640
5/21/84

(JB)

(1)

DR-0073-6

DOE/JPL/956312-06
(DE84009044)

Energy

S
O
L
A
R

STRESS STUDIES IN EFG

Quarterly Progress Report for the Period October 1—December 31, 1983

February 15, 1984

Work Performed Under Contract No. NAS-7-100-956312

Mobil Solar Energy Corporation
Waltham, Massachusetts

Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A03
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts (ERA)*; *Government Reports Announcements and Index (GRA and I)*; *Scientific and Technical Abstract Reports (STAR)*; and publication NTIS-PR-360 available from NTIS at the above address.

DOE/JPL/956312-06
(DE84009044)

Distribution Category UC-63b

Mobil Solar Energy Corporation
16 Hickory Drive
Waltham, Massachusetts 02254

STRESS STUDIES IN EFG

Program Manager: Juris P. Kalejs

Quarterly Progress Report - Subcontract No. 956312

Covering Period: October 1, 1983 to December 31, 1983

Distribution Date: February 15, 1984

"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."

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

ABSTRACT

Stress analysis at Harvard University has examined the implication of non-zero interface stresses on model predictions. Stress distributions at distances greater than about 1 mm from the interface are shown to be independent of the interface stress at high creep intensities, and the predictions based on zero initial stress can be used with confidence. Numerical models for growth dynamics developed at MIT are compared with experimental data on t - V relationships and on interface shape obtained from impurity redistribution in aluminum-doped 10 cm wide ribbon.

Comparison of primary creep responses in FZ and CZ silicon above 1200°C using four-point bending indicates that oxygen has a significant influence on the creep rate. Both the strain rate and resulting dislocation densities generated in FZ silicon are an order of magnitude higher than for the CZ material at comparable applied stress levels.

A fiber optics probe suitable for temperature measurement during sheet growth has been constructed and tested. Study of the feasibility of using laser interferometric techniques for residual stress measurements has continued at the University of Illinois. The method has been successfully applied to CZ silicon, and is being evaluated for use with EFG ribbon.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

TABLE OF CONTENTS

<u>SECTION</u>		<u>PAGE</u>
	ABSTRACT	iii
I	INTRODUCTION	1
II	PROGRESS REPORT	3
	A. Advanced System Design	3
	1. Stress Analysis	4
	2. Temperature Field Modeling	6
	B. Creep Law Studies	13
	1. Stress-Strain Relationships	13
	2. Defect Characterization	17
	C. Fiber Optics Temperature Sensor	28
	D. Residual Stress Measurements	32
	REFERENCES	33
	APPENDICES	35

TABLE OF CONTENTS (continued)

List of Figures

FIGURE

1	Simplified model predictions for stress near the growth interface ($x = 0$) at ribbon centerline for $V = 0.1$ cm/min ...	5
2	Variations in the streamline function ψ with the pull rate for (a) 2.5 cm/min and (b) 4.0 cm/min	8
3	Variations in the streamline function ψ with the capillary length at a pull rate of 2.5 cm/min	10
4	Variations in the streamline function ψ , (a) and (b), and the isoconcentration lines C, (c) and (d), with the pull rate: (a) and (c) are at 2.5 cm/min; (b) and (d) are at 4.0 cm/min	11
5	Comparison of transient creep responses of FZ and CZ at 1215°C	15
6	Comparison of transient response of CZ silicon for 8-min and 10-sec loading at 1215°C	16
7	Dislocation-free Region A of FZ and CZ silicon after 10-sec load at 1215°C	18
8	Region B of intermediate stress of FZ and CZ silicon after 10-sec load at 1215°C	19
9	Region C of maximum stress of FZ and CZ silicon after 10-sec load at 1215°C	20
10	Region B of intermediate stress and Region C of maximum stress for CZ silicon after 8 minutes at 1215°C	21
11	High magnification micrographs of (111) FZ and CZ silicon after 10-sec loading at 1215°C	24
12	Variation of dislocation density through thickness of (111) CZ silicon after 8-min loading at 1215°C	25
13	(111) FZ silicon creep response at 1215°C	26
14	(111) CZ silicon creep response	27
15	Schematic of optical thermometer	29
16	Ratio of $0.6\text{ }\mu\text{m}$ to $0.7\text{ }\mu\text{m}$ signal as a function of temperature of reference thermocouple	31

I. INTRODUCTION

A satisfactory model that can account for stresses generated in silicon sheet grown at high speeds is not yet available. Many attempts to calculate residual stresses have been made, but all of these suffer from inadequacies in one area or another. 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 silicon ribbon EFG. In one subtask, a computer code developed at Harvard University to calculate stress with plastic deformation in steady-state sheet growth is being applied to study temperature field-stress relationships. The stress state is parameterized by a two-dimensional temperature field and growth speed. Incorporation of time dependent stress relaxation effects is through a creep law to model the impact of plastic flow on the sheet residual stress state. A second aspect of the program deals 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 studies experimental means to verify the model. This work is attempting to integrate temperature field modeling in the solid which takes into account real system geometries with a more detailed heat transfer model developed at MIT for the die top and meniscus regions below the growth interface.

The predictions of the stress analysis and temperature field models are supported on a qualitative level by experimental results obtained from characterization of 10 cm wide ribbon grown in an EFG cartridge system. Residual stresses in both buckled and unbuckled as-grown ribbon are much lower than predicted for the thermoelastic case. These can only be achieved through high rates of creep for the temperature profiles that are calculated for the 10 cm

ribbon. Defects are observed in the ribbon cross section which originate from strain instabilities.

Both modeling and experimental work are in progress to attempt to apply the stress analysis at a more quantitative level. The modeling is examining stress-temperature field relationships in a search for low stress growth configurations. The experimental efforts are in study of the high temperature creep response of silicon to provide information on the constitutive relationship, development of a technique to measure the temperature field in the sheet during growth, and of means to measure residual stress distributions. A temperature sensor based on fiber optics has been constructed for the temperature field measurements. A laser interferometry technique is being evaluated for residual stress measurements at the University of Illinois.

Attempts to verify aspects of the stress and temperature field modeling with the 10 cm cartridge system have met with limited success. Due to the complexity of the growth interface environment it has not been possible to use the fiber optics sensor. The cartridge system has lacked flexibility to allow imposition of changes in growth conditions and system temperature fields in a known and controllable manner in order to study temperature field-stress relationships at a quantitative level. A system that has the required flexibility for testing of the stress analysis model is being constructed.

II. PROGRESS REPORT

A. Advanced System Design

Preliminary design work has been started on constructing an EFG system for testing some quantitative aspects of the stress analysis model. The purpose initially will be to use a very simple post-grown environment to obtain relationships between the external system component temperature field, the ribbon temperature field, and stress. Speed capacity initially will not be a consideration. The complicated cartridge environment above the growth interface is replaced by graphite insulation that is shaped in a wedge of a fixed angle surrounding the plane of the ribbon. The wedge angle will be varied so as to generate different view angles at the growth interface, hence to obtain known and systematic changes in the ribbon surface heat flux and vertical temperature profile.

The other features of this test system will be that it will be open above the growth interface to a degree sufficient that the current design of fiber optics temperature probe can be tested more easily, and used to verify the temperature profile changes due to design variations. The profile will also be monotonically decreasing to eliminate the complication in stress relief associated with the reheat region of the active afterheater region of the current 10 cm cartridge. The first experiments will be with 5 cm wide ribbon.

Development of models for stress analysis and temperature field modeling for the EFG sheet growth system has reached a stage where examination of real system geometries at a semi-quantitative level is warranted. Various aspects of these models have been discussed in previous reports and qualitative predictions are shown to be in agreement with available experimental results. The next step is to provide a more rigorous test of the models by application to the case of the simplified growth system described above.

Two particular problems related to the modeling work in progress are examined next. A simplified analysis of near-interface region creep behavior is given in Section 1. The influence of surface tension-driven flow on interface impurity segregation is discussed in Section 2.

1. Stress Analysis (J.W. Hutchinson, Harvard University)

Work on the effect of the initial stress boundary condition was carried out, and a clear picture of how to deal with this condition has now emerged. A simplified model governing the behavior of the stresses very near the melt interface has been proposed and solved for a number of different initial stresses.

As pointed out in earlier reports, the steady-state ribbon growth model allows for the specification of an arbitrary initial stress distribution σ_{yy} at the melt interface at $x = 0$ (see Fig. 1 for conventions). This initial stress is basic to solidification under growth conditions and has not been clearly understood from the point of view of fundamental physics of the solidification process. In some of our earlier calculations using the full steady-state numerical formulation we found inconsistencies in our numerical results when this initial stress was non-zero and we noticed numerical difficulties very near the melt.

Fig. 1. Simplified model predictions for stress near the growth interface ($x = 0$) at ribbon centerline for $V = 0.1$ cm/sec.

To gain further insight into the stress variations very near the melt we have developed an approximate equation governing the component of stress σ_{yy} along the centerline of the ribbon ($y = 0$). Results for the variation of σ_{yy} very near the melt interface ($0 \leq x \leq 0.2$ cm) are shown in Fig. 1 for different values of the initial stress $\sigma_{yy}(x = 0) \equiv \sigma_0$. The parameters of the creep law are the same as those used previously for the "high creep" case. The important result which is clear from Fig. 1 is that beyond about 0.1 cm the stress distribution is independent of the initial stress and the prediction based on zero initial stress can be used with confidence. Furthermore, the exceptionally steep gradients in σ_{yy} when $\sigma_0 > 20$ MPa are what caused the difficulty in the full numerical calculations.

2. Temperature Field Modeling (H.M. Ettouney and R.A. Brown, MIT)

The theoretical analysis of the experimental measurements of variations in the crystal thickness and dopant segregation along the melt/solid interface as a function of the growth rate is continued by studying effects of the gradient of surface tension along the melt/gas interface on the flow field and the dopant distribution in the melt and its segregation along the melt/solid interface. Calculations of the field variables are made by using the finite element models developed in reference [1].

Flow Field and Mass Transfer Models

The velocity field and the solute distribution in the melt are calculated by solution of the two-dimensional Navier-Stokes equation and the material balance equation; see reference [2] for details of the equations and boundary conditions. The thermo-capillary flow field is introduced by replacing the zero shear condition along the melt/gas interface with the balance of surface

tension gradient and the viscous shear stress. In dimensionless form

$$\mathbf{n} \cdot \mathbf{\tau} = \mathbf{t} \cdot \nabla T,$$

where $\mathbf{n}$ and $\mathbf{t}$ are the unit normal and tangent vectors to the melt/gas interface, $\mathbf{\tau}$ is the viscous stress tensor, and ∇T is the temperature gradient. The field variables are dimensionalized by using the characteristic velocity $V^* = d\gamma/dT \cdot \Delta T / \mu$, where $d\gamma/dT$ is the surface tension coefficient derivative, ΔT is the characteristic temperature scale which measures the difference in temperature between the solidification temperature and the top of the die, and μ is the dynamic melt viscosity. For example, taking $\Delta T = 10^\circ\text{K}$ and the properties of silicon into account, where $\mu = 0.0088 \text{ dyne s/cm}^2$ and $d\gamma/dT = 0.104 \text{ dyne/}^\circ\text{K cm}$, results in a characteristic velocity of 118.18 cm/s, which is three orders of magnitude larger than typical growth velocities.

Results

Inspecting the above shear stress condition, in its dimensionless form and taking into account the large magnitude of the calculated temperature gradients along the melt/gas interface, where $\mathbf{t} \cdot \nabla T$ in the melt is varied between 700 and 1200 K/cm as the growth velocity is increased from 2.5 cm/min to 4 cm/min (see reference [2]), one expects development of very vigorous flow field. Figure 2 shows variations in the calculated streamlines for the molten zones and temperature gradients calculated in reference [1] as the growth rate was increased. All dimensions are referenced to a length scale of 0.025 cm. At a growth rate of 2.5 cm/min, the maximum V_x and V_y , which occur along the melt/gas interface, are 304 and 815 cm/min, which are two to three orders of magnitude greater than the growth velocity. As a result, the flow cell occupied all of the

Fig. 2. Variations in the streamline function ψ with the pull rate for (a) 2.5 cm/min and (b) 4.0 cm/min.

molten zone at the top of the die and also it expanded into the capillary spacing. At these conditions, the streamlines of the growth convection are tightly packed along the boundaries of the molten zone (the melt inlet, the die inner walls, the melt/gas interface, and the melt/solid interface).

For the purpose of calculating the dopant distribution in the melt, the flow fields shown in Figure 2 are not appropriate because of end effects. First, close to the melt inlet boundary, the large size of the flow cell generates a two-dimensional flow field. As a result, the assumption of no back-mixing breaks down, and the solute mass cannot be conserved in the molten zone. This problem is solved by increasing the capillary length. The resulting streamlines are displayed in Figure 3, where capillary lengths of 0.5 and 3, in dimensionless units, are used at a growth rate of 2.5 cm/min. As shown, at the capillary length of 3 a long region of fully-developed and one-dimensional flow is generated close to the melt inlet boundary. The second end effect, also caused by the size of the flow cell, is encountered along the melt/solid interface, where the streamlines are poorly resolved because they turn and bend within a very thin zone in order to satisfy the normal velocity condition. This problem can be solved by refining the finite element mesh within the zone next to the melt/solid interface. This type of solution was not attempted. Instead, a lower surface tension coefficient ($d\gamma/dT$) was used to calculate the flow field. This results in a smaller flow cell, more accurate streamlines, and a better material balance for the solute.

Figure 4 shows the streamlines ψ and the isoconcentration lines $C(x,y)$ normalized to the incoming concentration C_0 , for a dopant with equilibrium distribution coefficient $k_0 = 0.1$ and a mass diffusivity $D = 0.0005 \text{ cm}^2/\text{s}$, calculated at growth rates of 2.5 and 4 cm/min, with a surface tension

(a)

(b)

Fig. 3. Variations in the streamline function ψ with the capillary length at a pull rate of 2.5 cm/min.

Fig. 4. Variations in the streamline function ψ , (a) and (b), and the isoconcentration lines C , (c) and (d), with the pull rate: (a) and (c) are at 2.5 cm/min; (b) and (d) are at 4.0 cm/min.

coefficient ($d\gamma/dT$) of 0.0104 dyne/cm K. As shown, the streamlines close to the melt inlet are one-dimensional and there is good resolution near the melt/solid interface. The high intensity of the flow field (Figures 4(a) and (b)), resulted in a region of uniform solute concentration in the center of the molten zone (Figures 4(c) and 4(d), and also large solute segregation along the melt/solid interface is obtained because of the large lateral component of velocity along the interface. These results, maximum V_x and V_y , the stream function at the center of the flow cell, and solute segregation, are tabulated below. Further increase in the magnitude of the surface tension coefficient ($d\gamma/dT$) results in a slight increase in solute segregation along the melt/solid interface; e.g., at a growth rate of 2.5 cm/min, ΔC increased from 41.4 to 42.1% as $d\gamma/dT$ is increased from 0.0104 to 0.0156 dyne/cm K. Eventually, ΔC starts to decrease as $d\gamma/dT$ is continued to be increased, because the large velocities in the flow field increase the region of uniform solute concentration (see reference [2]).

Table of Calculated Variables with Surface Tension-Driven Flow.

Growth Rate (cm/min)	$\Delta C^\dagger$ (%)	$\langle k_o C_I \rangle$	V_x (cm/min)	V_y (cm/min)	ψ Cell
2.5	41.4	1.08	43.7	100.1	-0.0169
3	36.5	1.08	48.2	103.4	-0.0161
3.5	31.5	1.05	48.1	98.1	-0.0144
4	24.3	1.02	47.1	91.1	-0.0132

$$\dagger \Delta C = 100 k_o [C(x=0) - C(x=t)]$$

However, at this point, the assumptions in the model, i.e., conduction-dominated heat transfer in the melt and small viscous stresses along the melt/gas interface, must be questioned. These assumptions may no longer hold because of large velocities in the molten zone. If these enhance convective relative to conductive heat transfer and also increase the viscous stress

contribution in the stress balance equation along the melt/gas interface, then simultaneous solution of the flow field and thermo-capillary equations becomes essential for correct solutions.

B. Creep Law Studies

1. Stress-Strain Relationships

The high temperature ($\geq 1000^{\circ}\text{C}$) creep response of silicon is being studied using four-point bending. Emphasis here is on relatively short (≤ 10 minutes) time non-steady-state behavior. Displacement-time graphs are generated, from which the stress-strain and stress-strain rate transient relationships are established.

Two extreme cases for stress distributions in the sample during the time of deformation are of interest. In the one case, a simple linear distribution of stress through the sample thickness is assumed. The maximum surface stress σ_s is then related to the loading moment M by

$$\sigma_s = \pm \frac{6M}{Wt^2} \quad (1)$$

for width under constant stress W and sample thickness t . In the second case, the applied stress is assumed to be instantaneously distributed through the sample thickness with an average (constant) magnitude $|\sigma_{av}|$ that is now related to the bending moment by

$$\sigma_{av} = \pm \frac{4M}{Wt^2} \quad (2)$$

As is deduced from examination of defect distributions below, the experimental situation for high temperature creep likely produces a stress distribution that results in effective stresses acting on the sample interior somewhere between

these two extremes. For the purposes of creep law estimates, the uncertainty on stress is thus of the order of 50%; i.e., of the order of the numerical constant ratio in Eqs. (1) and (2).

Linear strain conditions with pure bending are assumed for estimation of the strain magnitude. Then the strain distribution through the thickness of the sample from the centerline ($y = 0$) to the surface is

$$\epsilon = \pm ky \quad (3)$$

with

$$k = 8\delta^2/\ell^2 \quad (4)$$

where δ is the deflection over the sample length ℓ . The maximum surface strain is

$$\epsilon_s = \pm \frac{4t}{\ell^2} \delta^2 \quad (5)$$

This strain ϵ_s has been calculated from measured displacements under load as a function of time, and the results are plotted in Figs. 5 and 6 for four-point bending carried out at 1215°C. Figure 5 compares very short time (≤ 10 seconds) responses of FZ and CZ silicon wafers. Constant strain rate curves are also shown for the assumption that $\epsilon = At$, so that $\dot{\epsilon} = A$, the slope of the ϵ - t curve, for the sake of illustration of strain rate response. The strain rate for the FZ silicon in the transient stage is more than an order of magnitude greater than that for the CZ wafers. This is attributed to oxygen influence, which impedes dislocation multiplication and motion in the Cz silicon. Figure 6 compares the CZ silicon wafer responses at very short (≤ 10 seconds) to longer times (~ 10 minutes) of loading. (In all cases shown, the load is removed after the time under bending shown and the sample is cooled to room temperature under zero load.) The values of σ_s for FZ and CZ loading were 12.7 and 11.1 MPa,

Fig. 5. Comparison of transient creep responses of FZ (dashed curve 1) and CZ (dashed curve 2) at 1215°C . Solid curves represent constant strain rate paths of: a. $3 \times 10^{-4} s^{-1}$, b. $1 \times 10^{-4} s^{-1}$, c. $2 \times 10^{-5} s^{-1}$, d. $6 \times 10^{-6} s^{-1}$, and e. $3 \times 10^{-6} s^{-1}$.

Fig. 6. Comparison of transient creep response of CZ silicon for 1. 8-min, and 2. 10-sec loading at 1215°C . The solid curves represent constant strain rate paths of: a. $6 \times 10^{-6} \text{ s}^{-1}$, and b. $3 \times 10^{-6} \text{ s}^{-1}$.

respectively, the difference arising from a wafer thickness variation. The average stresses calculated using Eq. (2) are 8.5 and 7.4 MPa, respectively.

The defect distributions achieved after loading in these three samples are discussed next.

2. Defect Characterization

a. Comparison of FZ and CZ Silicon

An initially dislocation-free CZ wafer was placed in the four-point bending apparatus and heat treated for one hour at 1215°C with zero load to provide a reference. No dislocations or sample bending were observed after this treatment along the entire length, showing that the heat treatment itself and the load provided by the sample weight are not a factor in defect generation at this temperature. This sample, as well as the others used here, had a (111) surface orientation and a $\langle 211 \rangle$ direction along the long axis of the sample (perpendicular to the bending axis). The bending axis (and the normal to the cross-sectional surfaces shown below) was $\langle 110 \rangle$. The CZ samples had measured substitutional carbon at 4.5×10^{17} at/cc and interstitial oxygen of 1.0×10^{18} at/cc. The FZ wafer had nondetectable levels ($\leq 1 \times 10^{16}$ at/cc) for both impurities.

Cross-sectional micrographs for the FZ and CZ material after a 10-second load application are compared in Figs. 7 to 9 at three locations along the sample length where the stresses are different: Region A is stress free; Region B has an intermediate and varying level of stress; Region C is the uniform stress portion between the innermost pivots. In Fig. 10, Regions B and C are shown for a CZ wafer that was subjected to the same loading stress as the sample in Figs. 7 to 9, but this time for 8 minutes. In all photographs, the top is the tension

(a)

(b)

Fig. 7. Dislocation-free Region A of (a) FZ, and (b) CZ silicon after 10-sec load at 1215°C. $\langle 110 \rangle$ bending axis is perpendicular to plane of photograph (652X).

(a)

(b)

Fig. 8. Region B of intermediate stress of (a) FZ, and (b) CZ silicon after 10-sec load at 1215°C. $\langle 110 \rangle$ bending axis is perpendicular to plane of photograph (70X).

(a)

(b)

Fig. 9. Region C of maximum stress of (a) FZ, and (b) CZ silicon after 10-sec load at 1215°C . $\langle 110 \rangle$ bedding axis is perpendicular to plane of photograph (70X).

(a)

(b)

Fig. 10. Region B of intermediate stress (a) and Region C of maximum stress (b) for CZ silicon after 8 minutes at 1215°C. $\langle 110 \rangle$ bending axis is perpendicular to plane of photograph (70X).

side of the wafer and the $\langle 110 \rangle$ bending axis is perpendicular to the plane of the photographs.

Significant differences are in evidence in the defect distributions of the three samples. Several preliminary observations can be made at this time:

(1) The dislocation density in the CZ silicon is between one and two orders of magnitude lower than in the FZ material (e.g., Fig. 9) for the 10-second loading. Nucleation conditions near the rough surface, which also generates the highest dislocation density in its proximity, in the CZ material are equivalent to those in the FZ wafer. As was argued for the strain rate above, this difference between FZ and CZ wafers can be attributed to the presence of oxygen and carbon impurities at higher concentrations in CZ silicon. Oxygen, in particular, has been shown to act to increase barriers for dislocation movement. The influence of carbon has not been studied previously, and is under investigation. The lower CZ wafer dislocation density is attributed here to the action of these impurities in increasing barriers for dislocation multiplication which produces the higher FZ wafer dislocation densities. This difference is reflected in the strain rate responses, as seen in Fig. 5.

(2) The CZ wafer exhibits a defect asymmetry through the thickness not present in the FZ sample. This persists after the 8-minute loading also (compare Figs. 9 and 10). Each sample had one polished and one rough surface. The latter was always positioned on the tension side. It is known that a rough surface is a preferential region for dislocation generation and provides a homogeneous distribution of nucleation centers. The CZ response is again shown to be qualitatively different from that of the FZ material for these transient conditions in that the asymmetry persists for the longer times examined (8 minutes), whereas already at 10 seconds the FZ dislocation distribution is closer

to being symmetric (Fig. 9). This asymmetry must be related to the decreased rate of dislocation generation and movement from the rough (tensile stress) surface of the wafer toward the centerline. Once the defect asymmetry is established during the early transient stages, this also produces an asymmetric distribution of stresses internally, which must further lengthen the time required for the CZ distribution to reach the more symmetric distributions seen in the FZ wafer.

(3) All stressed samples exhibit denuded zones where outdiffusion of dislocations has occurred.

High magnification micrographs in Fig. 11 of the sample cross sections shown in Fig. 8 illustrate the dislocation configurations in the FZ and CZ silicon after 10-second loading at 1215°C (the loading of Fig. 5). Additional details of the asymmetric CZ silicon dislocation distribution shown in Fig. 10b for a longer time of 8 minutes are given in Fig. 12.

b. Creep Dependence on Stress and Temperature

Extensions of the initial studies have included varying the load and going to a higher temperature of 1360°C . Figure 13 shows displacement-time curves for transient creep for (111) FZ silicon at 1215°C at three different bending stresses. Similar data for CZ silicon is given in Fig. 14, where response at two loads at 1215°C and one at 1360°C are shown. Additional characterization and experiments will be done to complete these studies at 1360°C .

(a)

(b)

Fig. 11. High magnification micrographs of (111) FZ (a), and CZ (b) silicon after 10-sec. loading at 1215°C. The dislocation densities are for (a) $1.4 \times 10^7/\text{cm}^2$, and for (b) $2.5 \times 10^5/\text{cm}^2$.

(a) tension surface

(b) center

(c) compression surface

Fig. 12. Variation of dislocation density through thickness of (111) CZ silicon after 8-min loading at 1215°C:
(a) $N_D = 4.1 \times 10^6/\text{cm}^2$, (b) $7.8 \times 10^6/\text{cm}^2$, (c) $3.5 \times 10^5/\text{cm}^2$.

Fig. 13. (111) FZ silicon creep response at 1215°C for loads of:
1. 8.6 MPa, 2. 12.7 MPa, and 3. 15.6 MPa with
<110> bending axis.

Fig. 14. (111) CZ silicon creep response: 1. 1360°C, 7.0 MPa; 2. 1215°C, 11.1 MPa; 3. 1215°C, 13.6 MPa with $\langle 110 \rangle$ bending axis.

C. Fiber Optics Temperature Sensor

A new design of fiber optics temperature probe has been constructed and tested. It incorporates a water-cooled jacket to surround the optical fibers so that they are shielded from stray radiation. The new probe has been evaluated and calibrated and will be used to attempt to measure ribbon temperature in the test system being constructed for stress model verification. Non-reproducibility of signal has been traced to two sources. It is found that the optical quality of the sensor tip may be susceptible to degradation with thermal cycling. Polishing of the Al_2O_3 sensor tip is found to significantly increase the signal strength. Consequent degradation is not rapid, however, as several cycles up to 1400°C and prolonged use at the highest temperatures do not immediately reduce the signal strength to levels typical of those with non-polished tip values. The second source of non-reproducibility has been found in mechanical shifts in the fiber optics configuration. Because there are not available sufficiently long lengths of Al_2O_3 (0.5-1 m), the composite fiber circuit shown in Fig. 15 has been constructed. This consists of about a 3 cm long Al_2O_3 rod 1 mm in diameter tapered at its tip; an intermediate region quartz rod; and a low temperature quartz bundle consisting of up to 30 very small (0.1 mm) diameter quartz fibers. The need to couple and hold these three sections in a stable configuration has required construction of a casing. However, during heating and cooling, the individual sections are free to move, and these adjustments are thought to contribute to non-reproducibility. Mechanical alignment problems were also experienced with the photomultiplier tube and filter adjustments, and these have now been for the most part corrected.

Fig. 15. Schematic of optical thermometer.

The current design of probe appears to have reasonable stability and reproducibility sufficient to make it useful for measurements of ribbon temperatures. It has been calibrated in crystal growth Furnace 17 under conditions that expose the fiber optics circuit to conditions encountered in an actual system. The results of several calibration runs which involved thermal cycling through the range from 1100°C to 1400°C are shown in Fig. 16. The probe tip was located about 1 mm from a graphite plate, and about 0.5 cm away from a thermocouple that was inserted and glued into a recess in the plate (see Fig. 16 inset). The probe and thermocouple were located in the main zone heater region.

The total spectral flux entering the fiber tip is

$$I(\lambda, T) = \frac{a \epsilon_o c_1}{\lambda^5 [\exp (c_2/\lambda T) - 1]} \text{ [W/m]} \quad (6)$$

where a = area of the cavity exit, m^2
 ϵ_o = apparent emittance of the cavity
 c_1 = first radiation constant, $3.7418 \times 10^{-16} \text{ W m}^2$
 c_2 = second radiation constant, $1.43879 \times 10^{-2} \text{ m K}$
 λ = wavelength in vacuum, m
 and T = temperature, K .

If measurements of intensity are made at two wavelengths λ_1 and λ_2 , and the ratio is taken as

$$R = \left(\frac{\lambda_2}{\lambda_1} \right)^5 \frac{\exp (c_2/\lambda_2 T - 1)}{\exp (c_2/\lambda_1 T - 1)} \quad (7)$$

then R is a function only of T . The two wavelengths chosen here are $\lambda_1 = 0.6 \mu m$ and $\lambda_2 = 0.7 \mu m$. The experimental ratio R_{exp} is found to be further dependent on two correction factors:

$$R_{exp} = C_F C_o R$$

Fig. 16. Ratio of 0.6 μm to 0.7 μm signal (R_{exp}) as a function of temperature of reference thermocouple.

where

$$C_F = 1.377902$$

$$C_O = 1.588310$$

are to account for the filter band widths and the photomultiplier tube response, respectively. C_F has been obtained using IR spectrophotometer scans of the filters, and C_O is obtained from the calibration run, as shown in the data of Fig. 16. A reasonably constant average slope is obtained for the plot of R_{exp} vs. the thermocouple temperature above about 1200°C which is the same as that obtained from Eq. (7) above, while the level of non-reproducibility in the current probe shown in Fig. 16 is about $\pm 3-5^\circ\text{C}$.

It is expected that improvements in this uncertainty can still be made. Examination of different probe tip geometries and coatings is one area that will be investigated.

D. Residual Stress Measurement

A question has been raised recently regarding the influence of geometrical effects on residual stress distributions in ribbon samples cut to a typical blank size, say, $10 \times 5 \text{ cm}^2$. The differences in stress level from stress redistribution caused by cutting of long sheets into shorter lengths is of concern in efforts to relate measured stress levels in convenient laboratory-size blanks to original sheet residual stress distributions.

The analysis of Andonian and Danyluk [1], presented for interpretation of stress distributions calculated from blank deformation modes, incorporates a non-zero shear flow. This shear flow is zero in the regions of a sheet which are removed from those affected by end effects, which extend about a blank half-width from each end. The shear flow functional form is a factor in the residual stress

calculation, and this may need to be examined more closely in interpreting deformation mode measurements.

The laser interferometric measurement of residual stress demonstrated previously for CZ silicon [1] has not worked on EFG ribbon. An increase in surface roughness of the latter in comparison to CZ material has made interpretation of fringe patterns difficult. For this reason, a new approach to obtaining fringe patterns, the shadow-Moire technique, is being evaluated. Also, a four-point bending fixture for applying stress to rectangular 5 cm by 10 cm blanks has been constructed. Preliminary results indicate that fringe patterns on EFG material can be obtained from shadow-Moire interferograms.

REFERENCES

1. J.P. Kalejs et al., JPL Quarterly Report (July 1, 1983 to September 30, 1983), December 15, 1983.
2. H.M. Ettouney and R.A. Brown, "Mechanisms for Lateral Solute Segregation in Edge-defined Film-fed Crystal Growth", J. Applied Physics, in print, 1984.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX I
WORK BREAKDOWN STRUCTURE AND PROGRAM PLAN
July 9, 1983 - July 8, 1984
"STRESS STUDIES IN EFG"

'84

Subject	Description	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL
Theoretical	Modeling of reduced stress growth configurations.	X-----X												
Experimental	Advanced system design concept testing.	X-----X												
	High temperature creep measurements in silicon.	X-----X												
	Yield stress studies above 1000°C in silicon.	X-----X												
	Temperature measurements with optical fiber systems in support of modeling.	X-----X												
	Development of improved optical fiber tips.	X-----X												
Program Management	Residual stress measurement technique development.	X-----X												
	Progress Reports, etc.	X-----X												