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WEB-DENDRITIC RIBBON GROWTH

Quarterly Report, January 1, 1977—March 31, 1977

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MASTER

April 26, 1977

Work Performed Under Contract No. NAS-7-100-954344

College of Engineering
South Columbia University
Columbia, South Carolina

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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Web-Dendritic Ribbon Growth

USC Solar Report No. Q-5

Quarterly Report for Period 1-1-77 to 3-31-77

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TABLE OF CONTENTS

<u>SUBJECT</u>	<u>PAGE</u>
Technical Content Statement	1
Abstract	1
Man-hours and Cost Totals	1
Summary of Results	1
Review by Tasks	3
Interpretation of Results	24
Tentative Conclusions	25
Projection of Next Quarter's Activity	25
Program Plan	Attachment

Technical Content Statement

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Abstract

This is a report of the sixth quarter's work on the web-dendritic ribbon growth process at the University of South Carolina. The goal of the program is to develop a multivariate semi-empirical computer model for use in understanding the web-dendritic process and defining the basic limitations of the process with particular regard to the growth rate and width. A brief description of the work carried-out and the results achieved during the past quarter is given in this report.

Man-Hours and Cost Totals

<u>Previous</u>		<u>Current Quarter</u>		<u>Cumulative</u>	
Man-hours	Cost	Man-hours	Cost	Man-hours	Cost
16,604	\$221,083	2575	\$40,824	19,179	\$261,907

Summary of Results

During this past quarter successful reproducible growth of two-dendrite web ribbon was achieved. To date, ribbons have been grown up to 108 cms in length and 1.2 cms in width. The lengths and widths were

limited to these values because of either premature pull-out of the web or the initiation of third dendrites due to a variety of mechanical and/or thermal control problems.

An investigation of the effect of twin lamella spacing in the seed crystal for two different twin spacings was carried out with the result that use of a seed with spacings of 2μ and 8μ limited the pull rate of the web ribbon to less than 4 cm/min. With seeds of this twin spacing attempts to pull at higher rates caused the edge dendrites to lose one of the twin planes which resulted in dendrite branching occurring in the edge dendrites. Seeds with twin lamella spacings of $.9\mu$ and 2μ have shown no upper limit on the pull rate up to values as high as 10 cm/min.

Work continued on the model development during this period to include the effect of the position of the top heat shield and the geometry of the slot in the shield on the temperature distribution in the melt. Results of this model can now be used to design heat shields and slot geometries to achieve desired temperature distributions in and around the melt surface.

Model studies of the effect of the spacing between the top heat shield and the melt surface on the web pull rate were made. The results indicate a significant reduction in pull rate will occur as the spacing between the melt surface and heat shield increases. An increase in the spacing from 0.8 cms to 1.2 cms indicates a decrease of 40% in the achievable pull rate. Further increases in the spacing have less effect on the pull rate.

Review by Tasks

T.E. 1.2: Web Growth

The work effort in this area during this report period has been concentrated in four (4) areas: (1) selection of the most promising furnace configuration from data previously acquired, (2) optimizing seeding and button growth with the furnace configuration of (1), (3) maximizing the widening rate during ribbon growth, and (4) achieving maximum pull length.

Furnace Configuration

From temperature profile data and from observations of button formation, dendrite and web ribbon growth, the most promising furnace configuration was chosen for completing our studies of dendritic-web-ribbon growth. This configuration consists of a 4 inch diameter tapered molybdenum susceptor with a 1/4 inch lip at the top (See Figure 1) setting upon a hollow, 1 3/4 inch O.D., alumina tube pedestal. A slotted top heat shield of 0.06 inch molybdenum is held 1/8 inch above the top of the susceptor by four symmetrically placed tantalum pins. This entire assembly is contained within a 130 mm diameter, O.D., quartz tube. The r.f. coil is 6 inches, I.D., and consists of 11 turns of 1/4", O.D., copper tubing placed as close to each other as practical. The length of the coil is 3.75 inches. The coil surrounds the outside of the quartz tube and can be moved vertically up and down to all positions from above the susceptor to below the susceptor. The approximately 1 cm clearance between the walls of the quartz tube and the r.f. coil allowed some lateral adjustment of the coil to establish the correct thermal symmetry in the susceptor.

The inert argon gas is injected into the quartz tube through ports in the bottom plate and exits through the ribbon pull slot in the top plate.

A tungsten ground wire is used to ground the susceptor which is otherwise susceptible to corona discharge during operation.

Figure 1. Tapered susceptor with radiation ports

Although studies in our system indicated little effect on button formation and ribbon growth of the size of the silicon charge in the crucible, a 150 gram charge was adopted as the standard in order to have a full crucible at the start of each run.

Button Formation

Our studies on reproducibility in seeding and button formation showed the following. It was found seeds having the 8 - 2 μ m twin lamellae spacing had a maximum pull rate usability in our furnace of 4 cm/min. Above this pull rate, the 8 μ m lamella appear to lose its effect causing branching in the ribbon growth typical of the two twin structure that remained when the 8 μ m lamella is lost. Studies with seeds having a 2 - .9 μ m twin lamellae structure failed to reveal this problem at pull rates up to 12 cm/min. All seeds were etched to a fine point prior to dipping in the melt to insure an oxide free surface of uniform cross section for each dip. Reproducible button growth was found to be dependent on the vertical temperature gradients at the surface of the melt; as such, the relative position of the coil and susceptor had to be determined to optimize button growth. For our furnace the best position was found to be with the top of the coil located a distance of 0.4 cms. above the top of the susceptor. With this configuration the only remaining parameter to be determined to optimize the incidence of good button formation was the degree of supercooling to impose after the seed was inserted into the melt. The best results were obtained with the following procedure. The temperature of the melt is raised by 1°C to insure that the seed is "wetted" by the melt when inserted. The seed crystal is inserted into the melt, and the set point of the controller is then set 8 - 10°C below the set point corresponding to the melting temperature. When adequate button growth has been achieved, pulling of the

ribbon is commenced with a simultaneous change in the set point of the controller to 4 - 5°C below the set point for melting. With this configuration and procedure, the incidence for the growth of buttons suitable for web-dendritic growth is nearly 100%.

Widening Rate

The experiments conducted to increase the widening rate during the initial phase of the ribbon growth were done at pull rates between 2 and 4 cms/min.

It was desired to increase the widening rate from the 0.5 cm/m, achievable initially, to a value as large as possible in order to achieve the maximum width of the ribbon in as short a time as possible after the initiation of dendritic-web growth. The major difficulty in attaining high widening rates is the increased probability of introducing 3rd dendrites into the web prior to achieving the desired width of the ribbon. A high widening rate requires a large negative temperature gradient in the direction of widening. The negative temperature gradient is achieved by tailoring the thermal geometry near the surface of the melt such that the temperature decreases from some maximum value beneath the center of the button to minima beyond the two ends of the button separated by a distance that will determine the maximum steady state growth width of the ribbon. In order to prevent the introduction of "3rds", these minima must be above some limiting value and the temperature must decrease monotonically from the maximum to these minima. At the same time the maximum of the temperature under the center of the button must be maintained at some, as yet undetermined, temperature, estimated to be approximately 2 - 5°C below the melting point, depending on the pull rate being used, in order to maintain web growth and prevent "pull-out" or "freeze-out" of the web ribbon. The temperature difference between the maximum and the minima, ΔT_g , is estimated

to be of the order of 4 - 5°C, and is probably independent of the steady state width of the ribbon being pulled. Now it is the negative temperature gradient, $\Delta T_g / \Delta W$, where ΔW is half the steady state ribbon width, that determines the widening rate. The higher the value of $\Delta T_g / \Delta W$, the higher the widening rate. This being the case, and as ΔT_g is a relative constant quantity, then the maximum widening rate that can be achieved is inversely proportional to the final width of the ribbon that is desired.

In view of the preceeding, the effort expended during the Quarter in increasing the widening rates for the ribbon growth centered on experimentally determining the effects of: slot geometry in the top heat shield, after heaters, radiation port geometry in the susceptor and combinations of these, on the location of the temperature minima and on the magnitude of the negative temperature gradient, $\Delta T_g / \Delta W$. During these experiments it was found that we had insufficient fine temperature control to enable the operator to reproducibly set melt temperatures from run to run, and during the same run, with sufficient accuracy to carry out the experiments. A fine tuning, 10 turn, potentiometer was installed on the controller which gave the operator sufficient reproducible control. (This fine tuning pot. also helped in obtaining the improved incidence rate of good button growth.)

The after-heaters, Figure 2, consisted of Mo plates parallel to and extending above the slot in the top heat-shield. The heat shields used are shown in Figures 3-9. The susceptors used were as shown in Figure 1. The radiation ports were of 1/4" diameter in one susceptor. 3/8" diameter in a second susceptor, and 3/8" wide by 1/2 inch long in a third susceptor. The best widening rates have been obtained using either the 3/8" ported susceptor with the medium dog-bone slotted heat shield susceptor (Figure 5) or the larger dog-bone slotted heat shield (Figure 6) and the modified after heaters. In each case the level of the melt was 4.8 mm below the heat shield. It is estimated

AFTER SHIELDS

Figure 2. After heaters

Figure 3. Narrow dog-bone slotted heat shield

.06" MOLY.

Figure 4. Slotted heat shield

Figure 5. Dog-bone slotted heat shield

Figure 6. Wide dog-bone slotted heat shield

Figure 7. Dumbell slotted heat shield

Figure 8. Bow-tie slotted heat shield

Figure 9. Bow-tie slotted heat shield

that the maximum width attainable with this configuration is 1.4 inches. Widening rates at the start of these studies were usually below .5 cm/meter. We are now getting between .7 and 1.3 cm/m for our widening rates at pull speeds of 4 cm per min. Thick webs, above 15 mils thick tend to give a higher widening rate, about 1.8 cm/meter. Pull rate also affects widening rates. Faster pulling decreases the widening rate.

Pull length

Premature pull-out of the ribbon from the melt appears to become increasingly more of a problem as the width of the ribbons are increased. Control of the growth parameters becomes more critical and operator error combined with mechanical and thermal fluctuations associated with the pulling apparatus make it increasingly difficult to sustain the proper growth conditions. Several modifications were incorporated into the puller that helped considerably in preventing premature pull-out of the web. The first of these has already been mentioned, namely, the addition of the 10-turn fine control potentiometer on the temperature controller. This enabled the operator to make much finer adjustments to the melt temperature during the run, decreasing the likelihood of introducing too great a change in temperature as adjustments are made to control the ribbon geometry during a growth run. An additional change which was of considerable help was the incorporation of an 8 inch drive wheel with a new quieter motor and a new gear box on the ribbon take-up reel. This gave greater control on the pull rates over the range of pull speeds found most appropriate for pulling the ribbons, i.e. 1-5 cms/min. The pull-speeds attainable with the 3 inch drive wheel, the 3 inch wheel with a new motor, and the 8 inch wheel with new motor and gear box are shown in Figure 10. Some difficulty was encountered due to mechanical vibration introduced when the seed holder and take-up ribbon were wound up onto the take-up reel. This was improved by fabrication of a slimmer seed holder and the addition of mechanical guides to feed the steel

pull ribbon into the slot on the take-up reel with a minimum of mechanical vibration.

Experiments were conducted to determine the effect of the melt height on growth of web ribbons and little if any difference could be observed for charges ranging from 100 to 150 gms. In fact, the seeding, widening rate and pulling characteristics of the configuration now being used appear to be nearly independent of melt height, in that operator adjustments can be made to coil position, pull speeds and temperature control to compensate for whatever thermal geometry changes occur as a result of different melt heights.

The longest two-dendrite ribbon pulled during the quarter was 130 cms. It appears that further work is needed in either improving operator proficiency or mechanical and thermal control in order to eliminate premature pull-out of the web.

T E. 1.3 Web Growth Analysis

a. Effect of Slot Geometry on Melt Temperature Distribution

Radiation heat losses through the slot in the thermal shield influences the temperature distribution of the melt in the region of web growth. Experimental studies have shown that growth characteristics are altered by modifications in the slot geometry. Experimental studies have been performed for a number of slot geometries and it has been shown that the slot geometry has an appreciable effect on the growth characteristics. This influence is probably due to a modification of the temperature distribution in the melt.

To assist in understanding the role of the slot geometry on the web growth, a thermal model of the melt in the region of the slot has been developed to calculate the temperature distribution. The LION-4 thermal modeling computer program is used to develop the thermal model. The heat transfer is modelled using the LION-4 thermal modeling computer program. The model contains 80 melt nodes and 20 thermal shield nodes. Because of thermal symmetry of the melt, a 90° sector of the melt was modeled. Three-dimensional heat transfer occurs in the melt and heat transfer between the thermal shield and the melt surface is by radiation.

Figures 11 and 12 show numerical results calculated for a rectangular and dogbone slot respectively. These figures show the isotherms at the surface of the melt in the region of the slot which is shown in a dashed line. The right edge of the figure is the centerline of the slot and, therefore, the isotherms are shown only for the left side. In figure 12 rectangular shaped openings were assumed at the ends of the slot in the model rather than the circular openings of the experimental model, for modeling convenience. The temperature distribution in the melt is not expected to be significantly different for these two geometries.

ISOTHERMS AT MELT SURFACE

Figure 11. Isotherms at surface of melt for 0.955 cm x 6.48 cm rectangular slot

ISOTHERMS AT MELT SURFACE

Figure 12. Isotherms at surface of melt for dog-bone slot 6.48 cm long

By comparing Figures 11 and 12 it is seen that the surface temperature distributions are similar. However, it can be seen that the temperature gradient along the slot is slightly lower for the dogbone and the isotherms are somewhat more rounded.

b. Effect of Spacing between Thermal Shield and Melt Surface on Growth Rate

Surfaces of the web below the thermal shield receive considerably higher radiation heat flux than that above the thermal shield. Web surface below the thermal shield receives thermal radiation from the surface of the melt and the lower surface of the thermal shield. The web surface above the thermal shield receives radiation primarily from the upper surface of the thermal shield since the remainder of the furnace surroundings are at considerably lower temperature. The irradiation of the web surface is approximately 38.4 watts/cm^2 below the thermal shield compared to approximately 6.4 watts/cm^2 immediately above the shield.

Since the pull rate is determined by the rate of heat transfer of the heat of fusion from the liquid-solid interface, the pull rate will increase as the irradiation of the web surface is decreased. Thus, the pull rate is increased by decreasing the spacing between the thermal shield and melt as shown in Figure 13. Pull rates are shown for web thickness of 0.10, 0.15, and 0.20 mm and a thermal shield temperature of 1300°C . A meniscus joining angle of 10° was assumed. As expected, the pull rate increases with decreasing spacing. At the lower values of the spacing, the pull rate is seen to be more sensitive to spacing changes than at higher values. During the pulling operation, it will be necessary to reduce the pull rate as the melt level is lowered in the crucible if the crucible is not continuously replenished with silicon.

Figure 13. Pull rate as a function of spacing between thermal shield and melt surface

T. E. 2.0. Characterization

No further characterization work was conducted in this quarter due to a lack of samples and a change in program direction.

Interpretation of Results and Applications to Program Goals

a. Web-Growth

The results clearly demonstrate that two-dendrite web ribbon can be grown in our facility with the modifications described in the text of this report. It appears uncertain at this time as to just how wide ribbon can be grown in this facility. The premature pull-out is also indicative that additional "tuning" of the thermal and mechanical system is needed before very long ribbons of maximum width can be grown. However, the achievement of reproducible web ribbon growth has been a major accomplishment towards the realization of the program goals.

b. Web Growth Analysis

The inclusion of the slot geometry of the top heat shield in the thermal model has given greater insight into the design of the appropriate top heat shield to accomplish the desired thermal geometry in the melt. It is clearly a further step in completing the thermal model of our growth facility.

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 13.

Tentative Conclusions and Recommendations

The results of this past quarter's work indicates that the growth facility

has been "tuned" sufficiently to achieve, reproducibly, two-dendrite web ribbon growth. However, further refinement of the thermal and mechanical parameters associated with the furnace is still necessary before maximized width and length ribbons can be routinely grown.

The analytical work is getting to the stage of completing the thermal model of the growth facility.

It would thus appear that the growth development and the modeling development are both at the point where they can, for the first time during this contract, be of mutual benefit to each other in their further development

Projection of Activities for Succeeding Three Months

For the next three months extension of the original contract the following program will be followed:

1. Investigate the thermal stability in and around the melt.
2. Investigate the mechanical stability of the system.
3. Demonstrate limits of pull length with system.
4. Complete the modeling and use to predict the limits on the growth parameters for our facility.

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PROGRAM PLAN

WEB-DENDRITIC RIBBON GROWTH

PERIOD: 1 OCTOBER 1975 - 1 MAY 1977

UPDATED 31 SEPTEMBER 1976

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

UNIVERSITY OF SOUTH CAROLINA

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

[illegible]

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

	CCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.
		▼ H10	▼ H8		▼ H8	▼ H8		▼ H8	▼ F8		▼ H8	▼ H8		▼ H8	▼ H8		▼ H8	▼ H8	
		▼ F8	▼ H16		▼ H8	▼ H16		▼ H8	▼ F16					▼ H8	▼ H16				
											▼ H12	▼ H24							
																	▼ H12	▼ H24	
																			▼ H69 F87 R42
		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	
	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		
				H30			F30			H30			F30			H30			
												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 S87	H16 S87	H16 S87	H12 S87	H16 S87	H16 S87	H16 S87	H16 S87	H16 S87	H16 S87	H4 S87	H16 S87	H16 S87	H16 S87	H12 S87	H16 S87	H16 S87	H16 S87	H16 S87
12.5	16.8	14.2	14.3	12.7	13.0	13.3	12.6	17.3	19.1	17.2	15.5	13.5	12.8	13.6	13.4	12.8	13.2	7.1