

Initial crack paths in glass: influence of temperature and composition

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Fracture initiation sites in soda-lime glass are similar for fracture at room temperature and at -196°C , where slow crack propagation should be absent. For most samples the measured depths of these sites were greater than the actual depths of the fracture initiating flaws. Micrographs of fracture initiation sites in Pyrex borosilicate glass and a lithium aluminosilicate glass-ceramic are also shown.

1. Introduction

Measurements of depths of fracture initiating flaws in engineering materials are difficult and often uncertain. One way to observe the particular flaw that initiated fracture is to examine the fracture surface after failure. A study of fracture surfaces of rods of soda-lime glass showed that the initial stages of crack propagation are complex, and it is often difficult to identify the initiating flaw [1]. This study also showed that a circular feature on the fracture surface, sometimes taken as the initiating flaw, is actually much deeper than this flaw and results from the initial stages of crack propagation after application of the failure load. These rods were broken at room temperature, where water in the ambient air influences the initial stages of crack propagation ("static fatigue"), even for very short loading times. Thus one goal of the present work was to examine fracture of glass rods at low temperature where static fatigue or slow crack propagation did not take place. The results show that the initial stages of crack propagation in soda-lime glass are complex even in the absence of crack growth induced by reaction of water with the glass.

The fracture origins in Pyrex borosilicate glass and a glass-ceramic were also examined. The Pyrex fracture surface showed surprising irregularities, and the fracture origin in the glass ceramic was quite shallow.

2. Experimental methods

The methods were similar to those used in reference [1]. Samples were a soda-lime glass (Kimble R-6, nominal composition 73% SiO_2 , 15% Na_2O , 5% CaO , 4% MgO , 2% Al_2O_3 and 1% B_2O_3), Pyrex borosilicate glass (Corning 7740, 81% SiO_2 , 4% Na_2O , 13% B_2O_3 , 2% Al_2O_3), and a partially crystallized glass ceramic supplied by Owens-Illinois Corp. of lithium aluminosilicate composition. Rods of these materials about 3 mm in diameter were broken between two thumbs and two fingers to approximate a four-point bending stress. In most cases fracture occurred between the fingers in the region of maximum tensile stress. Rods were broken either in air at room temperature or after being held some time in liquid nitrogen. The broken segments were mounted for observation and the fracture surface cleaned with compressed air. The specimens were coated with a thin layer of gold, and observed in an MAC-700 scanning electron microscope.

3. Experimental results

Figs. 1 to 10 are representative micrographs showing fracture origins and surfaces of the different samples. Fracture temperatures and dimensions of fracture features are given in Table I. The apparent flaw depths were measured from the glass surface in the direction of crack propagation into the glass to the deepest part of the apparent flaw. Mirror

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TABLE I Fracture stresses and apparent flaw depths

Sample	Fracture temp. (° C)	Apparent flaw depth d (μm)	Mirror radius (mm)	Fracture stress S_f		$S_f\sqrt{d}$ (MN m^{-2})
				MN m^{-2}	kp.s.i.	
Soda-lime glass						
1S	-196	67	≈ 1.5	54	7.8	0.44
2S	-196	5	0.27	127	18	0.28
3S	-196	10	0.30	121	18	0.38
4S	-196	9	0.21	145	21	0.44
5S	25	14				
Pyrex borosilicate glass						
3P	-196	≈ 10	0.17	160	23	0.50
5P	25	18	0.28	128	18	0.54
Lithium aluminosilicate glass-ceramic						
6C	-196	6(?)	0.25	373	54	0.92

radii were measured from the glass surface at the initiation site into the glass (not along the surface) to the point of rough hackle, known as the "outer mirror radius." Mirrors and hackle from which such measurements can be made are shown in Figs. 5, 8 and 10. Many authors, [2-4], have shown that this mirror radius r is related to the fracture stress S_f by the equation:

$$r = \left(\frac{A}{S_f} \right)^2 \quad (1)$$

where A is a constant equal to about $2.1 \text{ MN m}^{-3/2}$ for silicate glasses. Fracture stresses calculated from this equation and value of A are given in Table I. For the glass ceramic A was taken to be 5.9 [5].

4. Discussion

The hackle boundary of sample 1S did not form a complete semi-circle (Fig. 1), probably because the fracture origin was not at the point of maximum stress. Thus the fracture stress calculated for this sample is somewhat uncertain. Sample 1S fractured at a comparatively low stress; the initial flaw or flaws were probably at the irregular regions in the centre of the semi-circular feature in Fig. 2, and were considerably deeper than the flaw depths of five to ten microns usually found for abraded soda-lime glass [6]. The two opposite surfaces shown in Figs. 2 and 3 both show the same semi-circle, but the central irregularities are somewhat different, suggesting that chips were removed from the glass either before, during, or after fracture.

The apparent flaw depth for sample 2S (Fig. 4) was only $5 \mu\text{m}$, which is about the same as the depths measured for soda-lime glasses of similar

strengths by an etching technique [6]. Therefore in this sample it appears that the original line crack in the sample surface was nearly perpendicular to the applied tensile stress, so the measured depth is close to the actual depth of the initiating flaw (compare with the discussion in [1]).

The surface features on samples 3S and 4S (Figs. 3 and 4) are irregular and deeper than those for sample 2S, even though the fracture stresses for these three samples are not much different, suggesting in agreement with reference [1] that the depths of these features in Figs. 3 and 4 are not the depths of the initiating flaws, which are shal-

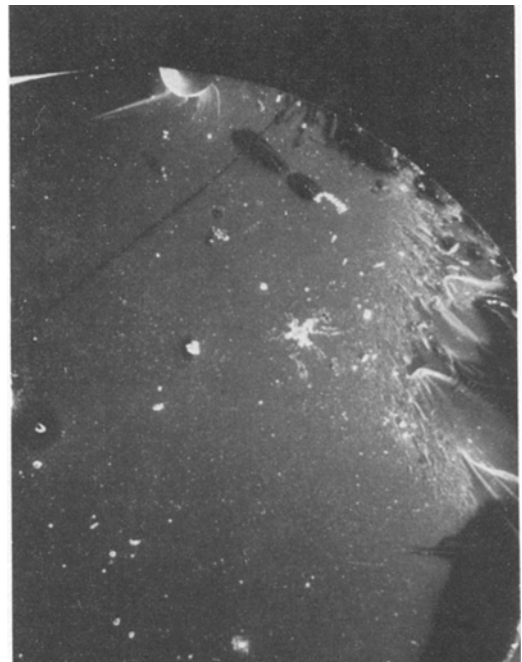


Figure 1 Scanning electron micrograph of the fracture surface of sample 1S. Fracture origin at the left ($\times 40$).

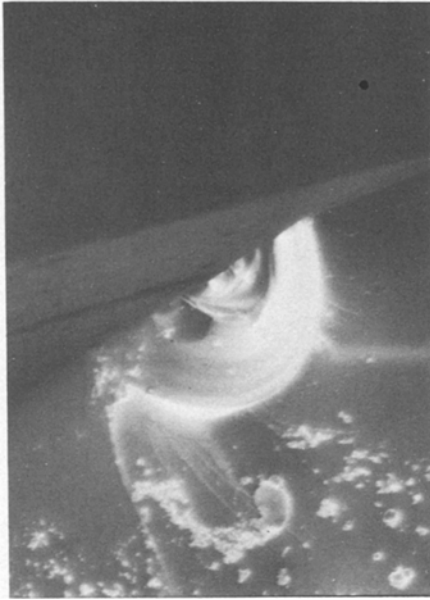


Figure 2 Scanning electron micrograph of the fracture origin for sample 1S. Top, original glass surface, bottom, final fracture plane (mirror). Opposite side to that in Fig. 1 ($\times 276$).

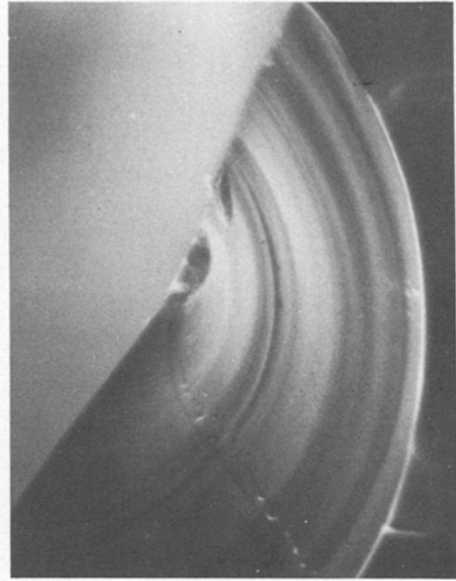


Figure 3 Scanning electron micrograph of the fracture origin for sample 1S. Top, original glass surface. Same side as in Fig. 1 ($\times 690$).

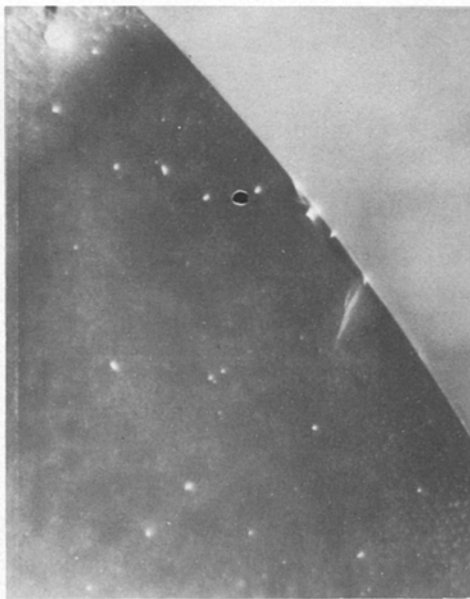


Figure 4 Scanning electron micrograph of the fracture origin for sample 2S. Top, original glass surface, bottom, fracture mirror ($\times 276$).

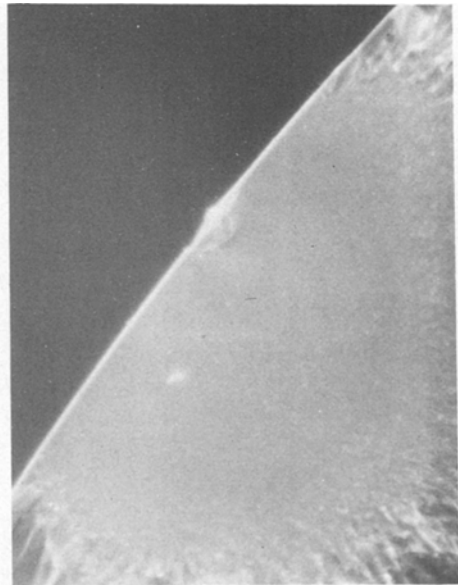


Figure 5 Scanning electron micrograph of the fracture origin, mirror, and hackle for sample 3S ($\times 345$).

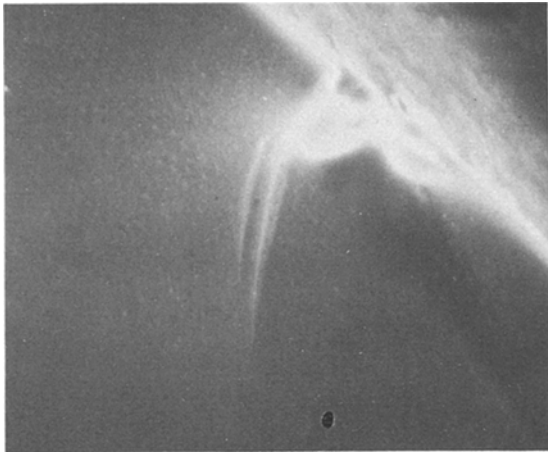


Figure 6 Scanning electron micrograph of the fracture origin of sample 4S. Top right, original glass surface ($\times 1380$).

low, but result from the initial stages of crack propagation.

Sample 5S was broken at room temperature, and its fracture initiation feature is similar to those in reference [1], which had depths of 56, 17, 16 and $7.4 \mu\text{m}$. There is no apparent difference between initial fracture paths in soda-lime glass at room temperature and at -196°C , where slow crack propagation should be absent.

The influence of the stress accelerated reaction of water at the crack tip, which results in slow

crack propagation and static fatigue, on the crack shape is uncertain. The rate of reaction of water with glass or a crystalline oxide is strongly dependent upon the applied tensile stress; exponential and high power dependencies have been assumed. In their elegant theory of static fatigue, Hillig and Charles [7] deduced that sharpening of cracks is much more important in weakening a brittle material than their lengthening. The stress is high only at the crack tip, and the strong dependence of reaction rate on stress means that the reaction is only significant right at the crack tip, where it reduces the tip radius. Hillig and Charles therefore concluded that the lengthening of the crack was negligibly small. Since there is no direct experimental evidence about shapes of crack tips, one can only examine this assumption indirectly. Many investigators have found that large cracks (perhaps 1 cm long) in brittle oxides do lengthen appreciably when held under stress in an atmosphere containing water. In particular Wiederhorn [8] found that centimetre long cracks in soda-lime silicate glass grow slowly up to at least half their initial length before propagating rapidly to failure, when they are held at tensile stress less than the short-time failure stress.

The fracture mirrors of some samples of Pyrex borosilicate glass broken at -196°C showed star-like roughening, as shown in Fig. 8; this roughening obscured the fracture-initiating site. The fracture surface of the glass ceramic broken at liquid nitrogen temperature looked much like the fracture surfaces of the soda-lime glass, see Fig. 10.

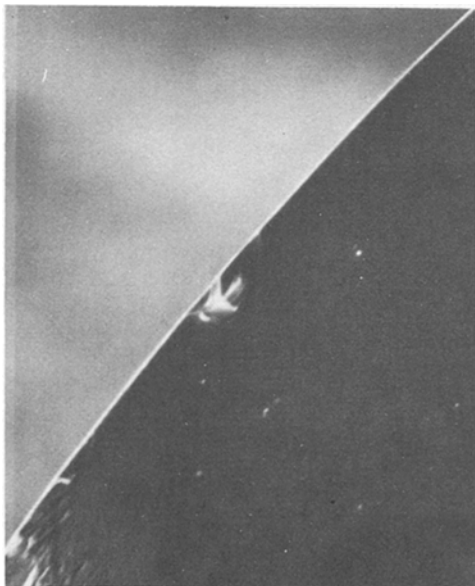


Figure 7 Scanning electron micrograph of the fracture origin of sample 5S. Top, original glass surface ($\times 345$).

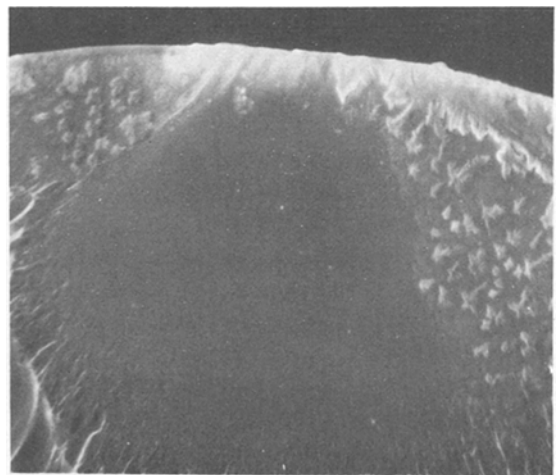


Figure 8 Scanning electron micrograph of the fracture surface of sample 3P ($\times 345$).

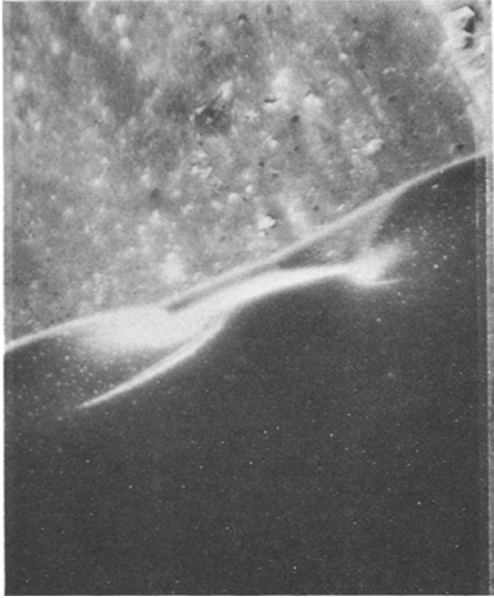


Figure 9 Scanning electron micrograph of the fracture origin for sample 5P. Top, original glass surface ($\times 345$).

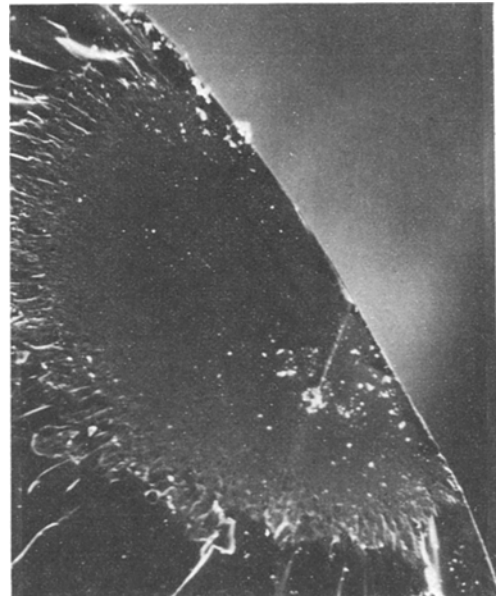


Figure 10 Scanning electron micrograph of the fracture surface of sample 6C ($\times 140$).

The present work confirms the conclusion of reference [1] that the initial stages of crack propagation in glass are complex, and that the flaw depth is usually less than the depth of features on the fracture surface at the initiation site.

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