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Roughness of glass surfaces formed by sub-critical crack growth

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Abstract

This paper presents a study on the roughness of glass fracture surfaces formed as a consequence of sub-critical crack growth. Double-cantilever-beam specimens were used in these studies to form fracture surfaces with areas under well-defined crack velocities and stress intensity factors. Roughness depends on crack velocity: the slower the velocity, the rougher the surface. Ranging from approximately 1×10^{-10} m/s to approximately 10 m/s, the velocities were typical of those responsible for the formation of fracture mirrors in glass. Roughness measurements were made using atomic force microscopy on two glass compositions: silica glass and soda lime silica glass. For silica glass, the RMS roughness, R_q, decreased from about 0.5 nm at a velocity of 1×10^{-10} m/s to about 0.35 nm at a velocity of 10 m/s. For soda lime silica glass, the roughness decreased from about 2 nm to about 0.7 nm in a highly non-linear fashion over the same velocity range. We attributed the roughness and the change in roughness to microscopic stresses associated with nanometer scale compositional and structural variations within the glass microstructure. A theory developed to explain these results is in agreement with the data collected in the current paper. The RMS roughness of glass also depends on the area used to measure the roughness. As noted in other studies, fracture surfaces in glass exhibit a self-affine behavior. Over the velocities studied, the roughness exponent, ζ, was approximately 0.3 for silica glass and varied from 0.18 to 0.28 for soda lime silica glass. The area used for these measurements ranged from $(0.5\ \mu m)^2$ to $(5.0\ \mu m)^2$. These values of the roughness exponent are consistent with values obtained when the scale of the measurement tool exceeds a critical size, as reported earlier in the literature.

Keywords: Mechanical properties; Crack growth; Fracture; Microscopy; Atomic force and scanning tunneling microscopy

1. Introduction

Examination of fracture surfaces in glass by atomic force microscopy (AFM) suggests that these surfaces are never flat in a Euclidian sense, despite their appearance under the optical or scanning electron microscope. Gupta et al. [1] have shown that the root-mean-square (RMS) roughness of fracture surfaces ranged from 0.34 nm to 0.83 nm depending on the glass composition and the method of making the glass. The variation in the fracture path through the glass was attributed to the structure of the glass, specifically to inhomogenieties in the structure. In an AFM study on silica glass, Poggemann et al. [2] obtained results similar to those obtained by Gupta et al. The spacing of the high points on the surface of their glass correlated with molecular dimensions within the glass structure: e.g. the Si–O and the O–O distances. Hence, the authors attributed the distances to typical features of the silica network.

Other AFM studies concentrated on the self-affine nature of fracture surfaces in glass. Various techniques were

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used to extract the roughness exponent, ζ, from fracture surfaces formed under a variety of conditions [3,4]. The roughness exponent depended on the length scale of the measurement. For length scales less than a critical value, ξ, the exponent is about 0.8 and is purportedly the same for all fracture surfaces. For length scales greater than ξ, ζ takes on the value 0.4. The critical length scale, ξ, depends on the crack velocity, ranging from about 20 nm to about 80 nm for crack velocities ranging from about 10^{-10} m/s to 10^{-5} m/s [4]. Theoretical justifications for these values have been given [4].

Although AFM studies have quantified the roughness of glass fracture surfaces, only one systematic study has investigated the effect of crack velocity on the roughness of glass for fracture surfaces formed by sub-critical crack growth [4]. Usually, specimens are simply broken and roughness measurements made on the flat area surrounding the fracture origin. This flat area is known as the 'mirror' region and is bounded by an area of fine scale roughness (the 'mist' region) that can just be detected optically. Beyond the mist region is the 'hackle' region where macroscopic roughness can be detected [5,6]. It is generally thought that the RMS roughness of glass is relatively constant in the mirror region, but increases as the glass fracture approaches and then passes through the mist into the hackle region [6]. Within the mirror region, near the crack origin, surfaces are optically smooth and featureless [6]. At approximately one-third the distance from the origin to the boundary between the mirror and mist regions, r_m, small scale roughness increases [7], but is still too small to be seen with an optical microscope so that the surface still looks smooth. At r_m the RMS roughness further increases so that features are now large enough to be seen optically. A similar scenario was reported for a brittle, glassy, isotropic epoxy resin [6].

In this paper, we examine topographic features in fracture surfaces for cracks moving at crack velocities ranging from about 1×10^{-10} m/s to approximately 10 m/s. All studies are carried out in water on double-cantilever-beam specimens (DCB); by using the DCB specimen, crack velocities can be closely correlated with topographic features detected by atomic force microscopy. Results show that the RMS roughness decreases with increasing crack velocity within the range of velocities studied. Furthermore, the RMS roughness over the range of velocities studied is greater for soda lime silica glass than for silica glass.

2. Experimental procedure

All crack velocity measurements on soda lime silica glass microscope-slides were made under deadweight loading on double-cantilever-beam specimens, $(75 \times 25 \times 1)$ mm in size using a diamond scribed scratch to guide the crack [8]. The composition of the soda lime silica glass by mass fraction (%), determined by X-ray fluorescence analysis, is as follows [9]: Na_2O – 14; MgO – 3.7; Al_2O_3 – 1.8; SiO_2 – 74; K_2O – 0.36; CaO – 5.9. The silica glass specimens were Corning C7980[2] slides, $(75 \times 25 \times 1.5)$ mm, with midline notches approximately 0.5 mm deep to guide the direction of crack growth; the crack width was about 1 mm [9]. Crack velocities ranged from approximately 1×10^{-10} m/s to 10 m/s. All experiments were carried out in water at room temperature (≈22 °C).

Crack velocities, v, were measured using either a traveling telescope (accuracy ±10 μm), or a digital camcorder (accuracy ±10 μm). The camera was capable of taking individual pictures at fixed intervals. In the course of the study, crack growth curves were obtained over a velocity range from 1×10^{-10} m/s to 1×10^{-2} m/s. Given an applied load, the experimentally determined crack growth curves, log v versus K_I, where K_I is the applied stress intensity factor, were used to calculate crack velocity as a function of position along the surface on the specimen. Initially, a load was applied to the specimens such that the crack velocity was in the 1×10^{-11}–1×10^{-9} m/s range. The crack was then permitted to grow to produce a large identifiable region that could be later studied by atomic force microscopy. Fracture surfaces were produced at higher velocities by increasing the load on the crack so that the crack velocity was in the range 1×10^{-9}–1×10^{-8} m/s. The crack was then allowed to grow under a constant load until the specimen failed. Since the load was constant, the value of K_I could be calculated as a function of crack length alone. Then, using the experimental $v - K_I$ curve, the crack velocity, v, could be calculated at every point along the crack trajectory. With this information, the fracture surface roughness could be determined as a function of crack velocity, or stress intensity factor at every point on the fracture surface of the slide. The highest velocities on the slide were not measured, but estimated as 10 m/s.[3] This value is consistent with the fact that the fracture surface was optically smooth to the very end of the specimen.

After the specimens were fractured in two, the crack velocity was determined as a function of distance along the slide, and optical micrographs were taken to identify the areas associated with the velocities. Height images were then made as a function of crack velocity for square areas having edge lengths of 0.5 μm, 1 μm, 2 μm and 5 μm. Regardless of AFM scan-area size, the number of scan lines was the same, 512, as the number of points in each scan line, also 512. The scan area was square; therefore, in the discussion below, the length of the edges of the square scan-areas, L_0, were used to represent the roughness data. The RMS roughness, R_q, of each area was determined by using the software package included with the AFM (Digital 3100, Veeco Metrology Group, Santa Barbara, CA,). Prior to measuring the roughness all images were flattened to 0

[2] The use of commercial names is only for purposes of identification and does not imply endorsement by the National Institute of Standards and Technology.

[3] The estimate was made from the intersection of the crack growth curve in water with that in dry nitrogen, using Ref. [8].