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STRESS STUDIES IN EFG

Program Manager: Juris P. Kalejs

Secondly Quarterly Progress Report - Subcontract No. 956312

Covering Period: October 1, 1982 - December 31, 1982

Distribution Date: February 15, 1983

"The JPL Flat Plate Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of flat plate solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE."

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ABSTRACT

A computer code which can account for plastic deformation effects on stress generated in silicon sheet grown at high speeds is fully operative. Stress and strain rate distributions are presented for two different sheet temperature profiles. The calculations show that residual stress levels are very sensitive to details of the cooling profile in a sheet with creep.

Experimental work has been started in several areas to improve understanding of ribbon temperature profiles and stress distributions associated with a 10 cm wide ribbon cartridge system.

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I. INTRODUCTION

A satisfactory model that can account for stresses generated in silicon sheet grown at high speeds is not yet available. Numerous attempts to account for residual stresses have been made, but all of these suffer inadequacies in one area or another. The most prominent of these are the lack of a fully two-dimensional treatment of stress and temperature fields in the sheet width dimension, and the omission of plastic deformation effects. Two significant factors preventing the development of such a model have been the absence of adequate information on creep behavior of silicon at high temperatures that could be applicable to the sheet growth situation, and of experimental data on temperature fields and deformation modes of growing sheet that could guide the modeling effort.

This report describes the work in progress under this subcontract to attempt to develop and test in the laboratory a stress-temperature field model for EFG silicon ribbon that deals with the above deficiencies. In one subtask, a computer code to predict stress-temperature field relationships in steady-state sheet growth is being developed at Harvard University. The stress state will be parameterized by a two-dimensional temperature field and growth speed. Incorporation of time dependent stress relaxation effects through a creep law is also planned to model the impact of plastic flow on the sheet residual stress state. A second aspect of the program will deal with the development of a model to predict the temperature field in a moving sheet from given system component temperatures (i.e., the sheet environment), and study experimental means to verify the model.
The goal of the program is to combine the results of these two areas of study to arrive at a model that can predict stress-temperature field relationships in steady-state silicon sheet growth under realistic conditions. Minimum stress configurations will be sought, and an attempt to construct an EFG silicon ribbon growth system that can verify this model will be made if it appears such configurations can be achieved experimentally. To aid the effort to correlate modeling results to experimental conditions, experimental means to examine temperature fields and stress generating process during growth are also under study.

II. PROGRESS REPORT

The results of modeling of stress distributions associated with growth of a silicon sheet at high speeds under several conditions of creep are described in the first section below. This work was carried out at Harvard University. Experimental work is being carried out in an EFG system for 10 cm wide ribbon at Mobil Solar in support of the stress analysis subtask. This is described in the second section below.

A. Stress Analysis of Steady-State Ribbon Growth (J.W. Hutchinson and J.C. Lambropoulos, Harvard University)

1. Model Description

The method of analyzing steady-state ribbon growth from the silicon melt was described in the previous quarterly report.\(^1\) In this quarterly period the computer code which implements the method was thoroughly checked out and has been used to analyze two prototypical cases. Results from these cases are discussed below. The computer program performed successfully in all respects; in particular, the iterative procedure converged with no difficulty. The program is
now ready to be "turned loose" to analyze the effects of temperature profile and pull speed on the stresses throughout the ribbon during growth, including the residual stresses in the end product. There are several open issues related to material properties and boundary conditions at the melt interface which need further investigation before the program can be fully exploited.

In the two calculations executed so far the ribbon is taken to have a width of 8 cm and length of 20 cm. For the temperature profiles considered this length is more than sufficient to ensure that the stresses at the cold end of the ribbon are the residual stresses at room temperature. Several finite element grids were used and some feeling for the sensitivity of the numerical results to grid spacing was obtained. A plot of one of the fine grids is shown in Fig. 1.

The method has been applied to calculate stress and strain distributions for the two sheet temperature profiles given in Fig. 2 in order to illustrate plastic deformation manifestations in silicon sheet growth. Profile 1 is representative of a temperature distribution in a 300 µm thick ribbon growing in an EFG cartridge system developed for growth of 10 cm wide ribbon. Cooling elements are located about 0.1 cm from the interface to enhance the speed capability, and an active afterheater is placed 1 cm from the interface for annealing purposes. This profile has been obtained from a combination of experimental data and detailed heat transport modeling, as described elsewhere. The second profile studied, curve 2 of Fig. 2, is derived in part from the experimental profile: Both profiles are identical between the growth interface and about 0.4 cm; thereafter this second profile has no reheat region but gradually turns into a linear profile beyond about 2 cm from the interface.
ULTRA FINE GRID

\[ Y \quad \longrightarrow X \text{ (growth direction)} \]

Fig. 1. Grid pattern for calculation domain.
Fig. 2. Temperature profiles along growth direction used in modeling of sheet stresses: 1., EFG system, and 2., idealized system.
The creep-rate was expressed in terms of the stresses by the following relation

$$\dot{e}^c_{ij} = c f(T)(\sigma_e/\mu)^{n-1}S_{ij}$$

(1)

where

$$f(T) = \frac{\exp(-\beta/T)}{T}$$

and where $\mu = 165$ GPa is the elastic shear modulus, $S_{ij}$ is the deviator stress and $\sigma_e = \sqrt{(3/2)S_{i}S_{j}S_{ij}}$ is the effective stress.

Complicated creep behavior has been reported at high (≥ 1000°C) temperatures in silicon. A creep law expression of the form of Eq. (1) valid for all stress levels and temperatures has not been found. $\beta$ is reported to be temperature dependent and exponents, $n$, ranging from 2 to 11 have been observed. Since creep behavior appropriate to high speed sheet growth conditions has not been investigated, we have chosen to model steady-state creep with a stress dependence of $n = 5$, and $\beta = 5.976 \times 10^4$ K, representative of intermediate stress level creep with an activation energy typical of self-diffusion. The constant C is fixed at $1.05 \times 10^{29}$ by a fit of creep data at 1300°C in one case (the "low creep" condition); a second case with C increased by a factor of $10^2$ is also examined (the "high creep" condition). The choices made for a representative creep relation for the modeling do not appear to invalidate an examination of plastic deformation process at the intended qualitative level of the discussion that follows.
2. Results

Comparisons of stress distributions for growth of an elastic sheet with a solid capable of creep according to Eq. (1) are given in Figs. 3 to 6. Figure 3 gives the variation of the stress component $\sigma_{xx}$ (residual stress) across the ribbon width at room temperature ($x = 20$ cm). Solutions at growth speeds of 3 and 6 cm/min are given here for high creep conditions. Distributions for $\sigma_{xx}$ and $\sigma_{yy}$ in an elastic solid along the ribbon center line for the cartridge system are compared in Fig. 4 for the two creep conditions at $V = 6$ cm/min. In Fig. 5, the variation of the stress components $\sigma_{xx}$ and $\sigma_{yy}$ with growth speed for the high creep condition is given over the length of the sheet near the ribbon center line for the idealized profile of Fig. 2. Strain-rate component distributions along the sheet center line are given under high creep condition for the two temperature distributions in Fig. 6.

3. Discussion

The calculated distributions in Figs. 3 to 6 show that the extent to which stresses are reduced from those in an elastic solid is particularly sensitive to details of the temperature profile in the sheet. In the range of growth speeds considered, variation of peak $\sigma_{xx}$ and $\sigma_{yy}$ values with speed is less than that produced by the hundredfold change in the creep both in the low and high temperature regions. The creep strain rate varies proportionally to growth speed (Fig. 6). This dependence on speed is considerably stronger than for $\sigma_{xx}$ and $\sigma_{yy}$.

At this stage in the work, these results should only be taken as indicative of what kinds of predictions are possible. Two important uncertainties which must be considered further are whether the creep law is adequate and, if it is,
Fig. 3. \( \sigma_{xx} \) variation across the sheet width at \( x = 20 \text{ cm} \) (room temperature) for the high creep case. Elastic solutions for idealized system, 1., and EFG system, 2., are compared to the solid with creep: idealized system at \( V = 3 \text{ cm/min} \), 3., and \( V = 6 \text{ cm/min} \), 4.; EFG system at \( V = 3 \text{ cm/min} \), 5., and \( V = 6 \text{ cm/min} \), 6.
Fig. 4. $\sigma_{xx}$ and $\sigma_{yy}$ variations along growth direction near ribbon centerline ($y/H = 0.025$) for KRG system at $V = 6$ cm/min.
Fig. 5. $\sigma_{yy}$ and $\sigma_{xy}$ variations along growth direction near ribbon centerline $(y/H = 0.031)$ for idealized system and high creep condition.
Fig. 6. Creep strain rate variations along growth direction near ribbon centerline for high creep conditions: 1. and 3. are \( \dot{\varepsilon}_{yy} \) for EFG system at 3 and 6 cm/min; 2. and 4. are \( \dot{\varepsilon}_{yy} \) for idealized system at 3 and 6 cm/min; 5. and 7. are \( \dot{\varepsilon}_{xx} \) for idealized system at 3 and 6 cm/min; 6. and 8. are \( \dot{\varepsilon}_{xx} \) for EFG system at 3 and 6 cm/min.
whether our choice of constants in the relation are appropriate. Secondly, all calculations reported above were carried out using the boundary condition that $\sigma_{yy} = 0$ at $x = 0$; i.e., at the solid interface at the melt. The mathematics of the steady-state growth process is such that any initial distribution $\sigma_{yy}(y)$ at $x = 0$ can be prescribed. On the basis of physical considerations we must decide whether $\sigma_{yy} = 0$ is correct.

B. Experimental Work (R.O. Bell and J.P. Kalejs, Mobil Solar)

1. Ribbon Growth

Ten centimeter wide ribbon has been grown with aluminum doped melts to attempt to provide information on ribbon temperature distributions to guide the modeling. Furnace 17 with the standard 10 cm cartridge with cold shoes has been used for this work. A series of growth velocity-ribbon thickness curves has been generated. The additional variable examined was the face heater power level which parameterizes the $V$-$t$ data, as shown in Fig. 7. Characterization of the ribbon by spreading resistance profiles to monitor aluminum redistribution through the ribbon thickness is planned to examine the sensitivity of the interface shape to operating conditions. This information will be used to arrive at temperature boundary conditions more representative of this growth system for use in calculating ribbon temperature profiles during growth.

A second experimental subtask for support of the above work has been started to develop a fiber optics system for ribbon temperature measurement during growth. The measurement technique chosen uses the light piping properties of aluminum oxide fibers. These can be used up to the melting point of silicon (1420°C), and can improve spatial resolution of the measurement over conventional thermocouples, while also offering the opportunity to measure temperatures while
Fig. 7. Thickness-velocity curves for 10 cm cartridge system with cold shoes at face heater temperature settings of a) 1420°C and b) 1431°C. The dashed line in Fig. 7b is for a continuously imposed velocity change that terminated in a ribbon pull-out or meniscus break at the highest speed.
the ribbon is moving.

The utility of optical fibers in crystal growth applications has been demonstrated already, and the principles of detection of radiation using either quartz or aluminum oxide fibers have been developed for several applications. The approach chosen here uses $\text{Al}_2\text{O}_3$ fibers, as described by R.R. Dils of the National Bureau of Standards. Equipment procurement for this subtask is underway at present.

Additional experimental work has been carried out on growth of 10 cm wide ribbon for residual stress studies. The standard 10 cm cartridge with cold shoes was used, and the experiments were done in Furnace 17. Ribbon was grown at several afterheater thermocouple settings, which were progressively decreased from the normal operating point of 1100°C to 1000°C, 950°C, and 900°C. Temperature profiles in the ribbon will be calculated for these settings and will be used as input data for stress analysis to obtain residual stress states for the ribbon.

A new experimental subtask was initiated this quarter to examine means to measure creep in silicon ribbon at the high temperatures where the above modeling results indicate the creep has the most significant effect on residual stress. The initial feasibility study will use a three-point bending method. This equipment will be set up in the hot zone of Furnace 17.

2. **Temperature Field Modeling**

A number of computer runs were made to examine the sensitivity of the model used to calculate temperature profiles in ribbon for use in stress analysis to various parameters. Complete temperature profiles were calculated, but for ease of comparison only the gradient in the solid at the interface is
shown in Table I. The thermal conditions were for a "cold shoe" system as shown in Figs. 1 and 2 of the First Quarterly progress Report.\(^\text{(1)}\)

The baseline conditions were for a ribbon thickness of 200 \(\mu\text{m}\), a velocity of zero, semi-transparent silicon with free carrier absorption, and a die-radiation shield gap temperature, \(T_B\), of 1500\(^\circ\text{C}\). Under these conditions, the gradient is 1147\(^\circ\text{C/cm}\) which would allow a growth rate of 3.7 cm/min if latent heat contributed entirely to the gradient.

Table I shows that the most sensitive parameter appears to be \(T_B\). Also quite important is the optical model used for the silicon; i.e., is it a black body or semi-transparent for radiation below its band gap. Black body means the total energy radiated is given by \(\varepsilon \sigma T^4\) where the emissivity, \(\varepsilon\), is one minus the reflectivity and here \(\sigma\) is the Stefan-Boltzmann constant. The growth velocity is important, although not nearly as critical as the other two factors in influencing the interface temperature gradient in the solid.

3. Model Application

The temperature distributions calculated from a combination of experimental data and temperature measurements in the EFG system for 10 cm wide ribbon growth described here have been used in the testing of the model for stress analysis, as seen in Section IIA. The additional characterization of the system through the subtasks outlined above will be used to guide future development of a more sophisticated model for calculation of temperature profiles in EFG ribbon. Information regarding the residual stress state of the ribbon grown under different conditions is also required. Preliminary studies of the best means to obtain such information are underway.
Some observations give insight into a number of areas that may be valuable in helping to guide future modeling directions. These come in the form of information on residual stress and buckling associated with 10 cm wide ribbon grown in the EFG system, represented by profile 1 in Fig. 2. Average residual stresses in this ribbon are observed to be low, of the order of 1 MPa, as estimated by an order of magnitude level using a "split-ribbon" technique. While the residual stress level does not measurably increase with speed over the range from 2.5 to 4.5 cm/min, ribbon buckling does become more severe with the increases in speed. The buckles are permanent; i.e., not elastic deformations, and appear to be essentially "frozen in" by high temperature stress relief.

Tentative conclusions that can be made from these experimental observations are:

(i) Creep rates comparable to the highest values calculated (Fig. 6) are needed to reduce residual stresses from those typical of an elastic sheet to levels of the order of those observed in ribbon grown with the EFG system. At this point, the validity of the creep law of the form modeled cannot be evaluated because of other factors that influence the solutions generated (see Section IIA).

(ii) The stresses likely to cause the buckling observed in EFG ribbon are dominated by $\sigma_{yy}$, which has its peak values within 1 cm of the interface (Fig. 4). For all the conditions examined, the peak values of $\sigma_{yy}$ remain much greater than $\sigma_{xx}$ and they do not undergo the large variations with creep intensity that are observed for the residual stress. If appreciable stress relief is to be accomplished after buckle formation near the interface with the EFG system, then creep rates typical of the high creep conditions modeled again are required.
(iii) In the absence of the annealing region provided by the active afterheater in the EFG system; i.e., for profile 2 of Fig. 2, $\sigma_{xx}$ can be decreased only by assumption of a higher creep rate than that modeled, or by an extension of the linear portion of the profile closer to the interface.

Table I. Effect of Various Parameters on the Gradient in the Solid. Baseline Case Conditions are $t = 200 \mu m$, $T_B = 1500^\circ C$, $V = 0.0$ cm/min and Semi-Transparent Silicon with Free Carrier Absorption. Deviations from the Baseline Case are Shown in the Column Labeled Conditions.

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<th>Conditions</th>
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<td>$T_B = 1200^\circ C$</td>
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<td>$V = 6$ cm/min</td>
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<tr>
<td>Black body</td>
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<td>Black body, $V = 2$ cm/min</td>
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<tr>
<td>Semi-transparent, no free carrier</td>
<td>1037</td>
</tr>
</tbody>
</table>

REFERENCES

1. J.P. Kalejs et al., First Quarterly Report, DOE/JPL 956312/82/01 (October 1982).


## APPENDIX

**WORK BREAKDOWN STRUCTURE AND PROGRAM PLAN**

*July 9, 1982 - July 8, 1983*

"STRESS STUDIES IN EFG"

<table>
<thead>
<tr>
<th>SUBJECT</th>
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