

Stress profile determination in chemically strengthened glass using scattered light

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A method is described for determining stress profiles in thin specimens of chemically strengthened glass by progressively etching away the surface compressive layer and measuring the change in magnitude of the centre tensile stress using the scattered light technique. The profile is derived from the relationship between thickness change and centre tension change. Some specimen results are presented for a commercial glass (Corning Code 0319) and other chemically strengthened glasses:

1. Introduction

The most practical way of strengthening thin glass components and plates is to introduce a surface compressive stress by an ion-exchange treatment. In addition to overcoming the strength-impairing effects of surface defects, the compressive stress generates a balancing internal tension which with suitable glass compositions and ion-exchange treatments can be above the threshold for fracture branching and thus give rise to the so-called frangible glasses. These materials have a characteristic and predictable disintegration pattern which can be initiated by the application of a small amount of local energy, just sufficient to penetrate the surface compressive layer.

In some applications it is this predictable failure pattern with the associated release of stored strain energy that is of main interest; the increased mechanical strength being of secondary importance.

For basic studies on such materials, an accurate and sensitive method for determination of stress profiles is essential particularly when the thickness of plates and components is of the order of 1 mm thick (here the maximum plate thickness was 1.25 mm).

This paper describes a combination of techniques which has proved particularly suitable for such specimens.

2. Measurement of stress profiles

Traditionally, stress profiles (or more accurately

principal stress differences) in glass plates are measured by the conventional photo-elastic method (see, for example, [1]) in which a beam of polarized light is passed through the edge and parallel to the surface of the plate. An alternative optical method is the scattered light technique [2-5] in which the principal stress differences are calculated from the spacing of the scattered light fringes observed normal to an incident polarized beam.

A third method, the layer removal technique, consists of progressively removing surface layers from the stressed specimen and measuring the resulting length changes. The stress distribution is calculated from the relationship between the thickness decrement and the concomitant length change. The technique has been used for residual stress analysis in metals [6] and more recently to determine stress profiles in opaque glass-ceramics [7, 8].

Each of the above methods has disadvantages. The conventional photo-elastic method is unsatisfactory for use with chemically stressed glass: when long path lengths are used slight deviations from the parallel between the incident and the plate surface, in combination with the steep stress gradient characteristic of such glasses, can lead to errors in the stress values, and optical distortion will inevitably result if the ion-exchange treatment causes refractive index changes either as a result of the different ion or the compressive stress. Although short path lengths reduce optical

distortion, the relative contribution from the boundaries makes interpretation of the stress pattern difficult, and edge imperfections further complicate the situation when thin sections are prepared. For these reasons, conventional photoelastic measurements on chemically strengthened glasses are often confined to a limited portion of a section about the centre plane, where the stress gradients are small and where the effects of ion-exchange on refractive index and stress-optical coefficient are minimal.

Although the scattered light technique has been used at oblique incidence to measure stress profiles in relatively thick specimens of thermally strengthened glass [5] in which the stress gradients were small, the characteristic steep stress gradient in chemically strengthened glasses necessarily restricts measurements to regions about the centre plane.

The main problem with the layer removal method is the small magnitude of the length changes resulting from layer removal: these are comparable with those resulting from ambient temperature variations, and accurate temperature control is essential.

None of the above techniques on its own is satisfactory. However, by employing an optical measurement restricted to the centre plane and combining it with the layer removal method, the principal stress difference profile can be inferred.

Of the two optical methods, the scattered light technique has several advantages for this purpose. Longer path lengths can be used with suitable specimens and retardations of several tens of orders can be observed and easily measured. In addition, the uniformity of spacing of the fringes is an indication of the uniformity of principal stress difference.

This combination of methods (scattered light plus layer removal) has been used to measure stress profiles in a commercially chemically strengthened glass (Corning 0319) and of variants obtained by ion-exchanging its precursor (Corning 0317) in molten KNO_3 for various times at different temperatures. The technique has also been used extensively with specimens subjected to high-temperature ageing treatments which accelerate ion-diffusion down the concentration gradient and consequently modify the stress profile [10].

3. The scattered light technique

The technique relies on the scattering effect of

small suspended particles or local compositional variations on an incident beam of light. A basic characteristic of the light scattered in an unstressed isotropic material is that it is always plane polarized. If the incident beam is plane polarized, then the scattered light shows different intensities in different directions. This is illustrated in Fig. 1a, where the beam entering the glass plate is plane polarized at about 45° to the main plane. Light scattered normal to the beam shows two directions (one on either side of the plate) of maximum intensity and two directions of minimum intensity. When viewed from the positions of maximum intensity the beam appears as a luminous streak.

If the plate is stressed with principal stresses P and Q normal to the beam (Fig. 1b) then the incident beam splits into two polarized components which travel with different velocities through the plate. The differences in velocities are proportional to $(P - Q)$ and as the components travel through the plate they periodically come into phase.

When the scattered light is viewed at about 45° to the main plane of the plate, fringes are seen and the distance between each fringe represents a relative retardation between the components of one wavelength (Fig. 2). By measuring the retardation from the fringe separation and knowing C , the stress optical coefficient, the value of $(P - Q)$ can be calculated from:

$$(P - Q) = \frac{\lambda}{Cb},$$

where λ is the wavelength, C the stress-optical coefficient, or birefringence, and b is the fringe separation.

When the plate thickness is small compared to the other dimensions, the edge effects in the Q direction are very small, P is very much greater than Q [4, 5] and λ/Cb becomes a direct measure of centre tension.

4. Effect of layer removal on stress profile

For a plate stressed by a uniform surface treatment, if the thickness t is small compared with other dimensions, it can be assumed that the stress in regions away from the edges is a function of depth only, and if the plate has been treated similarly on both surfaces, the stress profile, away from the edges, will be symmetrical about the centre of the plane of the plate.

The initial equilibrium condition requires that if $\sigma_0(x)$ describes the profile then:

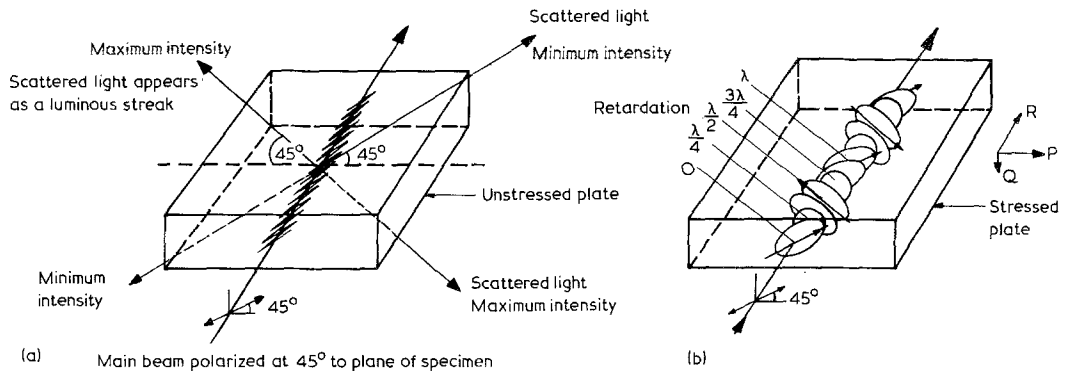


Figure 1 (a) Intensity of scattered light from an unstressed plate. (b) Formation of scattered light fringes in a stressed plate [4].

$$\int_{-t/2}^{t/2} \sigma_0(x) \cdot dx = 0$$

where x is the axis normal to the plane of the plate and $x = 0$ is the mid-plane.

If a thin layer of thickness Δx_1 is removed from each surface then the stress profile in the remaining section changes from the original $\sigma_0(x)$ to $\sigma_1(x)$ (Fig. 3). Removing this thin layer of surface compression is equivalent to applying a stress resultant to $\sigma_0(x)$ within the remainder of the section: if the elastic moduli are constant across the section, the result is a uniform change of stress across the section such that:

$$\sigma_1(x) = \sigma_0(x) + \Delta\sigma_1.$$

The surface compressive layer removed is equivalent to a stress resultant of magnitude:

$$2 \int_{(t/2)-\Delta x_1}^{t/2} \sigma_0(x) \cdot dx,$$

and since this produces a uniform stress change $\Delta\sigma_1$ over the remaining section then:

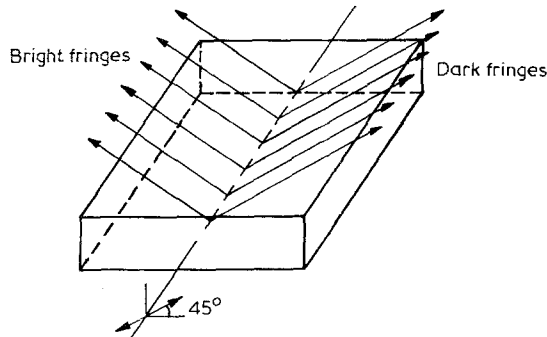


Figure 2 Scattered light fringes observed at approximately 45° to the stressed plate.

$$2 \int_{(t/2)-\Delta x_1}^{t/2} \sigma_0(x) dx = 2\Delta\sigma_1 \left(\frac{t}{2} - \Delta x_1 \right).$$

The integral in the above equation can be replaced by $\bar{\sigma}_1 \Delta x_1$, where $\bar{\sigma}_1$ is the average stress in the layer Δx_1 just before its removal. Then

$$\bar{\sigma}_1 = \frac{\Delta\sigma_1}{\Delta x_1} \left(\frac{t}{2} - \Delta x_1 \right).$$

Considering successive removal of layers, this expression can be generalized to give average value of stress in the n th layer just before its removal, i.e. after removing the previous $(n - 1)$ layers,

$$\bar{\sigma}_n = \frac{\Delta\sigma_n}{\Delta x_n} \left(\frac{t}{2} - \sum_{i=1}^n \Delta x_i \right). \quad (1)$$

The original average stress $\bar{\sigma}_{n_0}$ in the n th layer is determined from the total stress change on removing the previous $(n - 1)$ layers [7].

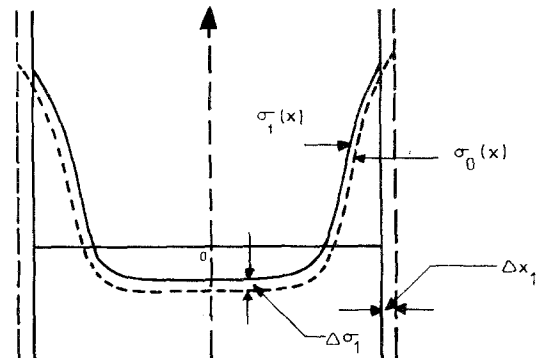


Figure 3 Modification of stress profile by removal of surface layer.

$$\bar{\sigma}_{n_0} = \bar{\sigma}_n + \sum_{i=1}^{n-1} \Delta\sigma_i, \quad (2)$$

and substituting Equation 1 into Equation 2,

$$\bar{\sigma}_{n_0} = \frac{\Delta\sigma_n}{\Delta x_n} \left(\frac{t}{2} - \sum_{i=1}^n \Delta x_i \right) + \sum_{i=1}^{n-1} \Delta\sigma_i. \quad (3)$$

The stress profile is calculated, using the last equation, from the graph of thickness change $\Sigma\Delta x_i$ against centre tension change $\Sigma\Delta\sigma_i$ when $\Delta\sigma_n/\Delta x_n$ becomes $(d\sigma/dx)_n$, the gradient of the curve.

5. Equipment and measurements

5.1. Centre tension

In the present work, a He-Ne gas laser with an output power of 5 mW was used. This gave visible fringes which considerably facilitated alignment of the beam along the centre plane of the plate.

Stress gradients reduce the sharpness of the fringes because of the varying stress across the beam width. To minimize this effect a lens was used to reduce the beam width to about 0.2 mm. Immersion oil of suitable refractive index was used to introduce the beam into the edge of the specimens. The specimen dimensions were, for Corning 0319 material, 125 mm \times 25 mm \times

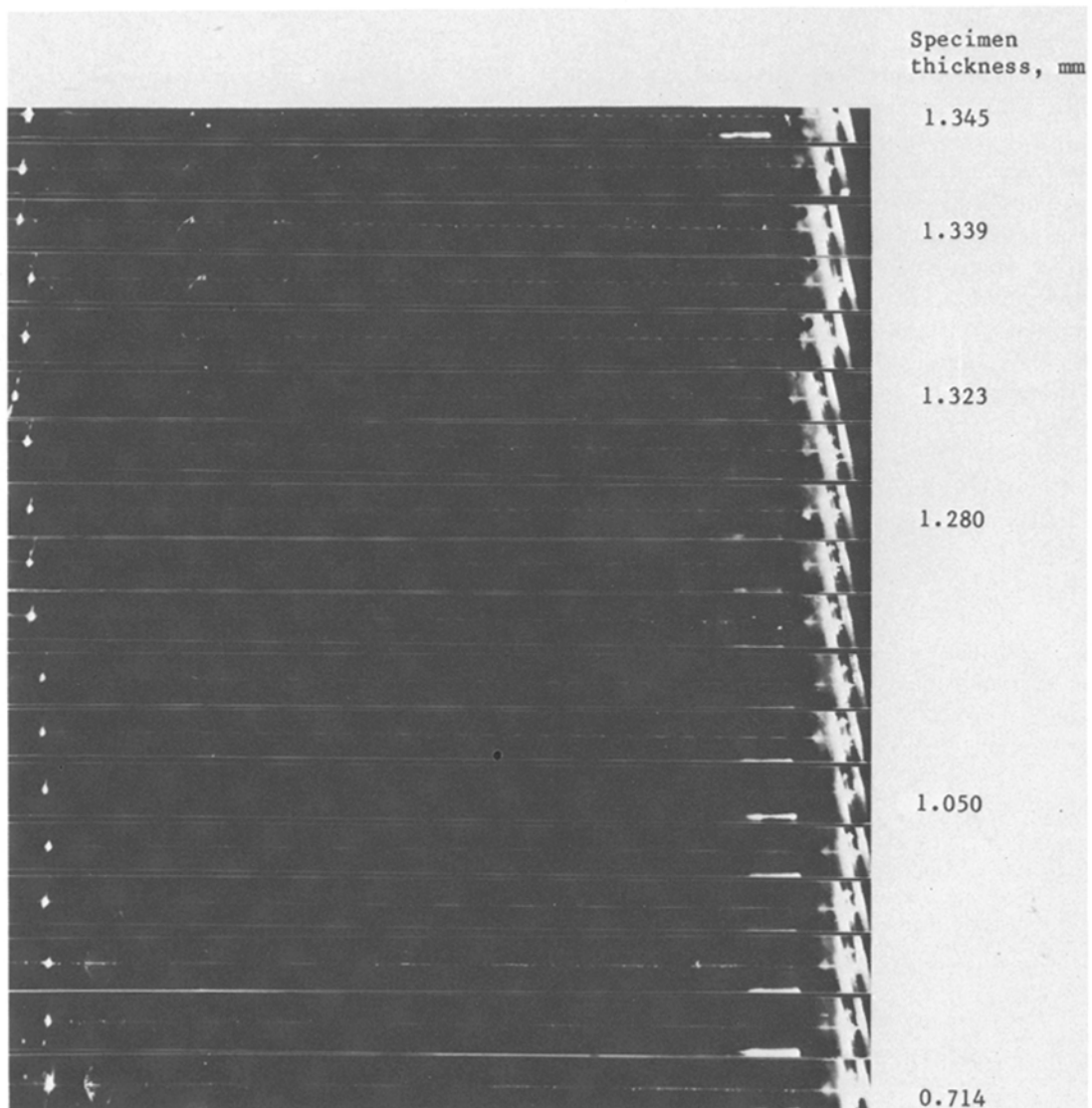


Figure 4 Fringe patterns for a specimen of 0319 glass showing increase of fringe separation with decrease in thickness.

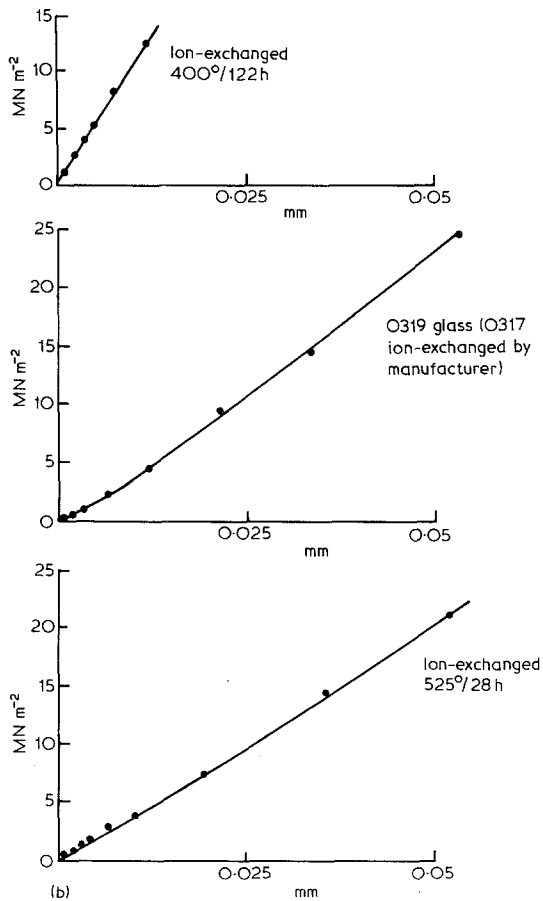
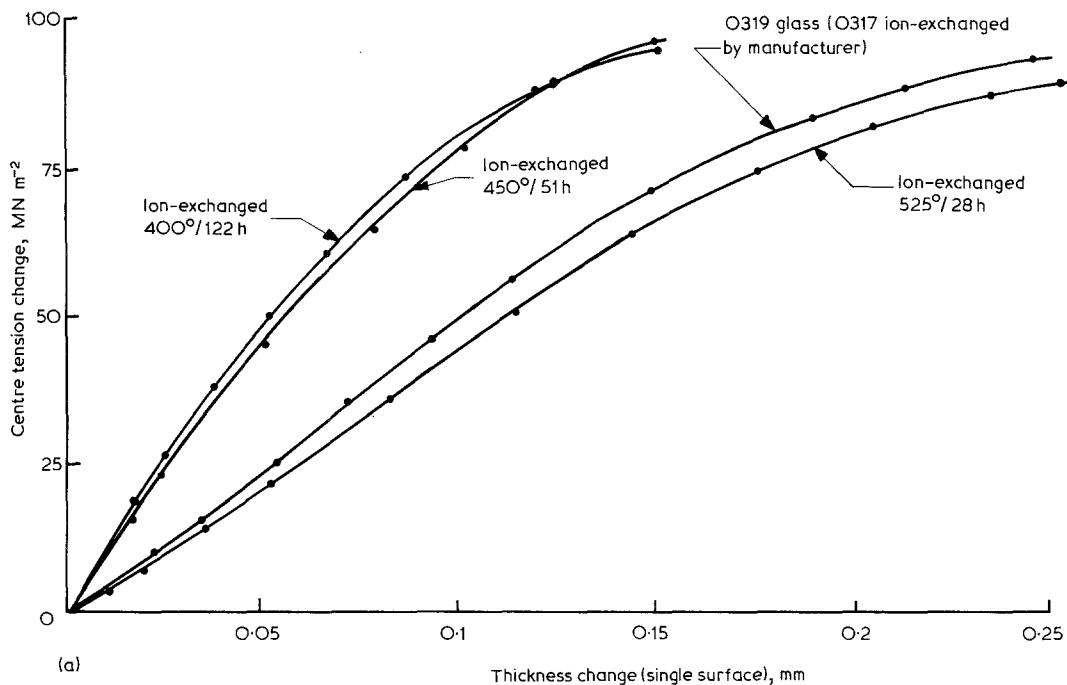


Figure 5 (a) Change of centre tension with thickness for Corning 0317 glass ion-exchanged in KNO_3 under varying conditions. (b) Details from (a) around origin.

1.25 mm (nominal) and for the other materials 65 mm × 15 mm × 1.25 mm (nominal).

With the above arrangement, clear patterns of about 50 to 60 fringes, stretching from end to end of 125 mm long specimens, were obtained during the initial stages of the profile determination. Fig. 4 shows a typical example. Fringe separations were measured from enlargements (× 4) of photographs of the fringe patterns.

Centre tension values were calculated from fringe separations using the Corning value of 2.75 Brewsters for the stress optical coefficient of both 0319 glass and the locally-treated materials.

5.2. Thickness and thickness change

Specimens were etched in a dilute mixture of HF + H₂SO₄. The initial thickness of each specimen was measured with a micrometer and thickness changes after etching were determined from the specimen weight change and micrometer measurements.

The thickness decrements produced by the etching treatment were, during the early stages of the profile determinations, too small for accurate measurement with a micrometer. Weight changes were recorded until the thickness change reached a value sufficient for accurate measurement, i.e. about 0.13 mm. The calibration of weight change against thickness change thereby obtained was used to obtain the smaller thickness changes from the corresponding weight changes.

Specimen weights ranged from about 3 g to about 10 g. Weight changes were recorded to ± 0.0001 g, the smallest weight change for etched layers being about 0.010 g.

5.3. Materials tested

The above techniques have been extensively used to determine stress profiles in plates of chemically strengthened glasses. Some specimen results are presented here. These consist of Corning 0319 glass and three specimens prepared by ion-exchanging its precursor glass (Corning 0317) in KNO₃.

The salt bath temperatures used were 400, 450 and 525° C and the associated immersion times, selected to give similar values of centre tension to 0319 glass, were 122, 51 and 28 h, respectively.

6. Results

The changes in centre tension with change in 2986

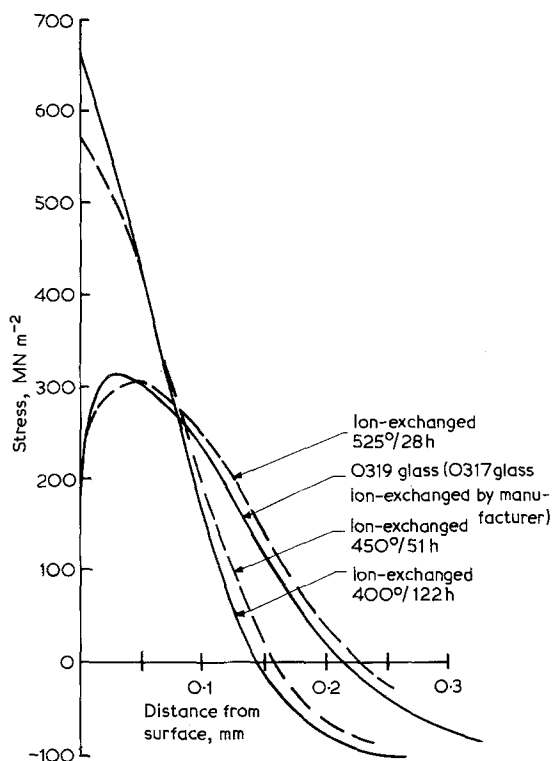


Figure 6 Stress profiles for 0317 glass ion-exchanged in KNO₃ under varying conditions.

thickness are shown in Fig. 5, for 0319 glass and 0317 glass ion-exchanged under varying conditions. The stress profiles derived from these curves are shown in Fig. 6. These show a fall-off in the peak compressive stress with increasing temperature of ion-exchange. This can be attributed to viscous relaxation of the stress in the salt bath: at the higher temperature viscous relaxation is becoming more significant even though the time in the salt bath is shorter.

The profile for 0319 glass shows close agreement with that of 0317 glass ion-exchanged in KNO₃ at 525° C for 28 h, and it is presumed, therefore, that this treatment condition is close to that used by Corning in the production of 0319.

After allowance is made for the effect of different specimen thicknesses on the value of centre tension, the measured profile for 0319 glass is similar to that published by Stephens and Beauchamp [8]. The main difference is that their profile, derived from measurements of length change versus thickness change, shows the peak stress at the surface whereas the present measurements indicate the peak stress to be just under the surface; profile determinations on several samples

from the same batch of ion-exchanged 0319 glass all agree very closely with the profile shown in Fig. 6.

7. Discussion

The present method seems to compare favourably with other techniques for profile determination. However, in common with these other techniques it has associated errors.

Equation 3, the expression from which the stress profile is determined, include the derivative $d\sigma/dx$. Accurate estimates of this term impose stringent requirements on the accuracy of measurement of stress changes and thickness change. The errors in the measurement of these two variables are discussed below.

7.1. Errors in centre tension measurements

The accuracy of the centre tension measurement depends on the length of specimen available for fringe measurements and the sharpness of the fringes. Both conditions are most favourable during the initial stages of the profile determination; the fringes are closely spaced, the position of the centre of a fringe is easily defined and the thickness of the specimen is uniform over a large proportion of its length. For centre tension changes of about 7 MN m^{-2} (1000 psi), the comparative accuracy is estimated to be $\pm 0.33 \text{ MN m}^{-2}$ (50 psi). During the later stages of the determination, the fringes broaden and the uncertainty in the position of the fringe centre increases; also the cumulative effect of differences in etch rates between the middle regions and the end regions of the plates becomes noticeable and fringe measurements are necessarily confined to the middle portion of the plate where the thickness is still uniform to $\pm 0.0025 \text{ mm}$ (0.0001 in.). During these later measurements, when the centre tension has dropped – by perhaps 90 MN m^{-2} (13 000 psi) for the specimens used here – the accuracy is estimated to be $\pm 1.5 \text{ MN m}^{-2}$ (200 psi).

7.2. Errors in thickness change measurements

Section 5.2 describes how during the early stages of the profile determination, thickness decrements were calculated from weight changes.

In practice there is an inherent error in this calculation since the density at the surface, where the K ion concentration is greatest, is higher than regions further into the body of the specimen. The

magnitude of the error depends on the thickness of layer used in the calibration of weight change against thickness change, but an estimate can be made of the maximum probable error from the composition and exchange characteristics of the glass. In the current work, it was found that approximately 90% of the surface Na is replaced by K from the bath, and from the composition of the glass and assuming no surface dilation, this should result in an increase in density of about 5%. If surface dilation, based on Hale's analysis [9], is included in the calculation the density increase should be about 3%. This is the maximum difference in density between the unchanged base glass and the 90% ion exchanged surface glass. The average density of the compressive layer will depend on the K ion concentration profile, which in this glass follows the complementary error function (i.e. diffusion is concentration independent [10]). If a linear concentration profile is assumed, then the average density of the compression layer is about $1\frac{1}{2}\%$ higher than the base glass and the error is comparable to that of the weight change measurement and also to that in the micrometer measurement used in calibration.

Where the calibration layer thickness is less than that of the compressive layer, the density error will be less, and the same applies to a glass in which viscous flow has led to some relaxation of the stress, and therefore of its density increase resulting from ion-exchange.

8. Operating experience

The above method has been found particularly useful for measuring profiles in thin specimens of chemically strengthened glasses where stress gradients are high and where refractive effects near the surface introduce uncertainties in conventional photo-elastic examination. The minimum thickness of specimen is governed only by the ratio of the effective width of the laser beam to the width of the uniform stress region in the tensile zone. Ideally, for sharp fringes, the scattered light should come from regions of uniform stress.

Although the method is destructive and requires flat plate test specimens, provided that these are ion-exchanged at the same time and under the same conditions, and have identical thickness, they should be adequately representative of components in which the curvature is large compared to their thicknesses.

The results described above represent just a few

of the stress profile determinations carried out using the technique: it has been used extensively with chemically strengthened specimens that have been subjected to high-temperature ageing treatments which accelerate ion diffusion down the concentration gradient and consequently modify the stress profiles. In all cases the results were consistent with the new ion concentration profile and the severity of the ageing treatment [10].

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