

STRESS IN SILICON RIBBONS CRYSTALLISED FROM A MOLTEN ZONE: A STUDY OF THE INFLUENCE OF GROWTH PARAMETERS

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ABSTRACT: Stress is frequently the limiting factor in silicon ribbon production processes. This is particularly true for processes using perpendicular growth from the liquid, such as EFG, SSP, S-ribbon or those being studied in our laboratory, based on growth from a molten zone.

Here, we report the results of a study of stress in silicon ribbons produced by one of our techniques. A series of samples were grown specifically for this study, in which only a few parameters were varied. Growth rate ranged from quasi-stationary conditions (6 mm/min) to very high (120 mm/min). The samples were grown in different atmospheres: argon, nitrogen and oxygen. Residual stress was measured by infrared photoelasticity.

The general behaviour of the residual stress field is reasonably understood. It was shown that there is a concentration of stress near the edges of the ribbons. The asymmetry of the stress distribution is shown to increase with the growth rate and the incorporation of impurities in the material.

Keywords: Silicon – 1; Ribbons – 2; Stress – 3

1. INTRODUCTION

Residual thermal stress is frequently a limiting factor in silicon ribbons production processes. This is particularly true for processes using perpendicular growth from the liquid, such as EFG, SSP, S-ribbon or those being studied in our laboratory, based on growth from a molten zone. Residual thermal stresses, which are proportional to $\partial^2 T / \partial x^2$ during the cooling process, are associated with the formation of dislocations and other defects that degrade the electrical properties of the material.

In this paper we present a new system that uses infrared photoelasticity to measure the residual stress ($\sigma_1 - \sigma_2$) of silicon ribbons¹. The method is based on the facts that (i) the optical absorption of silicon in the infrared is of the order of 20 cm^{-1} and therefore the material is transparent to infrared light and (ii) stress changes the birefringence properties of the material, hence the polarization of light travelling through the material will rotate, the amount of rotation being proportional to the stress.

We report the results of a study of stress in silicon ribbons produced by one of our techniques. A series of samples were grown specifically for this study, in different atmospheres (argon, nitrogen and oxygen). Growth rate ranged from low (6 mm/min) to very high (120 mm/min).

2. DESCRIPTION OF THE METHOD

For a transparent and isotropic material the principal components of the refraction index are coincident with the principal components of the stress and we can write

$$n_1 - n_2 = C(\sigma_1 - \sigma_2) \quad [\text{Eq. 1}]$$

where n_1 and n_2 are the principal components of the refraction index and σ_1 and σ_2 the principal components of

¹ The difference of the principal components of the stress tensor ($\sigma_1 - \sigma_2$) is known as the Tresca stress and, for a 2-dimensional problem, is equal to twice the maximum shear stress. It is therefore generally considered in most engineering fatigue models as the most damaging because it controls crack initiation [1].

the stress tensor. C is a constant, called stress-optic constant or photoelastic coefficient.

In a crystal, the direction of the principal components of the stress tensor are not, in general, coincident with those of the refraction index and therefore the photoelastic coefficient is not constant and depends on the orientation of the crystal [2].

Figure 1 shows the basic set-up for a photoelastic measurement. It includes (i) the light source followed by the polariscope, consisting of (ii) a linear polarizer P_1 and a quarter wave plate Q_1 , in order to produce circularly polarized light²; (iii) the sample, where the polarization of the light is shifted, proportionally to the stress and its thickness; (iv) a second quarter wave plate Q_2 and (v) the linear polarizer P_2 , called the analyser. Finally, the output signal is measured in (vi) a detector.

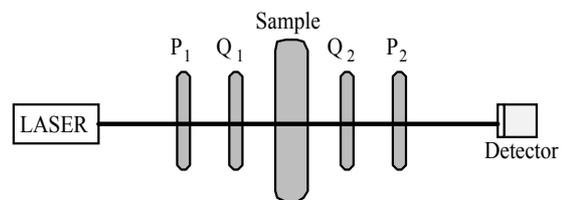


Figure 1: Set up for photoelastic measurement of stress.

The rotation of the analyser allows the formation of a fringe pattern from which the Tresca stress ($\sigma_1 - \sigma_2$) can be extracted. From equation 1, we can see that the condition for light extinction for a light beam with wavelength λ is

$$\sigma_1 - \sigma_2 = \frac{\Delta\lambda}{Cd} \quad [\text{Eq. 2}]$$

where Δ is the phase difference between the fast and the slow axis (also known as the isochromatic parameter or fractional order) and d is the sample thickness.

Experimentally, Δ can be measured using a phase stepping procedure. The output wave can be described by

² Using circularly polarized lights avoids the formation of isoclinic fringes that would make the analysis of the fringe pattern more difficult to interpret.

$$I = I_0 + I_m (\sin(2(\varphi - \phi))\cos\Delta) + I_m (\cos(2(\varphi - \phi))\sin(2(\phi - \theta))\sin\Delta) \quad [\text{Eq. 3}]$$

where φ is the angle of the analyzer P_2 , ϕ is the angle of the quarter wave plate Q_2 and θ the isoclinic parameter (the angle of the principal axis) [3].

If we run the system with 6 different configurations, as shown in Table I, we can independently determine the isocromatic parameter Δ .

Table I: Different configurations used to determine the isocromatic parameter Δ .

ϕ (Q2)	φ (P2)	Output
45	45	$I_1 = I_0 + I_m \cos 2\theta \sin \Delta$
45	135	$I_2 = I_0 - I_m \cos 2\theta \sin \Delta$
90	90	$I_3 = I_0 + I_m \sin 2\theta \sin \Delta$
90	180	$I_4 = I_0 - I_m \sin 2\theta \sin \Delta$
90	135	$I_5 = I_0 + I_m \cos \Delta$
90	45	$I_6 = I_0 - I_m \cos \Delta$

We have

$$I_m |\sin \Delta| = \frac{1}{2} \sqrt{(I_1 - I_2)^2 + (I_3 - I_4)^2} \quad [\text{Eq. 4}]$$

which leads to

$$\Delta = \arctan \left(\frac{\sqrt{(I_1 - I_2)^2 + (I_3 - I_4)^2}}{I_5 - I_6} \right) \quad [\text{Eq. 5}]$$

Since the thickness of the sample is measured directly on site, the only variable missing is the photoelastic coefficient, which depends on the orientation of the crystal. In the preliminary results discussed in this paper, we have considered an averaged value of $C = 20$ Br, which is valid for silicon crystals with a (100) orientation.

In further developments, the system will estimate the photoelastic coefficient at every point of the sample by measuring the stress with different (known) applied stresses. The residual stress at any point of the sample can thus be computed by extrapolation of the curve *Applied stress vs. Measured stress*.

3. DESCRIPTION OF THE SYSTEM

The light source is a 10 mW, 1310 nm infrared laser diode *Mitsubishi ML725B8F* modulated at a frequency of 250 Hz. The light detector is a *High Speed InGaAs detector, DET410*, from *Thorlabs* with an 800-1800 nm range. A HeNe laser was also installed for alignment purposes.

The output signal is measured by a *Stanford SR850* lock-in amplifier. The control of the XY-system and the trigger for the measurement is controlled automatically by software, through a GPIB connection. The total range of the XY-system is $10 \times 10 \text{ cm}^2$ and the maximum resolution is $25 \times 25 \mu\text{m}^2$, well below the 1mm laser spotsize.

The thickness of the sample is measured using a *NER 2.5 mm STD* transducer that allows measurements with a typical precision of a few micrometers (always less than 5%).

4. PRODUCTION OF RIBBONS

The polycrystalline silicon ribbons analysed in this study were produced by optical linear molten zone crystallization, a method being developed in our laboratory, which has been published elsewhere [4]. The pre-ribbons were Eurosil polycrystalline 350 μm thick wafers, 30mm wide.

A total of 12 samples were grown in different atmospheres (argon, nitrogen and oxygen), with growth rates ranging from low (6 mm/min) to very high (120 mm/min).

5. EXPERIMENTAL RESULTS

Figure 2 shows a typical distribution of the Tresca stress ($\sigma_1 - \sigma_2$) across a section of two silicon ribbons, grown at different growth rates.

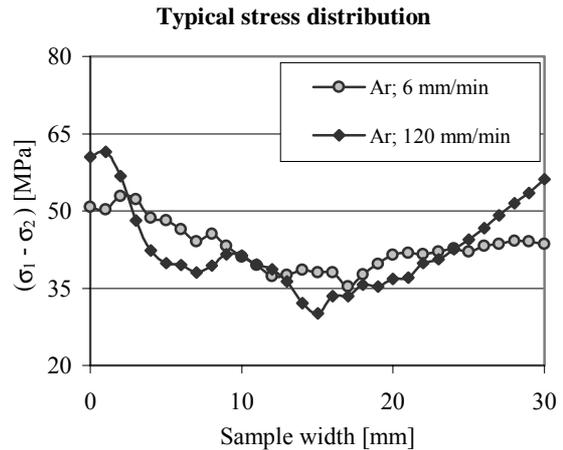


Figure 2: Typical distribution of Tresca stress ($\sigma_1 - \sigma_2$). Samples AMF 20 (Argon; 6mm/min) and AMF 36 (Argon; 120mm/min).

The short-scale variations of the stress field are directly correlated to the crystal grains since, as it was discussed in section 2, the estimated value of the stress is proportional to the photoelastic coefficient that was considered to be constant although it depends on the crystal orientation.

The maximum Tresca stress ($\sigma_1 - \sigma_2$), which is equal to twice the maximum shear stress in the sample, is of the order of 60 MPa, which should be compared to the minimum 250 MPa fracture shear stress [5]. All the samples studied have maximum Tresca stresses within the range 50-70 MPa.

Figure 2 also shows that there is an uneven distribution of the shear stress across the sample, with higher stress concentrations near the edges. This is due to the shape of the solid-liquid interface which, being ellipsoid-like, has a higher $\partial^2 T / \partial x^2$ near the edges.

The unevenness of the stress distribution can be characterised by an asymmetry factor, the ratio maximum stress/minimum stress. Figure 3 shows the effect of the growth parameters on the asymmetry factor.

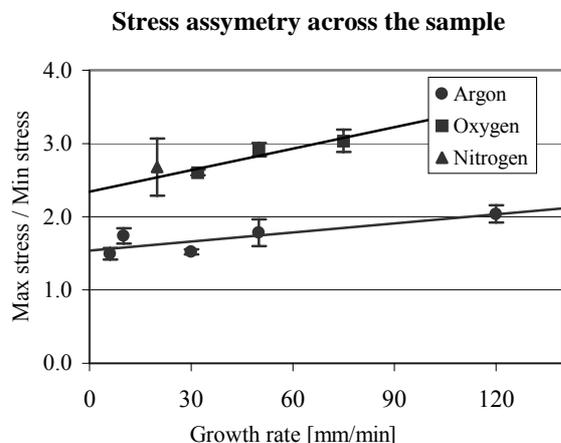


Figure 3: Stress asymmetry across the sample, for different growth parameters: growth rate and atmosphere.

From these results, it is clear that the asymmetry of the stress distribution is stronger for samples grown in oxygen or nitrogen than for the samples grown in an argon atmosphere. This is probably due to the incorporation of impurities in the material that leads to a decrease of the rate of strain relaxation [5].

It is also seen that the asymmetry of the stress distribution increases with the growth rate. This result was to be expected because higher growing speeds allow shorter relaxation times.

6. CONCLUSIONS

A fully automated infrared photoelastic system for measurement of stress in silicon ribbons has been developed. It is based on a phase stepping procedure with six different configurations of the polariscope, which enables the calculation of the isochromatic parameter, and thus the stress field, for any point of the sample, independently of its neighbours.

It was found that the maximum shear stress present in the samples is of the order of 30 MPa and does not show a clear dependence on the growth rate or the growing atmosphere. It has been shown that there is an uneven distribution of stress, with higher stress concentrations near the edges of the silicon ribbons. This concentration of stresses is due to the shape of the solid-liquid interface curvature, with sharper temperature gradients in the area near the edges.

As expected due to the decrease of relaxation time for high growth rates, the asymmetry of the stress distribution was shown to increase with the growth rate of the ribbon.

It was also shown that if the ribbon is grown in the presence of oxygen or nitrogen, the asymmetry of the stress distribution is enhanced. This could be related to the incorporation of impurities in the material that leads to a decrease of the the strain relaxation rate.

7. ACKNOWLEDGEMENTS

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8. REFERENCES

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