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**NEAR THE FATIGUE LIMIT IN GLASS**

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### ABSTRACT

The atomic force microscope is used to explore the nature of fracture surfaces in soda lime silicate glass formed near or below the apparent crack growth threshold. Conventional theory suggests that cracks in glass will blunt when subjected to stresses below the threshold. We see no evidence for blunting in this study. Cracks that are held below the apparent crack growth threshold for 16 h alter their mode of growth. The fracture plane changes from a flat surface to a surface that exhibits substantial out of plane growth. The crack changes its growth direction to an angle that lies between 3E and 5E to the original growth plane, leaving behind a wavy fracture surface. This change in crack morphology may be the cause of a time delay to restart crack motion when the applied stress intensity factor is again raised above the apparent crack growth threshold.

### KEYWORDS

Glass, crack growth, static fatigue limit, ceramics, stress corrosion, atomic force microscopy

### INTRODUCTION

Studies of crack growth in glass suggest that some glass compositions exhibit thresholds below which crack growth will not occur. In a study of this phenomenon, Wiederhorn and Bolz [1] showed that soda lime silicate glass and a chemically resistant borosilicate glass immersed in water gave evidence of a threshold stress intensity factor below which crack growth would not occur. Crack velocities greater than about  $10^{-7}$  m/s could be expressed as an exponential function of the applied stress intensity factor,  $K_I$ . Below about  $10^{-7}$  m/s, the crack velocity decreased at a rate that is faster than that suggested by

the exponential behavior. As the crack growth curve had a negative curvature, crack growth appeared to approach a threshold below which crack growth would stop as the applied stress intensity factor was reduced. These experiments on soda lime silicate glass have been confirmed by Kocer and Collins [2] to crack velocities as low as  $10^{-14}$  m/s. In the light of the stress-corrosion theory by Charles and Hillig [3], Wiederhorn and Bolz interpreted this behavior as evidence for crack tip blunting. The radius of curvature of the crack tip was assumed to increase with time below the threshold. Hence, the crack became more difficult to propagate.

In a later study, Michalske [4] reinforced the idea that the downward curvature of the crack growth curve was due to crack tip blunting. Michalske applied a value of  $K_I$  that lay below the apparent crack growth threshold and held it for a 16 h period. He felt that if crack blunting occurred, then a time delay would be needed to restart a crack when the stress was raised to a value of  $K_I$  that lay above the apparent threshold for crack growth. Michalske found that a time delay did occur and that it depended on the level of  $K_I$  used to restart crack motion. If  $K_I$  were just slightly above the threshold limit, several thousand seconds were required to restart crack motion. However, if  $K_I$  were substantially above the threshold, then the crack would restart its motion without delay.

In addition to the time delay to restart the crack, Michalske observed a pattern of marks on the fracture surface that were clearly associated with the “arrested crack.” These were a series of light and dark bands that lie along the crack front at the point of crack arrest. The nature of these bands was not obvious, but Michalske attributed them to the process of re-sharpening a blunted crack. To him it seemed that the crack became “segmented as it extends from its position during aging.” These segments were believed to result from the “nucleation and growth of sharp cracks from the rounded crack tip.” His conclusions on what was happening during the experiment were very reasonable. However, at the time there was no instrument available that would clearly define the nature of the markings at the arrested crack tip.

The atomic force microscope is an instrument that has both the lateral and normal resolution needed to fully characterize the marks first observed by Michalske. We use this instrument to show that these markings represent a non-planar propagation of the crack near the apparent crack growth threshold, such that some portions of the crack front propagate out of the projected crack plane and other portions propagate into the projected crack plane. The time delay for re-propagating the crack may be the time needed for the crack to again propagate on a single plane.

## **EXPERIMENTAL PROCEDURE**

The experimental procedure was identical to that used by Wiederhorn and Bolz [1] and very similar to the one used by Michalske [4]. Double cantilever beam specimens 75 mm by 25 mm by 1.5 mm were dead weight loaded using a small laboratory pan balance to transmit the force. The glass was from the same batch as used by Wiederhorn and Bolz. Narrow side-grooves in the specimen maintained the advancing crack along the midline

of the specimen. We monitored the crack position with a 40x microscope having a filar eyepiece and determined the position of crack to an accuracy of 5  $\mu\text{m}$ , using light reflected from the crack surface. Michalske did not use side-grooves and monitored the crack motion with transmitted light. Other than that, the same equipment was used in both experiments.

We adopted Michalske's experimental procedure to carry out our experiment. A crack was first propagated at a relatively high value of  $K_I$ . The value of  $K_I$  was then reduced to a value less than the apparent crack growth threshold. The crack was finally repropagated at the higher value of  $K_I$ . Studies reported in this paper were all carried out in water. The high  $K_I$  values for crack growth measurements ranged from about 0.35  $\text{MPa}\cdot\text{m}^{1/2}$  to about 0.55  $\text{MPa}\cdot\text{m}^{1/2}$ . The lower  $K_I$  values used to induce crack arrest ranged from 0.22  $\text{MPa}\cdot\text{m}^{1/2}$  to 0.30  $\text{MPa}\cdot\text{m}^{1/2}$ . The measurement carried out at 0.30  $\text{MPa}\cdot\text{m}^{1/2}$  was only held for eight hours; all others were held for 16 h. Once a crack started propagating, it was permitted to propagate approximately 1 mm and then again held at a lower stress intensity factor for 8 or 16 h. All measurements were carried out on a single specimen. Upon completing the crack growth experiments, we broke our specimen in half along the crack plane to expose both fracture surfaces and then examined and compared both surfaces with an optical and a Digital III atomic force microscope<sup>1</sup>, using the contact mode to image the fracture surface.

## EXPERIMENTAL RESULTS

We reproduced Michalske's earlier work in these studies. A time delay to restart the crack was observed when it was held at stress intensity factors in the range 0.22  $\text{MPa}\cdot\text{m}^{1/2}$  to 0.30  $\text{MPa}\cdot\text{m}^{1/2}$ . We also observed the same microscopic arrest features

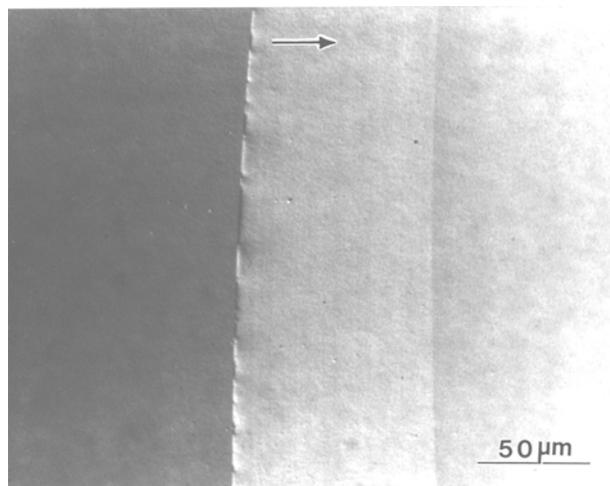


Figure 1: Crack tip that had been arrested and then restarted:  $K_I=0.23 \text{ MPa}\cdot\text{m}^{1/2}$ ; hold time 16 h. The arrow indicates the direction of crack growth.

<sup>1</sup>The use of commercial names is for identification purposes only and does not imply endorsement by the National Institute of Standards and Technology

reported earlier by Michalske. When the crack was held at stress intensity factors in the range  $0.22 \text{ MPa}\cdot\text{m}^{1/2}$  to  $0.30 \text{ MPa}\cdot\text{m}^{1/2}$  for 16 h, light and dark bands decorated the crack front, Figure 1. By contrast, unloading and restarting the crack immediately left only a single faint mark where the crack had changed direction slightly, Figure 1. We observed some variability in the appearance of the marks that decorated the crack front of the arrested crack. Sometimes only a few dark and light bands were observed; in other cases there was only one light or dark band on the crack front.

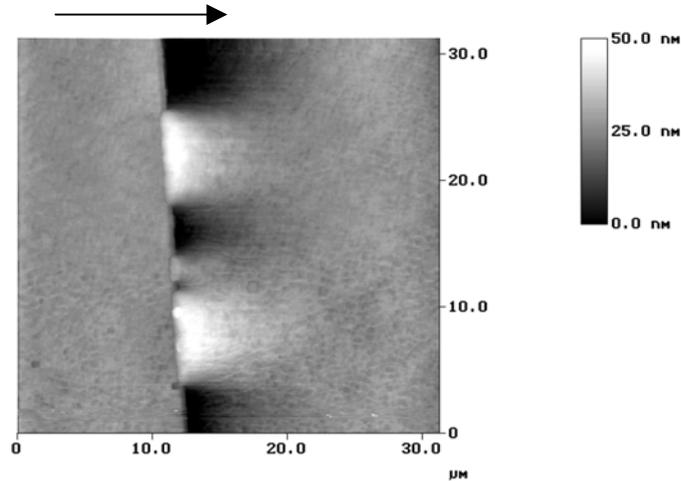


Figure 2: Direction of crack growth from left to right, see arrow. The lighter shades indicate an increase in the height of the fracture plane above the original fracture surface. This is not the same crack front as shown in Figure 1.

An AFM image of the fracture feature reveals what the optical microscope could not reveal, Figure 2. At the point of crack arrest, variations in the height of the fracture surface developed as a consequence of the hold time. As the crack advanced to the position where  $K_I$  was decreased, the crack was a smooth almost flat surface. The repropagated crack lay either above or below the original crack plane. As all parts of the repropagated crack were connected, these features suggest waviness to the crack front near the point of “arrest.” As the crack grew, the waviness gradually disappeared, and the crack again propagated on a single plane.

Sectioning of the features in Figure 2 provides more details on the crack growth, Figure 3. Sectioning lines parallel to the direction of crack advance show that the crack propagated on a smooth plane prior to unloading. After reducing  $K_I$ , holding it for a period and then increasing it again, the direction of crack growth is found to have changed; some portions of the crack propagated into the projected crack plane, others propagated out of the crack plane. The new angle of propagation was  $\pm 3^\circ$  to  $\pm 5^\circ$  to the original crack surface. With continued crack growth, the direction of crack propagation gradually changed back to a single plane.

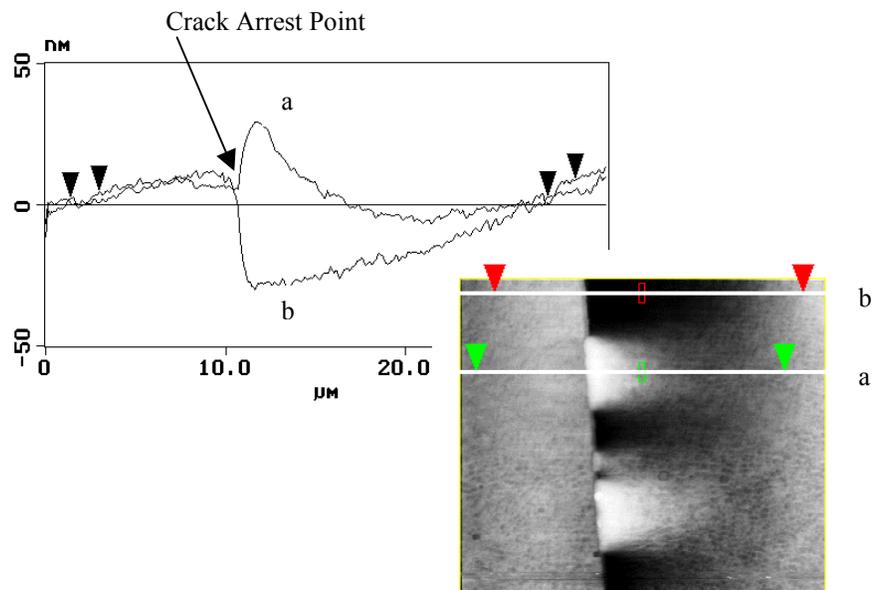


Figure 3: Sectioning of the features in Figure 2 shows a bifurcation in the crack propagation plane; part of the crack moves into the plane, the other part moves out.

In order to interpret the growth process, it is important to know how closely the opposing faces of the crack replicated one another. Presumably, if crack blunting occurred, the shape of the upper and lower fracture surfaces would be different. Hence, AFM pictures were taken from each surface of the crack, and the same feature on each surface was sectioned and compared. Overlaying the two sections, Figure 4, shows that the two sections replicate one another to an estimated accuracy of  $\pm 2$  nm.

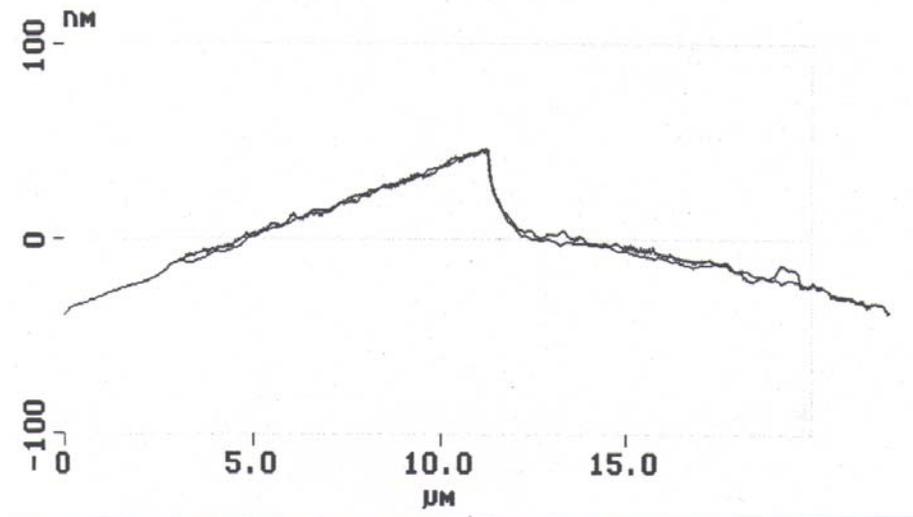


Figure 4: Section of the identical feature on opposite sides of the same crack. Within experimental scatter, the two images duplicate one another.

## DISCUSSION

Within the accuracy of our AFM measurement, the crack propagates as a “sharp” crack in soda lime silicate glass, and does not appear to blunt during the hold period. If the crack is in fact sharp, the time delay for restarting crack growth cannot be attributed to a resharping of the crack tip. Instead, the time delay to restart the crack may be the time needed for the crack to again propagate on a single plane.

Altering the far field applied stress will cause a change in the direction of crack growth. The changes along the crack front that we observe are clearly of the type that could be caused by Mode II loading, which cause the crack to propagate out of plane. However, if the features were due to a remotely applied stress, the entire crack front would be expected to propagate out of the original crack plane, and that does not happen. One could argue that the features are due to a stress applied to the crack surface near the crack tip. Accounting for the features in Figure 1 would require stresses to be active over a range of distances from 1  $\mu\text{m}$  to 10  $\mu\text{m}$ . As it is difficult to imagine the source of such surface stresses, we turn elsewhere for an explanation of our observations.

A possible source of the out of plane growth may be found in a modification of the Charles Hillig theory suggested recently by Chuang and Fuller [5]. In the Charles-Hillig theory, crack growth occurs because of a stress enhanced chemical attack of water on the glass surface of the crack. Charles and Hillig concentrated their theory on the curvature of the tip of the crack and the effect of the chemical reaction on that curvature. Their model suggested that below the threshold, cracks should blunt, whereas above the threshold, they should sharpen. The threshold was the stress at which the crack propagated in a self-similar manner. Chuang and Fuller expanded on this theory by exploring the change in surface corrosion rate over the entire crack surface, not just at the tip. Their model predicted that below the crack growth threshold, as defined by Charles and Hillig, the surface corrosion rate was no longer fastest at the crack tip, but at an angle to the original crack plane, which depended on the stress. Therefore, there is a tendency for out of plane crack growth. Whether this tendency can actually result in the kind of features we see has yet to be determined.

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