optimum twin spacing for web growth.

Interpretation of Results and Application to Program Goals

The primitive dendrites containing the appropriate range of twin spacing, 0.1 to 9 μm, have been grown. Seed dendrites for web growth have been produced from the majority of these primitives. Upon completion of the growth of the seed dendrites, this will complete the second phase of our growth program - the first phase consisting of setting up and putting in operation the growth facility having been completed in the first quarter of the contract. Having obtained the appropriate seed crystals, efforts will now be centered on investigating their suitability for growing web; and once having determined the optimum twin spacing for web growth, work will then be devoted to the determination of the maximum width and pull rate that can be obtained for the growth of web crystal from the dendritic seeds with the optimum twin spacing.

The computer programs for the analysis of web growth have been developed to the stage that feedback between the analytical results and experimental configurations is now possible, enabling a more systematic and quantitative approach to optimizing the thermal conditions for web growth.

Preliminary characterization data taken indicate that these facilities will be adequate in determining the quality of the web produced in our growth facility.

Tentative Conclusions and Recommendations

The experimental facilities and thermal analysis computer programs have been developed and are adequate for meeting the goals of this program. It is recommended that the program proceed as planned as it is felt that

the goals of the program will be met with this plan as scheduled.

Engineering Drawings and Sketches

The drawings and sketches generated during this report period are incorporated in the text of this report as figures 1 through 6.

Projection of Activities for Succeeding Three Months

The projected activities for the next three months are as listed below:

(a) Completion of growth of seed dendrites from primitive dendrites;
(b) Initiation of studies to determine optimum twin spacing in seed dendrites for web growth;
(c) Refinement and continued development of computer programs for the web-growth analysis;
(d) Thermal probing of the silicon melt and incorporation of the data into the thermal analysis of the web growth.

Summary of Characterization Data

Table I gives the number of twin planes and twin plane spacings for representative primitive dendrites grown during this report period.

Table II gives representative values of resistivity and resistivity type found for web samples grown during this report period.

TABLE I

Table of Twin Spacings from Primitive Dendrite Pulls

No. of Twin Planes	Twin Plane Spacings (μm)			
	1	2	3	4
5	0.4	0.4	0.1	1.9
4	1.6	2.4	1.0	
	8.0	2.0	10.0	
	19.2	1.0	8.8	
3	2.0	1.0		
	4.0	1.0		
	4.4	2.0		
	8.0	2.0		
	1.9	0.8		
	8.8	1.0		
	0.8	0.6		
	8.5	0.3		
2	14.0			
	12.0			
	9.4			
	7.0			
	6.4			

Table II

Data on Resistivity

Sample	Type	Resistivity (ohm-cm)
2-23-76-A-2	P	2.33
3-19-76-A-2	P	3.55
4-7-76-E-5	P	5.10
4-7-76-F-16	P	6.86
3-19-76-A-1	P	7.65
3-8-76-A-1	P	8.13
3-8-76-A-3	P	10.03
4-7-76-E-1	P	11.48
4-7-76-F-1	P	12.70
2-23-76-A-1	P	13.86
3-19-76-A-9	P	15.70
3-19-76-A-10	P	16.50
4-7-76-A-2	P	18.40
2-23-76-B-2	P	19.70
4-2-76-B-1	P	20.90
4-2-76-A-1	P	22.18
4-7-76-E-3.5	P	34.34

PROGRAM PLAN

WEB-DENDRITIC RIBBON GROWTH

UNIVERSITY OF SOUTH CAROLINA

LEGEND: H - HILBORN
             F - FAUST
           R - RHODES
             A - RESEARCH ASSOCIATE
             G - GRADUATE ASSISTANT
             S - SECRETARY

(Figures in columns represent man hours committed)

CONTRACT NO. 954344

PERIOD: 1 OCTOBER 1975 - 1 MAY 1977
UPDATED 26 May 1976

1. Web Growth, Growth Analysis

  1.1 Set up web furnace

    1.1.1 Level, align, calibrate gas flow, measure physical/mechanical parameters

    1.1.2 Instrument melt temperature sensors, run furnace and RF generator

    1.1.3 Calibrate RF generator controls to melt temperature, no web pulling

  1.2 Web growth, manual batch

    1.2.1 Pull nominal web based on 1965 experience, establish measurements baseline. (Instrument melt level measurement), correlate with crystallization kinetics and seeding requirements

    1.2.2 Vary pull rate, measure effects, correlate with crystallization kinetics and seeding requirements

    1.2.3 Vary RF heat transfer rate, measure effects, correlate with crystallization kinetics and seeding requirements

    1.2.4 Couple RF heat transfer rate and pull rate variations, measure effects, correlate with crystallization kinetics and seeding requirements

    1.2.5 Final experimental design

    1.2.6 Final manual variation of manipulated variables, correlation with crystallization kinetics and seeding requirements

Schedule chart (months OCT. through APR. spanning two years):

Task	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE	JULY	AUG.	SEPT	OCT	NOV.	DEC.	JAN.	FEB.	MAR.	APR
1.1.1	F35 A87 G44																		
1.1.2	F31 H2 A86 G43	F30 H2 A86 G44																	
1.1.3		F32 H2 A86 G43	F30 H2 A87 G44																
1.2.1			F27 H2 A86 G43	F60 H2 A173 G87	F53 H2 A173 G87														
1.2.2					F56 F2 A173 G87	F35 H2 A173 G87													
1.2.3							F56 H2 A173 G174	F56 H4 A173 G174											
1.2.4									F56 H4 A173 G173	F48 H4 A87 G44									
1.2.5										F8 H4 A86 G43	F10 H2 A87 G44								
1.2.6												F14 A86 G43	F25 H2 A173 G87	F50 H4 A173 G87	F50 H4 A173 G86	F50 H2 A173 G87	F50 H4 A173 G87	F50 H4 A173 G87	

1.3 Web growth analysis

    1.3.1 Develop semi-empirical multivariate static model from web growth manual experiments

    1.3.2 Thermal web growth analysis

    1.3.3 Correlate analysis, experimental data, and semi-empirical model

2. Web Characterization

    2.1 Development of sampling strategy, prototype technique

    2.2 Structural characterization

    2.3 Electrical characterization, web bulk silicon

    2.4 Electrical characterization, web solar cells

3. Documentation, Program Review

    3.1 Documentation

        3.1.1 Initial financial management report and program plan

        3.1.2 NASA Form 533M MAR 73, JPL 3645, JPL 3645-1

Note near SEP–DEC (second year): "Choice of Emphasis 1.3.1 or 1.3.2"

Task	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUNE	JULY	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR
1.3.1	F4 / F4 / E33	F4 / H2 / R32	F4 / R20	F4 / R20	F4 / R16	F4 / R16	F2 / R16	F4 / R20	F16 / H4 / R20	F16 / H4 / R20	F16 / H4 / R20	F2 / R15	F4 / R31 / G87	F4 / R30 / G87	F4 / R31 / G86	F4 / R30 / G87	F4 / R31 / G86	F4 / R30 / G86	
1.3.2		H2 / R13	H2 / R20 / G87	R17 / G87	R16 / G86	R17 / G87	R16 / G87	H4 / R48 / G36	H4 / R47 / G87	H4 / R48 / G87	R15 / G86								
1.3.3				F4 / H2 / R8	F4 / H2 / R8	F2 / H2 / R8	F4 / H2 / R8	F40 / H3 / R17	F40 / H8 / R17	F40 / H8 / R17	F2 / R8	F2 / H2 / R8	F10 / H2 / R8	F7 / H2 / R8	F10 / H2 / R8	F7 / H2 / R8	F10 / H2 / R8		
2.1	H17 / F8	H16 / F8																	
2.2	F3 / G173	F10 / G173	F20 / G174	F20 / G173	F20 / G173	F20 / G174	F12 / G173	F20 / G173	F51 / G174	F54 / G173	F51 / G173	F20 / G174	F20 / G173	F20 / G173	F20 / G174	F20 / G173	F20 / G174	F20 / G174	
2.3	H3 / G130	H13 / G130	H16 / G130	H18 / G130	H21 / G130	H20 / G130	H29 / G130	H24 / G130	H52 / G130	H51 / G130	H52 / G130	H4 / G130	H29 / G130	H23 / G130	H16 / G130	H18 / G130	H17 / G130	H14 / G130	
2.4	H3 / G130	H13 / G130	H15 / G130	H18 / G130	H20 / G130	H18 / G130	H28 / G130	H22 / G130	H51 / G130	H51 / G130	H51 / G130	H4 / G130	H28 / G130	H21 / G130	H15 / G130	H18 / G130	H18 / G130	H14 / G130	
3.1.1	H26																		
3.1.2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2	H2

3.1.3 Monthly technical progress report

3.1.4 Quarterly report

3.1.5 Interim summary

3.1.6 Annual report

3.1.7 Draft final report

3.1.8 Approved final report

3.2 Program review, work sessions

3.2.1 USC in-house review

3.2.2 JPL program review

3.2.3 Task integration sessions

3.2.4 Annual workshop

Task	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE	JULY	AUG.	SEPT	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.
3.1.3 Monthly technical progress report		H10	H8		H8	H8		H8	H8		H8	H8		H8	H8		H8	H8	
3.1.4 Quarterly report		H8	H16		H8	H16		H8	H16					H8	H16				
3.1.5 Interim summary																			
3.1.6 Annual report											H12	H24							
3.1.7 Draft final report																	H12	H24	
3.1.8 Approved final report																			H69 F87 R42
3.2.1 USC in-house review		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3		H3 F3 R3	
3.2.2 JPL program review	H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		H8 F6 R2		
3.2.3 Task integration sessions				H30			F30			H30			F30			H30			
3.2.4 Annual workshop												H36 F36							

3.2.5 Design and performance review

4. General Administration

5. Planned Cost   (Thousands of dollars)

    Incurred Cost (Thousands of dollars)

OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE	JULY	AUG.	SEPT	OCT.	NOV.	DEC.	JAN.	FEB	MAR.	APR.
H22	H16	H16	H12	H16	H16	H16	H16	H16	H16	H16	H4	H16	H16	H16	H12	H16	H16	H16
S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87	S87
12.5	16.8	14.2	14.3	12.7	13.0	13.3	12.6	17.8	19.1	17.2	15.5	13.5	12.8	13.6	13.4	12.8	13.2	7.1