PAPER 40 (SESSION III)

# Investigation of the influence of water vapour on crack velocities in glass by ultrasonic fractography

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#### Summary

Velocities of cracks in glass can be accurately determined by the method of ultrasonic fractography, the modulation of a fracture surface by continuous ultrasonic waves. This method, which is briefly described, is used here to measure crack velocities in the range 5 m/s to 1500 m/s in plate glass at room temperature as a function of specific fracture energy G and relative humidity. In particular  $G_0$ , the value G at which a crack becomes unstable is discussed: the corresponding initial crack front can easily be located on the surface due to the accumulation of lines produced there by the ultrasonic modulation. This value  $G_0$  is a function of relative humidity and lies in the range G to G to G to G is about G to G is about G in the range G to G in which a crack in simple tension branches. G is about G is compared with the value G which can be estimated from the so-called mirror-constant. For optical glasses the characteristic values G for normal room conditions and G for dry air are measured and found to be almost linearly dependent.

### Introduction

It is well known that the ultimate strength and the crack velocity at the beginning of fracture are complicated functions of the stress or of the elastic energy release G, of the water vapour concentration of the environment and of temperature. Recently experimental studies of this slow, socalled 'thermal' stage of fracture have been made by Irwin [1], Wiederhorn [2] and Klemm, Schönert, Umhauer [3]. The region of crack propagation which can be called the 'athermal' catastrophic stage of crack motion, however, has not yet been investigated experimentally in detail. Smekal [4] found by analysis of Wallner-lines that the crack velocity in rods of an optical glass in water reaches its maximum much more slowly than in air. However, this method cannot be used to measure crack velocities below about  $\frac{1}{4}$  of the maximum velocity, i.e. 400 m/s in sheet or plate glass. Therefore, to investigate the influence of water vapour on crack velocities between 5 m/s and the maximum velocity, ultrasonic fractography, a method which has been developed by Kerkhof and his co-workers since 1952 [5], was applied. In particular, we have studied the propagation of cracks in plates of simple plate glass under tension and have measured G-values for the onset of unstable, athermal fracture and for the beginning of fracture branching. We have also measured the G-value for the onset of unstable fracture and for the beginning of fracture roughness for some optical glasses.

## The method of ultrasonic fractography

A guiding principle of brittle fracture is that a crack always propagates perpendicularly to the instantaneous direction of the maximum principal tensile stress  $\sigma_I$ . We can symbolize this law in the following way:

$$\overrightarrow{v_f} \perp \sigma_I$$

This law obviously holds if a test plate of glass which is homogenous and isotropic is subjected to a simple uniaxial tensile stress *p*. In this case the fracture surface is a simple plane.

If we now apply ultrasonic waves in addition to the static tension p, the resulting principal tensile stress will change its direction in any part of the specimen as a function of time. Consequently, the presence of mechanical waves will cause undulation of the fracture surface (Fig. 1). Looking from above the fracture surface will contain shallow ripples (Fig. 2 and 3), whose separation will depend upon the ultrasonic frequency and the crack velocity.

Theoretically the best condition for producing these ripples are (Küppers [6]) if ultrasonic shear waves are propagated in a direction parallel to the static tension p and with displacement vector parallel to the direction of crack propagation (Fig. 1). In this case the fracture velocity  $v_f$  is simply equal to the separation of the ultrasonic ripples  $\lambda_f$  multiplied by the ultrasonic frequency  $\nu$ :

$$V_f = \lambda_f$$
.  $\nu$  (1)

By applying a frequency of 1 Mc/s and photographing the ultrasonic ripples under a microscope it is possible to measure fracture velocities as low as  $5 \, \text{m/s}$ . At this velocity the fracture is expected to have reached the athermal stage.

The method of ultrasonic fractography obviously enables one to determine the rate of development of fracture even in a complicated three-dimensional case, or in an opaque material, provided that the fracture surfaces are sufficiently smooth.

On such a surface (Fig. 2) the initial crack length  $a_0$  can easily be identified from the accumulation of ultrasonic lines that takes place there, and from it the critical value of the elastic energy release  $G_0$  can be calculated using the formula (Irwin [7]).

$$G_0 = 4.00 \frac{p^2 a_0}{E}$$
 (2)

for  $a_0 \leqslant b$  (E = Young's modulus). In order to calculate the G-values during further propagation of the crack it is only necessary to put the respective lengths a into this formula.

For higher values of a/b equation (2) must be modified; for this purpose we used the numerical calculations of Gross, Srawley, Brown [8].

#### Measurements on plate glass

According to the theory of N. F. Mott [9] and Dulaney and Brace [10] the development of crack velocity  $v_f$  as a function of the energy release G should be given by the equation

$$v_{f,th} = v_{f,max} \quad (1 - G_0/G) \tag{3}$$

where  $v_{f,max}$  is the theoretical maximum crack velocity. The experimental value of this maximum velocity

$$v_{f, \text{max}} = 1520 \text{ m/s}$$

is in good agreement with that predicted by Kerkhof [11] from the formula

$$V_{f,\max} \quad 3 = 2\sqrt{\alpha/\rho r} \tag{4}$$

where  $\alpha$  is the free specific surface energy of glass at the transformation temperature,  $\rho$  density and  $\bar{r}$  the mean ionic distance. Using the values  $\alpha = 305 \text{ erg/cm}^2$ ,  $\rho = 2.52 \text{ g/cm}^3$ ,  $\bar{r} = 2.00 \cdot 10^{-8} \text{ cm}$ , this gives

$$v_{f, max} = 1560 \text{ m/s}.$$

Formula (3) can be compared with typical measurements of crack velocities in plate glass (dimensions  $250 \times 50 \times 4$  mm) in dry air of ca. 5% relative humidity at room temperature (hereafter called rh). Since a/b < 0.1 in the region concerned, formula (2) can be used to calculate G with sufficient accuracy. It appears that, even in dry air, the experimental values of  $v_f$  are less than the theoretical values during the first part of the fracture and are greater during the second part.

It must be kept in mind that for all parts of the curves in Fig. 4 the static values of G were used. If one supposes that the dynamic values of G are lower than the static ones for high crack velocities, the experimental curve would presumably cross the theoretical curve at a lower value of G. At any rate our experiments, at least those at 5% rh, indicated that the maximum velocity is reached much earlier than is predicted theoretically.

The first part of the function  $v_f(G)$  was investigated experimentally in detail for various relative humidities at room temperature. The results in the region  $5 \text{ m/s} \leqslant v_f \leqslant 600 \text{ m/s}$  for three quite different humidities (ca. 5%, 48% and ca. 95% rh) are shown in Fig. 5. Although in these experiments the relative crack lengths were higher than before, a/b was still less than 0·5, so that the corrections of Gross, Srawley and Brown mentioned above could be applied. The curves give the positions of definite values of  $v_f$  for mean values of G at the respective relative humidities. The standard deviations are indicated by horizontal lines. The number of experiments carried out in each case was 12 at ca. 5% rh, 6 at 48% rh and 17 at ca. 95% rh.

# Crack velocities in glass by ultrasonic fractography

It is interesting to note that the three experimental curves are all less steep than the theoretical curve calculated for  $G_0 = 8600$ , corresponding to the initial point of the experimental curve for ca. 5% rh. It may be that the corrections of Gross, Srawley and Brown are not quite correct for higher velocities (see below). The values of G would then be too high. At any rate, no essential difference would be expected up to 100 m/s.

But more striking is that the values of  $G_0$  are not a monotonic function of the relative humidity. The results of a more detailed study of this function are shown in Fig. 6.

The decrease of  $G_{\rm o}$  from 8600 erg/cm² at ca. 5% rh to 6500 erg/cm² at about 40% rh can be explained by the corrosive attack of water vapour on the crack tip, which lowers the strength of the glass. What is surprising is the increase of  $G_{\rm o}$  with increasing relative humidity above about 40%.

We suppose that in this region we must take into account the influence of adhesion between the two surfaces of the opening crack, given that the separation between them is small enough. It is known (Davies, Rideal [12]) that the adhesion between two glass surfaces is negligible in dry air, but that in a humid atmosphere the adhesion increases with humidity towards a limit which is reached at saturation vapour pressures. This limit is also the adhesion in the presence of a small amount of liquid water between the surfaces, and can reach a value of about  $30 \ kp/cm^2$ . This compares with the tensile stresses in our experiments which were between 50 and 90 kp/cm<sup>2</sup>.

A reason for expecting small amounts of water between the fracture surfaces even at relative humidities smaller than about 95% is that of capillary condensation, a process governed by the Kelvin equation

$$\ln (p_0/p') = 2 a_w V/rRT$$

where  $p_0$  = water vapour pressure over a flat surface,

p' =water vapour over water of curvature r,

 $\alpha_{w}$  = surface tension of water,

V = molar volume of water.R = gas constant,

T = absolute temperature.

From this equation one obtains a meniscus radius r of about  $10^{-6}~\mathrm{mm}$  for relative humidities of less than about 35%. The spacing between water molecules in the liquid state is of the order of  $3 \cdot 10^{-7}$  mm. One can conclude that at vapour concentrations below about 35% liquid water is not present at the crack tip, and that above 35% capillary condensation becomes more likely with increasing relative humidity (Wiederhorn [2]). The increase of  $G_{\mathrm{o}}$  in glass begins at about the same point. Adhesion could also account for the delay in the development of fracture velocity at high relative humidities (Fig. 5).

Another effect which could produce liquid water between the crack surfaces, is that between fast opening crack surfaces there will be a lower pressure, where gas can expand adiabatically and can therefore condense.

A further characteristic of crack propagation is branching. As it is possible to measure exactly the length of a crack up to the point of branching, a corresponding value of the specific fracture energy,  $G_B$ , can be calculated using the simple equation (2). The corrections for finite plate width mentioned above are not necessary provided that the values of G calculated for static cracks are also applicable to dynamic problems. It can be easily shown that the correction becomes less necessary with increasing ratio  $v_f/v_i$  ( $v_i$  = velocity of longitudinal waves) and, for example, for  $v_f = 1470$  m/s in plate glass ( $v_f/v_1 = \frac{1}{4}$ ), the uncorrected formula (2) can be used for  $a_B/b \le 0.44$ . In all the experiments evaluated,  $a_B/b \le 0.2$ .

In Table 1 the results of the measurements of  $G_B$  are compared with the respective values of  $G_{\text{o}}$  for three different relative humidities, of which the value of ca. 40% corresponds approximately to the minimum of the curve in Fig. 6. It is interesting to note that the values of  $G_{B}$  also depend slightly on the relative humidity even when the corresponding crack lengths  $a_{\emph{B}}$  are measured in the centre of the fracture surface (see the numbers in brackets). However these differences only amount to the sum of the respective standard deviations. It should be pointed out that for simplicity the values of G for the centre of the fracture surface were also calculated according to equation (2). The constant factor  $1-\mu^2$  ( $\mu = Poisson$ 's Ratio), which would have been necessary for the condition of plane strain in the interior of the plate, has been omitted.

It is interesting to compare this value  $G_B$  with the specific fracture energy  $G_{M\!\!\!M}$  at the rim of the fracture mirror in tensile experiments on rods of a similar glass (AR-glass from Ruhrglaswerke). Photographs of such fracture mirrors are reproduced from Sommer [13] in Fig. 7. According to a formula of Shand [14],

$$G_{M} = \frac{4}{\pi} \frac{1 - \mu^{2}}{E} p^{2} R_{M} \left( 1 - 0.81 \frac{R}{D} M \right)^{2}$$

where p is the tensile stress,  $R_{M}$  the radius of the fracture mirror and Dthe diameter of the rod. Sommer found as the mean value of 11 experiments carried out in oil

$$G_{M} = 6.01 \times 10^{4} \text{ erg/cm}^{2} \pm 17\%$$

which is about half the value of the mean of the values of  $G_{\mathcal{B}}$  in brackets multiplied by  $(1-\mu^2)$ :

$$G_B (1 - \mu^2) = 14.3 \times 10^4 \text{ erg/cm}^2$$

with  $\mu = 0.23$ . This corresponds to the fact that in tensile experiments on plate glass the visible roughness of the fracture also begins at  $G\approx 5^{\circ}5\times 10^{4}$ erg/cm² (Kerkhof [15]).

## Measurements on optical glasses

For 15 optical glasses (of the Jenaer Glaswerke Schott u. Gen., Mainz) the characteristic values  $G_{\rm o}$  in dry air (ca. 5% rh) and  $G_{\rm M}$  in room atmosphere have been measured. The chemical compositions of these glasses which had also been used for the measurement of the maximum fracture velocity by Schardin, Mücke and Struth [16] and Kerkhof [17], are given in Table 2. The critical energy release  $G_n$  was measured on glass plates  $(180 \times 33 \times 4 \text{ mm})$  by the ultrasonic method mentioned above. Values of  $G_0$ were calculated by means of equation (2) using, when necessary, the corrections of Gross, Srawley and Brown [8].  $G_M$  was found in bending experiments on glass rods 100 mm long and 11 mm in diameter. For evaluation a similar equation to equation (5) was used, taking as correction  $(1-1.81 (R/D))^2$  instead of  $(1-0.81 (R/D))^2$  (Kerkhof [17]). The relative standard deviations were between 7% and 17%. In Fig. 8  $G_M$  is plotted versus  $G_0$  for the 15 optical glasses. Also plotted are the measurements of Sommer for  $G_M$  on AR-glass compared with the value of  $G_0$  for plate glass in dry air from Table 1. There is obviously an almost linear relation between  $G_{\mathbf{M}}$  and  $G_{\mathbf{0}}$ :

 $G_{\mathbf{M}} \sim 5G_{\mathbf{0}}$ .

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Table 1
Energy rates in plate glass

humi di ty	G <sub>0</sub> [erg/cm <sup>2</sup> ]	n <sub>0</sub>	$V_0$	$G_B \left[ erg/cm^2 \right]$	$n_B$	$V_{B}$	$G_B/G_0$
~ 5%	8. 6×103	13	10%	147×10 <sup>3</sup> (154×10 <sup>3</sup> )	18	10% (10%)	17 (18)
~40%	6· 4×10 <sup>3</sup>	12	9%	126×10 <sup>3</sup> (134×10 <sup>3</sup> )	10	17% (16%)	20 (21)
~95%	11·7×10³	32	22%	167×10 <sup>3</sup> (161×10 <sup>3</sup> )	10	8% (8%)	14 (14)

 $G_0$ : for onset of unstable fracture;  $G_B$ : for crack branching; n: number of experiments; V: relative standard deviation.

Measurements are in general taken at the rim of the fracture surfaces; numbers in brackets refer to measurements in the centre.

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Table 2
Chemical synthetic composition of the optical glasses investigated (in Wt%)

Nr.	SiO <sub>2</sub>	$B_2O_3$	A12O3	Na₂O	$K_2O$	РЬО	BaO	ZnO	CaO	Sb <sub>2</sub> C	) <sub>3</sub> As;
1	61.5			4.6	8.2	25.4					
3	53.7			2.5	7.9	35.6					0.3
8	45.6			3.6	100 (5)						0.8
11	40.9			2.5	5.0	45.2					0.6
14	34.3				4.0	52.3					0.3
16	30.8			1.5	2.5	61.4					0.3
17	27.1			0.9	2.0	66.0					0.3
17a	18.7	1.0		0.5	1.0	71.4					
9	64.1				0.6	78.7				1.0	
20	69.5	12.0	4.0	7.8				11-1		0-6	0.4
22	48.7	11.5		10.3	7.0		1.5	_	0-2	0.0	0-4
4		19.8	2.5	2.1	6-1		-		0-2	10.0	
	65.0	3.5		5.8	13.3	1.0		8.2	2 -	19.8	1.0
5	50•2	5.9		4.0	5.0	2.2	19.7		2.5		0.7
9	45-5			0.5	7.3	22.5		11.5		0.5	1.0
0	80.5	12.5	2.3	3.5	0.9	44.3	15.8	8•0 /IgO+Ca			0.4

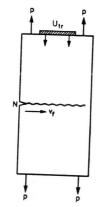


Fig. 1. Schematic arrangement for fracture surface modulation by transversal ultrasonic waves. (p=applied tensile stress,  $U_{tr}$ =ultrasonic transducer for transversal waves, N=notch.)

# Crack velocities in glass by ultrasonic fractography

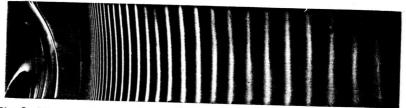


Fig. 2. Glass fracture surface modulated by ultrasonic waves at  $1\ \text{Mc/s}$ . On the left: accumulation point of ultrasonic lines.



Fig. 3. Glass fracture surface modulated by ultrasonic waves at 5 Mc/s. On the right: beginning of roughness and fracture branching.

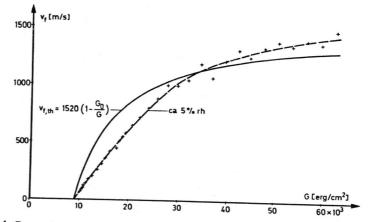


Fig. 4. Dependence of fracture velocity on specific fracture energy G (evaluated according to Irwin [7]). The unbroken line represents the theoretical dependence of fracture velocity on G according to Dulaney and Brace [10] with  $G_0 = 9000$  erg/cm<sup>2</sup>.

Fig. 5. Dependence of fracture velocity on specific fracture energy G (evaluated according to Gross, Srawley, and Brown [8] at various relative humidities at room temperature. The unbroken line represents the theoretical dependence of fracture velocity on G (at 5% rh) according to Dulaney and Brace [10] with  $G_0 = 8600$  erg/cm².

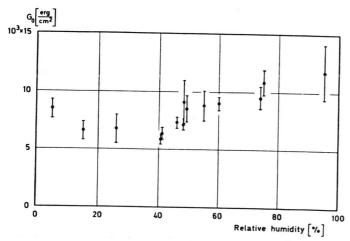


Fig. 6. Dependence of the critical specific fracture energy  $G_0$  on relative humidity at room temperature.

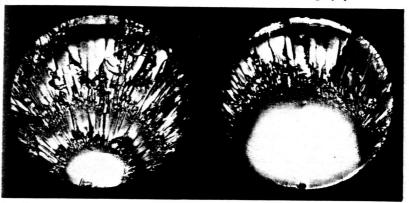


Fig. 7. Typical fracture surfaces of glass rods ( $\emptyset$ =9.5 mm) with fracture mirror (from Sommer [13]).

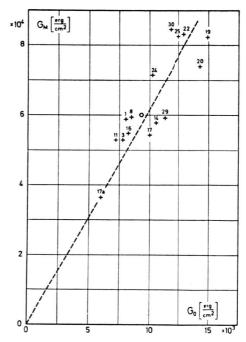


Fig. 8. Relation between fracture energy  $G_{M}$  at the beginning of roughness and the critical specific fracture energy  $G_{0}$  for some optical glasses. The circle represents the data for AR glass resp. plate glass.