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2. Investigation of Stresses at the Surface of Chemically-Toughened Sheet Glass*

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A theoretical investigation was made to explain why interference fringes appear at the boundary of total reflection when the light is reflected at a surface having a modified layer. The result shows that the stress-induced birefringence and the approximate thickness of a stressed layer are measured by surface refractometry, considering the shift of the fringes due to the change of the polarization direction of the incident light and the number of fringes.

Surface stresses of chemically-toughened glass sheets were measured with this method under various conditions of ion-exchange treatments. The measured values are related to the tensile strength of toughened samples.

1 Introduction

In recent years a chemical method of strengthening glass by ion exchange below the strain point, also known as "crowding" or "ion stuffing", has recieved a great deal of attention and has found some practical applications.(1),(2) In this procedure the surface compression arises by stuffing larger ions into sites formerly occupied by smaller ions. The stress profile thus produced is distinguishable from that of thermal tempering by the relatively thin compressed layer and the very high stresses.

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It is possible to determine a stress profile of this kind by means of a polarization microscope with the aid of a Berek compensator, when one observes a thin slice, 200 μ to 300 μ thick, cut perpendicular to the outside surface of the sample. However, this method has some shortcomings. It is laborious, it is not accurate in determining the real surface stress, especially when the stressed layer is extremely thin, and worst of all it can not be applied in a nondestructive manner.

The nondestructive measurement of surface stress in glass is accomplished by surface refractometry. An instrument wellsuited for this measurement, a type of critical angle refractometers, has been developed by Ansevin.(3) The magnitude of the surface stress σ is determined from the relationship:

$$\sigma=C(n_1-n_2)$$

where C is the stress-optic coefficient, and n_1 and n_2 are the refractive indices of the glass surface for rays polarized in planes parallel to and perpendicular to the surface, respectively. The difference is measured by observing the corresponding angles of total reflection for both polarized components.

However, the nature of the boundary of total reflection is influenced by the presence of index variation at the glass surface. When a chemically-toughened glass surface is viewed with the refractometer, interference fringes appear near the light-dark boundary.

The interference phenomenon at the boundary of total reflection was first treated by Kossel.(4) He observed interference fringes near the total reflection boundary, when the light is reflected at the surface of a glass plate coated with an evaporated film having a smaller refractive index than that of the glass plate. The refractive index of the compressed layer of a chemically-toughened glass is always higher than that of the interior, because the layer holds larger ions stuffed. Therefore, the index system of prism-layer-glass in our case is identical with the system of glass-film-air treated by Kossel. In the present study the theory of Kossel is further developed in order to show how to evaluate surface stresses of chemically-toughened glass from the fringe patterns. Surface stresses of sheet glass toughened under various ion-exchange conditions were measured with this method and compared with the results of strength tests.

2 Theory

First, let us consider a case where a prism of index n_p is placed on a glass plate of index n_g having a homogeneous surface layer of thickness d and index n_s (**Fig. 1**). φ is the angle of incidence, φ_T^{ps} and φ_T^{pg} are the critical angles for the prism-layer interface and the prism-glass interface, respectively. When n_s is larger than n_g, φ_T^{ps} becomes larger than φ_T^{pg}. The intensity of the reflected light I_R is given by:

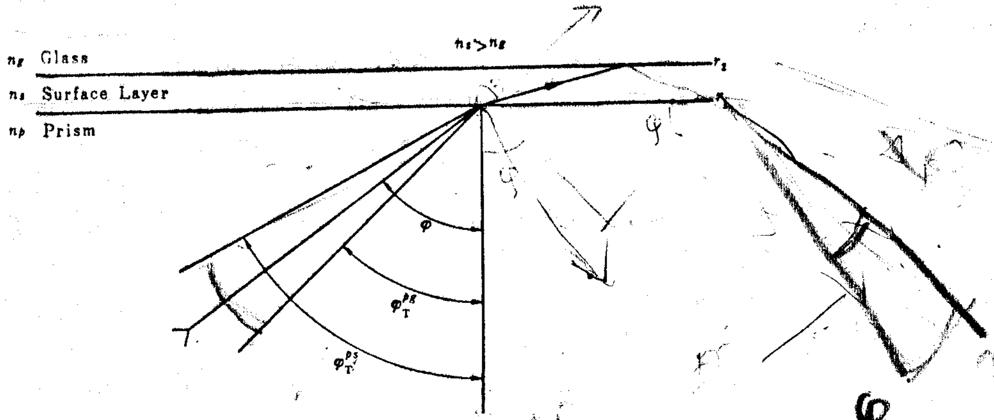

Fig. 1 Total reflection in the system of glass-surface layer-prism.

$$I_R=\frac{r_1^2+r_2^2+2\,r_1r_2\cos\Delta}{1+r_1^2r_2^2+2\,r_1r_2\cos\Delta} \tag{1}$$

$$\Delta=\frac{4\pi}{\lambda}n_s d\sqrt{n_s^2-n_g^2\sin^2\varphi}+\beta \tag{2}$$

where r_1 and r_2 are the amplitude reflectivities at the prism-layer and layer-glass interfaces, λ is the wave-length of light, β is the phase change by total reflection.

If the angle of incidence φ exceeds φ_T^{pg}, the reflectivity r_2 becomes 1, and then $I_R=1$ regardless of φ. Hence the boundary of the total reflection observed is to be associated with the critical angle φ_T^{pg}. It is concluded that no information concerning the surface layer is available from the light-dark boundary.

In order to explain the appearance of the interference fringes, Kossel assumes that in practice r_2 never reaches 1 because there is loss of light due to absorption or scattering in the surface layer. Hence the intensity of the reflected light varies with the value of $\cos\Delta$, that is, with the incident angle, and has maxima and minima. When $\cos\Delta=-1$, the intensity has a minimum value and a dark fringe appears. The position of fringes is given by the relation:

$$2\,d\sqrt{n_s^2-n_g^2\sin^2\varphi}=\frac{k\lambda}{2},\quad k=1,\ 3,\ 5,\ \cdots \tag{3}$$

if β is neglected as a small value*.

The ion-stuffed layer of the chemically-toughened glass has not a uniform refractive index. The index varies gradually from the surface value to the value of the bulk glass with the concentration of stuffed ions. The theory should be extended to the case of inhomogeneous layers. Then, the interference condition becomes:

$$2\int_0^d\sqrt{n_s^2(x)-n_g^2\sin^2\varphi}\,dx=\frac{k\lambda}{2},\quad k=1,\ 3,\ 5,\ \cdots \tag{4}$$

where n_s is a function of the depth from the surface x. For convenience, we assume in further calculations that the index is a linear function of x, as shown in **Fig. 2**, where d denotes the thickness of the surface layer. The left hand side of Eq. (4) is numerically calculated as a function of the incident angle, if we assign a set of values n_p, n_s and n_g**. An example of calculated results is illustrated in **Fig. 3**.

The patterns of interference fringes can be constructed from these theoretical curves. Examples for layers of thickness 5 μ, 25 μ and 50 μ are presented in **Fig. 4**. The light-dark boundary is associated with the critical angle φ_T^{gs} and gives the bulk index of the glass. The first fringe at the top is related to the

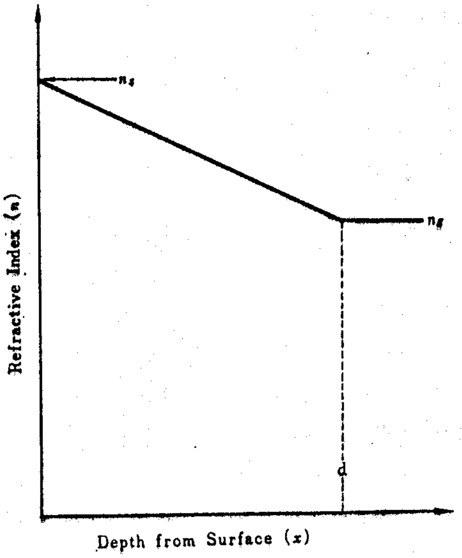

Fig. 2 Inhomogeneous layer model.

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* In case of inhomogeneous layers, β becomes exactly 0.

** The value of n_s is determined by experiments. In practice n_s varies with the progress of ion-exchange treatments.

interference condition $k=1$, the next $k=3$ and then successively $k=5$, 7 and so on. When the index of the layer is uniform, the fringes become denser at the top and seems to converge to the boundary associated with the critical angle φ_T^{ps}. On the contrary, the interval of the fringes increase with parting from the dark-bright boundary in case of the inhomogeneous layer. The exact value of φ_T^{ps}

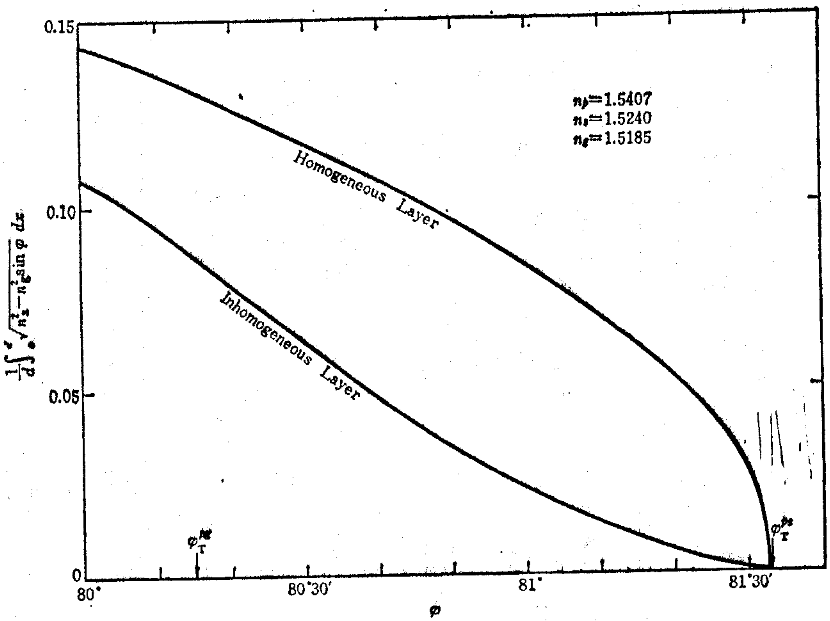
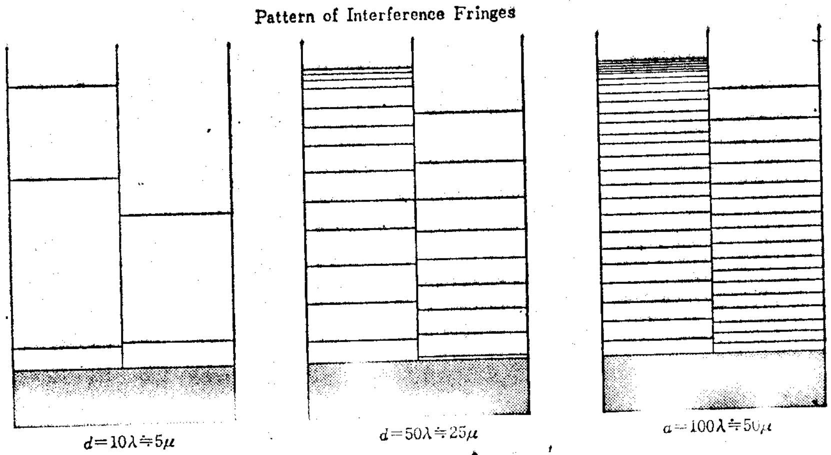


Fig. 3 Numerical values of the optical path differences as a function of the incident angle.



φ_{ps}

1
3
5

φ_{pg}

Left : Homogeneous Layer
Right : Inhomogeneous Layer

Fig. 4 Patterns of interference fringes for homogeneous and inhomogeneous layers.

must be sought at the point which corresponds to the condition $k=0$.

If the direction of polarization is converted, all the fringes are shifted because of birefringence. The stress values associated with this shift are plotted against k number, as illustrated in **Fig. 5**. The real stress value at the surface is found

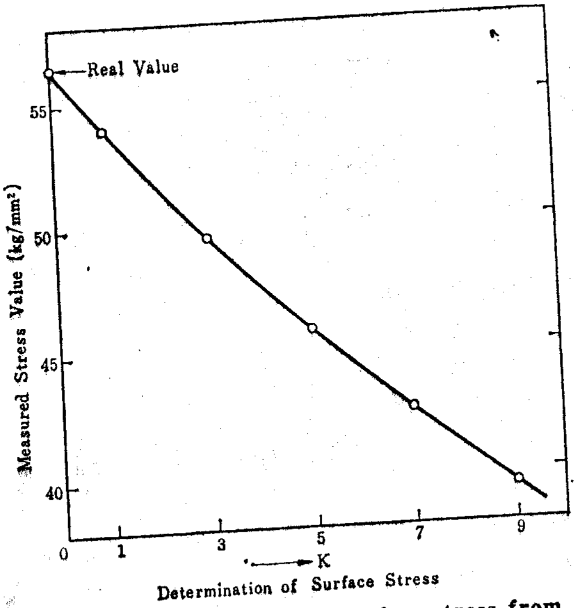


Fig. 5 **Determination of surface stress from the fringe shifts observed.**

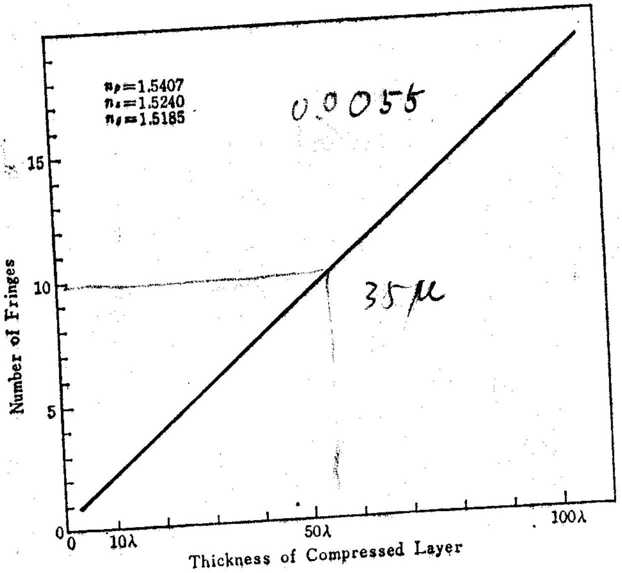


Fig. 6 **Relation between the layer thickness and the number of fringes.**

on the point where the extrapolated curve crosses the axis of $k=0$. Therefore, at least the measurement for two fringes is necessary for estimating the surface stress, preferably the measurement for three or more. The accuracy of the measurement will be raised with the increase of the number of fringes.

If we give the values of n_p, n_g and n_s as a function of depth from the surface, the relation between the number of fringes and the thickness of the compressed layer can be obtained, as shown in **Fig. 6**. By the use of this curve we can estimate approximate thickness of compressed layer from the number of fringes observed in a nondestructive manner. Adversely, the pattern of fringes observed will give us some knowledge of the distribution of refractive indices or stresses at the surface layer.

3 Experimental Results and Discussion

Pieces of ordinary sheet glass were dipped in molten potassium nitrate for various periods at fixed temperatures. After the ion-exchange treatment the surface of specimens were examined by a surface refractometer. The pair of photographs in **Fig. 7** shows a fringe pattern observed for a specimen treated for

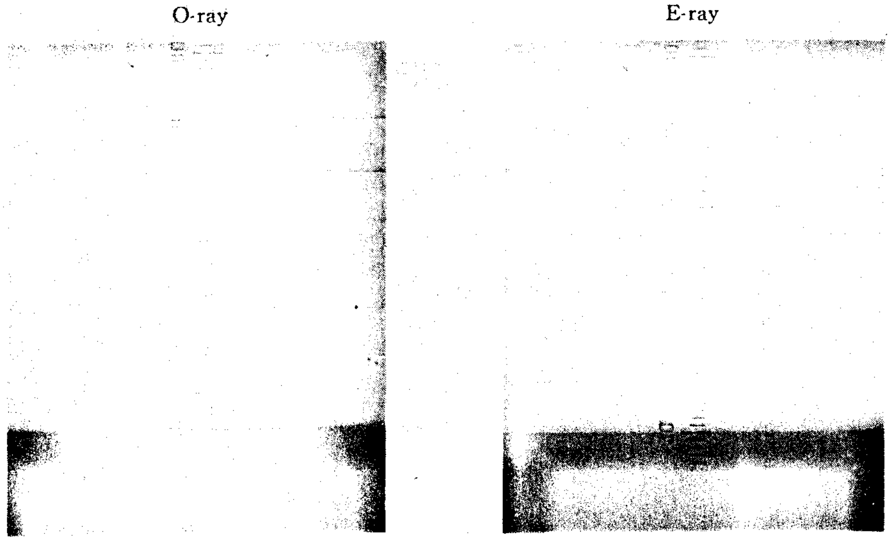

O-ray E-ray

Fig. 7 Images of interference fringes for a sample treated for 10 hours at 460°C in potassium nitrate.

10 hours at 460°C. The image on the left is formed by the light polarized parallel to the sample surface and the other is formed by the light polarized perpendicular to the sample surface. As theory predicted, the position of the light-dark boundary does not change by converting the polarization direction of the incident light, but the shift of the interference fringes are observed.

The number of fringes increases with the progress of the ion-exchange treatment. The thickness of the compressed layer obtained by use of the relation illustrated in **Fig. 6** was plotted against the treating time as shown in **Fig. 8**. The thickness is nearly proportional to the square root of the treating time as expected by the diffusion theory.

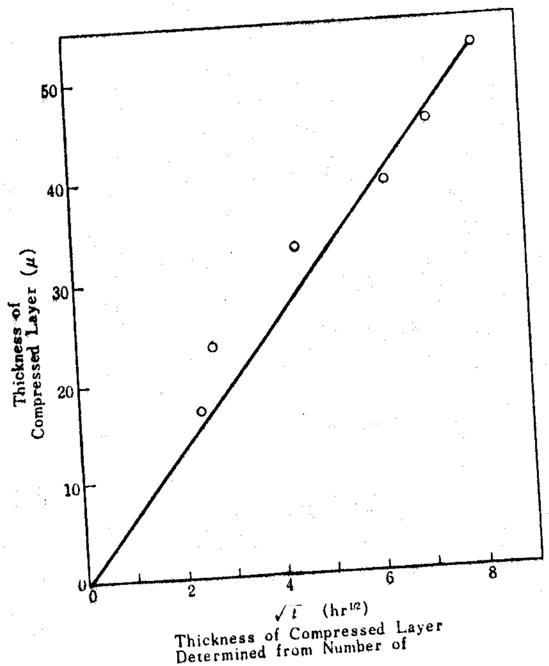


Fig. 8 Thickness of compressed layer determined from number of fringes.

Figure 9 shows how the surface compression measured varies with the treating time. The stress decreases monotonically with the progress of the ion-exchange procedure. This means that the stress arises at the very beginning of the treat ment and gradually relaxes by reason of viscous deformation.

The mechanical strength of the samples, however, does not behave in th same manner. We measured the modulus of rupture for specimens damaged b indenting a Vickers diamond under a load of 1000 g before or after ion-exchang and arrives the maximum before the thickness of compressed layer exceeds the depth of the crack. Thereafter the strength decreases gradually corresponding to the decrease of the surface stress. On the other side, in case the sample is damaged after ion-exchange treatment, the enough thickness of compressed layer is required for preventing the drop of the strength.

4 Conclusion

The interference phenomenon at the boundary of total reflection was theoretically discussed and applied to the measurement of surface stress of chemically-toughened sheet glass. The interference fringe patterns gives us information upon the surface layer whose thickness is fairly larger than the wave length of the light. Supposedly, the application of this phenomenon to investigations of surface films or layers will not be limited to the case of chemical strengthening.

Acknowledgements

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