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STRESS STUDIES IN EFG

Quarterly Progress Report for the Period January 1—March 31, 1984

July 15, 1984

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

Mobil Solar Energy Corporation
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STRESS STUDIES IN EFG

Program Manager: Juris P. Kalejs

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ABSTRACT

Four-point bending studies have been continued to compare primary creep response of FZ and CZ silicon above 1200°C. Results suggest that silicon can be treated as a completely plastic solid in the temperature range 1200°C-1400°C in its response to thermal stress generated during sheet growth. In the stress range of 5-15 MPa, the strain rate dependence on stress is $\dot{\epsilon} \sim \sigma^{10}$, and appears to be essentially temperature independent.

Work is underway to compare stresses predicted for two different thermal models used for temperature field calculations. Stress analysis has been performed to illustrate the dependence of sheet stresses on growth parameters such as sheet width, thickness and growth speed, and the coupling of these parameters to the details of the temperature field calculations used as input to the stress model.

The construction of a simplified EFG ribbon system to be used to test the stress model and to investigate means to achieve low stress growth configurations is completed.

The first results of measurement of residual stress distributions in EFG sheet using shadow-Moire interferometry have been obtained at the University of Illinois.

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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. 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

The work on this program at Mobil Solar has concentrated in two areas this quarter: development of experimental and theoretical capabilities to test the stress analysis model (Section A below), and study of high temperature primary creep response of silicon using four-point bending (Section B below). In addition, experiments are underway at the University of Illinois to demonstrate the feasibility of measuring stress distributions in EFG sheet with a shadow-Moire interferometric technique. The details of this work are given in the following sections.

A. Advanced System Design

1. Experimental

Construction of a simplified EFG ribbon growth system for purposes of testing the stress analysis model and for investigating means to achieve low stress growth configurations is completed and has been installed in Furnace 17. The main departures from the 10 cm EFG cartridge system under development for a high speed capacity involve simplification of the post-solidification cooling environment by use of simple graphite insulation geometries. It will be possible to more accurately determine the temperatures of these components and of the ribbon surface in order to give confidence to temperature field specification for the stress calculations.

2. Theoretical (H.M. Ettouney)

Work is underway at Mobil Solar to combine two heat transfer models developed for analysis of the EFG system. The first model, which was used to calculate sheet temperature fields and relate them to system components, included only heat transfer above the growth interface [1]. A detailed analysis of radiative contributions to heat flux at the sheet surface was incorporated, and solid radiative effects

were added to the usual treatment of lattice thermal conductivity. It is denoted here by the terms "gray body" model and calculations in order to emphasize these special features. However, this model is not capable of predicting meniscus influence on interface gradients or operating variable relationships that can be used to test the model.

The second effort to develop a heat transfer model has proceeded in collaboration with MIT, where a finite element scheme has been applied to model a growth system that includes the die and the meniscus [2]. This incorporates coupled solutions for heat transfer and capillarity. This model accounts for heat transfer in the meniscus and predicts thickness-velocity relationships, interface shape and solute segregation for given temperature boundary conditions in the environment. Experimental data are available for these variables to provide a basis of comparison, and the model predictions have been successfully fitted to the data. However, solid phase heat transfer is treated using the black body approximation in that a constant value of surface emissivity is used, and only lattice heat conduction is included. This model is referred to as the "2-D black body" model here.

The sheet temperature profiles obtained from these two models differ noticeably in their predictions, given a fixed environment temperature distribution, in the crucial region of high gradients very near the growth interface. The aim of future work is to integrate the full heat transfer representation of the gray body model with the 2-D black body model. The initial comparison of these models has been concluded, and it is discussed next. The first section below compares the predictions of the temperature profiles given by the two models. In the second part, dependences of maximum σ_{yy} and σ_{xx} stresses in the sheet as a function of sheet width and growth speed are given for the two models.

a. Temperature Profiles

The temperature profiles shown in Fig. 2 are obtained from the two models for the cartridge system environment temperature distribution of Fig. 1. The interface gradient and cooling rate predicted on the basis of the 2-D black body model (curve 2) is greater than that at an equivalent thickness sheet of 105 microns obtained in the gray body calculation (curve 1'). The previous EFG temperature profile used in stress calculations, curve 1, is also shown for the gray body model. This assumes a ribbon thickness of 300 microns. The relevant interface parameters for the calculations in Figs. 2 and 3 are compared in the table below:

Table I. Interface Quantities for Two Thermal Models.

Model	Sheet Thickness (μm)	V (cm/min)	T' ($^{\circ}\text{C}/\text{cm}$)	$(\alpha T)''$ ($^{\circ}\text{C}/\text{cm}^2$)
Gray body (1)	300	3	1337	1.9×10^{-2}
Gray body (1')	105	4	2033	4.8×10^{-2}
2-D black body (2)	105	4	2465	9.8×10^{-2}

The variation of $(\alpha T)''$ with axial coordinate is given in Fig. 3 for the three profiles in Fig. 2. The profiles converge beyond about 1 cm from the interface.

a. Stress Distributions

Figures 4 and 5 show variations in the maximum values of σ_{yy} and σ_{xx} as a function of sheet width and pull rate for the temperature profile 2 obtained from the 2-D black body model. The low creep condition defined in previous calculations [3] has been modeled in all cases. For comparison, the single points for 8 cm wide sheet reported in earlier calculations are also given.

Fig. 1. Experimental (environment) vertical temperature profile. Temperature is scaled by $T^* = 1783\text{K}$ and distance is scaled by $L^* = 0.025\text{ cm}$.

Fig. 2. Temperature profiles near interface ($X = 0$) calculated from environmental profile of Fig. 1 using: 1. gray body model with sheet thickness of $300 \mu m$ and $V = 3$ cm/min; 1'. gray body model with sheet thickness of $105 \mu m$ and $V = 4$ cm/min; 2. 2-D black body model, thickness of $105 \mu m$ and $V = 4$ cm/min.

Fig. 3. Variation of $(\alpha T)''$ with axial coordinate for the temperature profiles of Fig. 2.

Fig. 4. Variation of maximum σ_{yy} stress for 2-D black body model with sheet width at two growth velocities and ribbon thicknesses: (a) 3 cm/min, 144 microns, (b) 4 cm/min, 105 microns. The solid point is for the gray body model at 6 cm/min and 300 microns.

Fig. 5. Variation of maximum σ_{xx} stress for 2-D black body model with sheet width at two growth velocities and ribbon thicknesses: (a) 3 cm/min, 144 microns, (b) 4 cm/min and 105 microns. The solid point is for the gray body model at 6 cm/min and 300 microns.

The purpose of displaying the results in the form given in Figs. 4 and 5 is that this shows for the first time the trends in sheet stresses that may occur during dynamic growth condition changes. In real growth situations, changes in sheet thickness, growth speed and width often occur simultaneously, particularly during seeding. A second application is also relevant. In typical systems the cooling capacity is constant, and thus sheet growth speed increases are accompanied by a decrease in thickness. This restriction has often hampered interpretation of experimental results on buckling thresholds in the 10 cm cartridge, where the highest speeds of 4 cm/min were only reached with accompanying decreases in ribbon thickness. In the 2-D black body model, the curves at each velocity have been generated at constant values of temperature variables which have been used to fit experimental t - V relationships [1].

The advantage of the 2-D black body model, then, is that it can be used to predict sheet temperature profile changes that accompany growth condition changes traced out in the operating space allowed by real system boundary conditions, viz., those regions where coupled solutions of the heat transfer and capillary solutions allow stable growth. However, the thermo-capillary model predicts higher temperature gradients and in turn higher maximum stresses than those predicted by the detailed radiation model. This discrepancy is being examined by modification of the 2-D black body model to include the same details of the radiative heat transfer model developed in [1]. The completion of this step should enable us to vary and test operating parameters that have direct impact on the temperature distribution in the crystal and in turn on the magnitude of the maximum stresses in the crystal. The maximum axial stresses, calculated at room temperature, are higher here than the maximum practical limit at which buckling may occur, which is ≥ 50 MPa. Lower values are obtained by either using higher creep intensities, which relieve high stresses generated by large temperature gradients, or by using gray body radiation, which results in lower temperature gradients and in turn lower stresses. From the above, it is clear that further investigation of these two conditions is important in order to obtain a clear picture of the stress generation factors in silicon sheets related to plastic deformation mechanisms.

B. Creep Law Studies

Stress analysis of sheet growth with plastic deformation included has shown that residual stress is very sensitive to the shape of the cooling profile through the constitutive law for creep. The form of this expression appropriate to the sheet growth situation is not known. Expressions for strain rate as a function of stress and temperature derived in steady-state creep experiments have been used in the initial analyses. In the present investigation of creep response, the primary creep regime at relatively low strains ($\leq 10^{-2}$) and intermediate strain rates ($10^{-6} \text{ s}^{-1} \leq \dot{\epsilon} \leq 10^{-2} \text{ s}^{-1}$) has been studied in the temperature region between 1200°C and 1400°C , where little information is available. Variables such as the carbon and oxygen concentration, orientation effects and defect densities have been investigated for CZ and FZ wafers deformed in a four-point bending apparatus.

1. Four-Point Bending Results

The majority of the results reported are for samples with (111) surface orientation and $\langle 110 \rangle$ bending axis. In the as-received state, the FZ and CZ wafers were polished on one side and rough on the other. The influence of the surface finish was investigated by comparing the creep response of a CZ wafer stressed with the rough surface to that with the polished surface in tension. The results are shown in Fig. 6. After eight minutes under a constant load of 11 MPa at 1225°C , the strain with the rough surface in tension is about twice that when the polished surface is in tension. However, the strain rate responses up to about 2 min are fairly similar, while an accelerated creep stage appears to be developing in the case of the rough surface in tension after the initial transient creep region. This difference is attributed to the change in availability of nucleation sites on the tension surface, which then affects the rate of dislocation generation.

Fig. 6. (111) CZ silicon creep response for a stress of 11 MPa and temperature of 1225°C with (a) polished surface, and (b) rough surface in tension. $\langle 110 \rangle$ bending axis.

The dislocation distributions for the samples stressed as in Fig. 6 are shown in Fig. 7. The CZ wafers all show an asymmetry in defect distribution between the tension and compression sides, which is present with both the rough and smooth surfaces in tension. These wafers are not oriented for single slip, and two slip systems appear to be activated, which perhaps contributes to the asymmetry as a result of dislocation interactions between the systems.

Another aspect of creep investigated was the response of wafers under light loads at temperatures close to the melting point. The results of these experiments at 1370°C and above are shown in Fig. 8. In a preliminary test, an FZ wafer was loaded to 1.4 MPa and then slowly heated up to 1395°C. No deflection was measurable after 100 minutes of this treatment. At the end of the experiment it was found that one end of the sample was partially melted. The sample temperature was measured and found to be about ten degrees hotter than that indicated by the thermocouples used to monitor the experimental conditions. These are placed in the knife edges at either end of the sample. This correction to temperature is applied to all values reported here, and a correction to increase all previously reported temperatures for these experiments by 10°C is necessary. The dislocation configuration for this undeflected sample is shown in Fig. 9(a).

This high temperature experiment with FZ silicon was repeated under two other conditions, as shown by curves 1 and 4 in Fig. 8. In the first case (curve 1), the wafer was loaded to 0.7 MPa and heated up to a temperature of about 10°C lower than that found to lead to melting. No deflection was measurable after 110 minutes. The sample stress was then progressively increased, as indicated, up to 5.2 MPa. Transients of a few minutes' duration were produced after each incremental load application and then the strain tended to level off toward a reduced steady-state creep rate.

(a)

(b)

Fig. 7. Effects of surface roughness on dislocation configuration in (111) CZ at 1225°C and 11.0 MPa: polished surface in (a) compression, and (b) tension, with tension surface at top.

Fig. 8. High temperature, low stress creep response of several different silicon materials:
 1. multiple loading for FZ with a. 3.5 MPa, b. 4.2 MPa, c. 5.0 MPa; see text
 for other details.

(a)

(c)

(b)

Fig. 9. (111) FZ silicon dislocation configurations after very high temperature ($\sim 1400^{\circ}\text{C}$) loading: (a) $N_D = 5.5 \times 10^5/\text{cm}^2$; (b) $N_D = 4.0 \times 10^6/\text{cm}^2$, and (c) $N_D = 1.1 \times 10^6/\text{cm}^2$. Compression surface at bottom (165X) and $\langle 110 \rangle$ bending axis. See text for details.

The multiple loading curve 1 can be compared to the case of application of a single load (curve 4) with a stress 5.0 MPa approximately equal to the final stress of curve 1. The same lower temperature of 1375°C was used. The resultant strain for the single load applied for 50 seconds is half that of curve 1. The creep has generally leveled off to an approximately constant strain rate, although an accelerated creep stage appears to be developing. The dislocation configurations after the long and short times of stress are shown in Figs. 9(b) and 9(c). The long time multiple stressed sample of curve 1 (Fig. 9(b)) shows indications that significant polygonization has occurred. This is not the case for the sample of curve 4 (Fig. 9(c)).

Comparable stress conditions to that of curve 4 in Fig. 8 were applied to CZ wafers of two different carbon concentrations, with results as shown in curves 2 and 3. These show that carbon does additionally decrease the strain and creep response beyond that produced by the oxygen. The dislocation configurations for the samples of curves 2 and 3 in Fig. 8 are given in Fig. 10. The higher carbon sample of Fig. 10(a) has the lower dislocation density $N_D \approx 9 \times 10^4/\text{cm}^2$ as compared to $N_D \approx 4 \times 10^5/\text{cm}^2$ for Fig. 10(b).

The dependence of the creep response on impurity concentrations and orientation is further illustrated in Fig. 11, which examines a higher stress range. Here, CZ and FZ silicon data at 1370°C and stresses between 7 and 16 MPa are compared, including a (100) surface CZ silicon sample. Again, the higher creep response in FZ silicon is evident from comparison of responses at comparable stress levels. The (100) CZ sample, with very low carbon but 1×10^{18} at/cc oxygen does not exhibit a qualitatively different strain rate from that of the FZ silicon at the highest stress levels of 15 MPa. However, there is considerable uncertainty with respect to resolution of the time scale at these short times, so further conclusions cannot be drawn. The higher carbon CZ (curves 2 and 3) does show some indication that the creep response remains reduced from that of FZ silicon even at higher loads.

(a)

(b)

Fig. 10. (111) CZ silicon dislocation configurations after 50-second application of 5.0 MPa stress at 1370°C for (a) intermediate carbon ($C_s \sim 4 \times 10^{17}$ at/cc), and (b) low carbon ($C_s \lesssim 1 \times 10^{17}$ at/cc) material.

Fig. 11. Creep response of FZ and CZ silicon at 1370°C. $\langle 110 \rangle$ bending axis.

1. (111) CZ at 7.0 MPa	$C_s \approx 5 \times 10^{17}$ at/cc
2. (111) CZ at 12.0 MPa	$C_s \approx 5 \times 10^{17}$ at/cc
3. (111) CZ at 15.5 MPa	$C_s \approx 5 \times 10^{17}$ at/cc
4. (111) CZ at 13.5 MPa	$C_s \approx 1 \times 10^{18}$ at/cc
5. (100) CZ at 13.5 MPa	$C_s \lesssim 1 \times 10^{17}$ at/cc
6. (111) FZ at 8.0 MPa	
7. (111) FZ at 12.0 MPa	
8. (111) FZ at 14.0 MPa	
9. (111) FZ at 15.5 MPa	

Comparisons of dislocation configurations produced in a number of the samples of Fig. 11 are given in Figs. 12 and 13.

The stress and temperature dependences of the strain and strain rate observed in the experiments carried out here, and those at a lower temperature reported previously [4], make it possible to deduce mechanisms of stress relief that may be operative in sheet growth. This is illustrated with the help of Figure 14. The stress-strain relationships used to model plastic relaxation are those obtained for the region of large strains and high dislocation densities, where stresses are in excess of those occurring at the lower yield point (region 3). The primary creep relationships studied here are for very low strains ($\leq 0.1\%$) and dislocation densities not in excess of $5 \times 10^7/\text{cm}^2$ (region 1). The strain rates achieved are comparable, however, and in the range of $10^{-6} \text{ s}^{-1} < \dot{\epsilon} < 10^{-2} \text{ s}^{-1}$, typical of sheet growth.

Preliminary estimates of the stress dependence of the primary creep indicate $\dot{\epsilon} \sim \sigma^{10}$ for region 1. The response is moreover a very weak function of temperature, with an activation energy which cannot be resolved within the accuracy of the experiments. The results also suggest that silicon above 1200°C can be treated as an essentially plastic solid insofar as its responses to stresses above 5 MPa are concerned. The primary creep rate in region 1 of Fig. 14 is then limited solely by dislocation dynamics, viz., their velocity and the extent to which dislocations on different glide systems interact with each other and with other extended defects. In the case of EFG sheet, the latter consist mainly of twin boundaries. These will have a considerable influence on the extent of stress relief and final dislocation configurations. Their influence is not accounted for in the present studies because the starting material is dislocation and defect free silicon. Attempts are underway to obtain a more quantitative estimate of the primary creep magnitudes for the available data.

(a)

(b)

Fig. 12. Comparison of (111) silicon materials for 1370°C loading.
(a) High carbon ($\sim 10^{18}$ at/cc) CZ silicon, 13.5 MPa.
(b) FZ silicon, 15.5 MPa.

(a)

(b)

Fig. 13. (100) CZ silicon dislocation configuration for loading of 13.5 MPa at 1370°C.

- (a) Region B - intermediate stress.
- (b) Region C - high stress.

1. Primary Creep - Present Work

$$0 \leq \epsilon \leq 10^{-2} \quad , \quad 0 \leq \dot{\epsilon} \leq 10^{-3} \text{ s}^{-1}$$

$$N_D \leq 5 \times 10^7 / \text{cm}^2$$

2. Lüders Bands (Mahajan et al., Acta Met. 27(1979) 1165.)

Observed for $T \leq 1000^\circ\text{C}$

3. Secondary Creep - Steady-State

$$\epsilon \geq 1 - 10\% \quad , \quad N_D \geq 10^8 / \text{cm}^2$$

Fig. 14. Stress-strain regimes for creep.

Lüders strain (region 2 of Fig. 14) is an additional possible means of stress relief to be considered in sheet growth. It is generally observed only below 1000°C [5], when the upper yield point is clearly demarcated. There is evidence of Lüders bands in EFG sheet, as shown in the cross-sectional micrograph of Fig. 15. This shows regions of high dislocation density which occur always in bands of the order of a few sheet thicknesses in extent. An unmistakable characteristic of these bands is that they consist of dislocation arrays that are not aligned along the major (111) slip planes (Fig. 15(c)). In Fig. 15, the latter are demarcated by the twin planes. This corresponds to a characteristic feature reported also for Lüders bands, which have been observed to form preferentially with slip vectors $\pm 1/2 [101]$.

C. Fiber Optics Thermometer

A number of improvements in detector electronics are being implemented in order to improve sensor stability and reliability. No further work with the probe is planned until the system described in Section IIA is operational.

D. Residual Stress Measurements

The first residual stress distributions have been obtained on EFG material using a shadow-Moire technique of interferometry. The experimental results are described in Appendix I. The results are encouraging in that stress variations across the sample width are observed. Maximum stress levels also are not unreasonable. Additional testing needs to be carried out in order to evaluate the ability of the measurement to follow stress changes produced by variations in growth conditions.

a

b

c

Fig. 15. Cross-sectional micrographs of highly-dislocated regions in 10 cm wide EFG ribbon cross section associated with Lüders strain. (c) is a high magnification view of (b). Growth direction is normal to paper.

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APPENDIX I

Quarterly Report on Residual Stress Measurement in EFG Silicon Ribbon

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January 24, 1984

Submitted to Mobil Solar Energy Corporation

Residual stress measurements of EFG ribbon and nonagon sheet have continued this past quarter. A four-point bend fixture needed for measurements on rectangular geometries has been fabricated and laser double-beam and shadow-Moire interferometry have been used to obtain interference patterns. We have been successful in utilizing the shadow-Moire technique in producing interferograms on the "rough" EFG and nonagon silicon sheet. These experimental data were used to extract the residual stresses at the mid-section of two nonagon samples (#47R1-1 and -2), both grown at 2 cm/min.

A. Description of Four-Point Bend Apparatus

The four-point bend apparatus is shown in Fig. 1. The rectangular silicon sheet is supported by the inner-span loading pins and pressed against the upper-span loading pins by a 25-lb. load cell, the output of which measures the forces used to produce the out-of-plane deflection. The inner-span and outer-span dimensions are 6.35 cm and 8.89 cm, respectively.

B. Shadow-Moire Interferometry

A shadow-Moire interferometry technique developed specifically to produce interferograms on rough surfaces has been applied to nonagon silicon sheet. Interferograms were obtained from two nonagon sheets (#47R1-1 and #47R1-2), both grown at 2 cm/min. Residual stresses at the centerline of the sheet were computed and the results shown in Figs. 2 and 3.

As can be seen, there appears to be a slight asymmetry in the stress distribution at this position on the sample and the zero-stress line does not equally apportion the positive and negative stress areas which should sum to zero. In addition, compressive stresses are apparently at the edges and tensile stresses at the center section. Since there is no external force acting on the ribbon in the direction of the ribbon growth, the total axial effect of the residual stress distribution in the x direction should be equal to zero. Thus, the area between the stress curve and the y axis (see Figs. 2 and 3) should algebraically add up to zero. This discrepancy in our graphs is believed to be due to the assumed average values of Young's Modulus and Poisson's Ratio of 2×10^{11} Pa and 0.3, respectively. These values, together with surface deflections, were used to determine residual stresses. It can be shown that a change in Young's Modulus due to anisotropy of the preferred grain orientation shifts the zero axis of the stress distribution without producing a noticeable change in the stress profile. A shift of the zero axis that results in a neutral position which will equally apportion the positive and the negative areas can be used therefore to extract the Young's Modulus for the ribbon.

We are continuing to analyze this data, and measurements are being taken from two other nonagon samples grown at 1.75 and 2.25 cm/min.

Fig. 1. Schematic diagram of four-point bend apparatus.

EFG specimen 47R1-1

Fig. 2. Residual stress distribution at the centerline of a nonagon sheet grown at 2 cm/min.

EFG specimen 47R1-2

Fig. 3. Residual stress distribution at the centerline of a nonagon sheet grown at 2 cm/min.