International Journal of
MATERIALS RESEARCH

Zeitschrift für METALLKUNDE

Basic

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Do plastic zones form at crack tips in silicate glasses?

Dedicated to Professor A. G. Evans on the occasion of his 65th birthday

A number of recent studies claim that silicate glasses fracture by the formation, growth and coalescence of cavities at crack tips, in the same way as metals, but at a much smaller scale. Evidence for cavity formation comes from the examination of side surfaces of fracture mechanics specimens, at the point where the crack tip intersects the free surface. Such measurements exhibit small depressions in regions that are supposedly located in front of moving crack tips. These depressions were interpreted as cavities. In this paper, we summarize experimental results obtained using an atomic force microscope to characterize the fracture surfaces. The experimental results demonstrate an absence of residual damage on fracture surfaces that could be interpreted as cavity formation. We also observe cracks moving in glass and show that the features reported as cavities actually occur behind and not in front of the moving crack. A simulation of an atomic force microscope probe passing over the emerging tip of a crack in glass suggests that the features identified as cavities are in fact due to the roughness of the specimen surface. Our results support the view that cracks in glass propagate by brittle fracture. We find no evidence for nanoscale ductility in silicate glasses.

Keywords: Atomic force microscopy; Glass; Fracture surfaces; Simulation; Crack growth

1. Introduction

1.1. Plasticity at crack tips in glass

The idea that plastic deformation can occur at crack tips in glass and other brittle ceramics has always been a subject of some controversy. About 30 years ago, the debate seemed to end on the side of dislocation-free cracks in brittle ceramics (covalent, inorganic, non-metallic materials), and no plasticity at crack tips in silicate glasses. The debate had been activated by Marsh [1], who observed that silicate glasses could be deformed by a diamond Vickers hardness indenter. After indenting, impressions were left in the surface that looked like plastic impressions, suggesting that plastic deformation occurs in glass. The same kind of impressions could be made in ceramics such as Al_2O_3, SiC, or Si. By using transmission electron microscopy to examine the indented region, it was possible to show that these impressions were indeed due to plastic deformation [2]. The region surrounding the plastic impression was filled with dislocations, which are typical of crystalline materials that had been deformed plastically.

With these observations, one had to ask the question: if plastic deformation can occur at indentations in glasses and ceramics, why can it not occur at the tips of cracks in these materials? Lawn et al. [3] investigated this idea by using transmission electron microscopy (TEM) to look

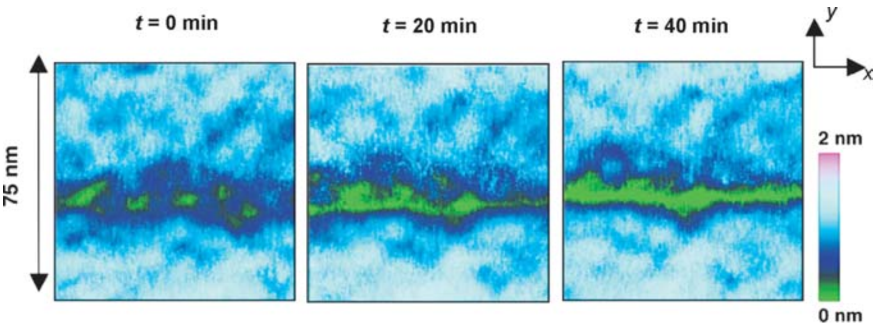

Fig. 1. AFM images presented in Ref. [5]. (a) A dark blue depression is seen to extend from left to right in frame; within the depression are deeper areas interpreted as "cavities". These grow and join up in frames (b) and (c) to form one continuous crack. Thus, the mechanism of crack growth is believed to be due to the nucleation and coalescence of cavities. The crack in this figure propagated from left to right.

through crack tips in brittle ceramics. Whereas glide dislocations formed around indentations in brittle ceramics, they were never observed at crack tips in these materials, at room temperature at least. Such dislocations as were observed at crack tips blended into Moiré fringes that characterized misalignment in healed fracture surfaces of crystalline materials [3]. Healing was clearly demonstrated between fracture surfaces of Si and Al_2O_3, without a trace of emitted dislocations near the crack tip [3].

Glasses cannot be examined in the same way as crystalline ceramics, since they do not diffract electrons coherently. However, the molecular structure of glasses is similar to that of silicate crystals, quartz for example, and these also show no indication of plastic deformation in tension. This similarity argument satisfied most ceramic scientists. Some, however proposed a different view: in Ref. [4], for example, the authors suggested that cracks in silica glass propagate by water penetration into the glass structure near the crack tip, resulting in a softening of the glass. The glass being softer would flow plastically and separation would occur between the two fracture surfaces as a consequence of this plastic flow.

1.2. AFM observations of crack tips in glasses

In a number of recent papers, a new group of authors claimed to have obtained direct experimental evidence of nanoscale plasticity at crack tips in glass [5–7]. Crack motion in glass fracture mechanics specimens was monitored by atomic force microscopy (AFM) [5, 6]. The authors examined the surface through which the crack tip penetrated, and monitored the position of the emerging crack tip as a function of time. In their study, the crack was normal to the plane of observation and moving from left to right at very low crack velocities, 10^{-13} m s^{-1} to 10^{-10} m s^{-1}, Fig. 1. Also monitored was the height of the stress-free surface that surrounded the emerging crack tip. The specimen was under a relatively low applied stress intensity factor, which forced the crack to propagate at about 10^{-11} m s^{-1}. The authors noted the presence of a shallow trough in front of the crack, about 0.5 nm deep and 5 nm wide and extending for about 100 nm in front of the crack tip, Fig. 1. This trough was believed to be a plastic zone, within which were deeper spatially localized depressions, >2 nm deep and limited in extent to about 20 nm in the direction of the trough. The depressions were alleged to be "cavities" growing near the surface of the glass. The growth and linkage of these "cavities" was thought to be responsible for the crack growth. From this evidence, the authors suggested that cracks in glass move by the growth and coalescence of cavities just as in metals, but on a much smaller scale.

The apparent fact that cavities can be observed at the surface of a tensile specimen is surprising, because of the change in the stress condition at the surface where the tensile stress component normal to the surface has to be equal to zero. At the surface, a condition of plane stress exists; as a consequence, the hydrostatic stress at the surface is smaller in magnitude than at some point away from the surface. It is for this reason that cavities in metals form ahead of the crack, but away from the stress-free surface of the fracture specimen [8]. Thus, cavity formation along the crack front should be greater within the solid than at the external surface. With this in mind, the present authors initiated a study to detect cavities on fracture surfaces formed by a slowly moving crack [9, 10].

1.3. Inspection of fracture surfaces by AFM

The experiment performed by Guin and Wiederhorn was in concept simple. These authors used the atomic force microscope to map the fracture surfaces formed by the propagation of cracks at speeds ranging from 10^{-10} m s^{-1} to 10^{-2} m s^{-1}. Once the crack growth experiment had been completed, the fracture mechanics specimen was broken in two and height images were made from both opposing fracture surfaces. The opposing images were digital and could be compared quantitatively. The comparison was done in two ways: first by forming a digital section profile through the opposing images and comparing the profiles; the second by direct comparison of the two images. Identical roughness marks on both surfaces were used to index the surface to each other and, thus, to assure alignment. Both techniques could be applied quantitatively since the distances normal to the fracture surfaces can be measured accurately to approximately 0.15 nm [10].

An example of the profile analysis is shown in Fig. 2. The light and dark height images at the top of the figure represent raw data from the AFM scan. Light indicates high, dark indicates low, Fig. 2a and b. The total range of the scale from light to dark in these images is 2 nm. The quality of the shading is identical for both images, suggesting that the height contours of both surfaces are the same. The stars on each surface were made by connecting principal dark and light features on each surface. The stars are close to being identical, as seen in Fig. 2a where the stars are compared. The white lines show the locations of the section profiles, which are compared in Fig. 2c. The two profiles, one given by the light curve and the other by the heavy curve, overlap over most of the figure. Differences between the two profiles are estimated to be less than 0.3 nm over the entire set of curves. The diamond shaped schematic, Fig. 2d, shows what a cavity, 20 nm by 5 nm, would look

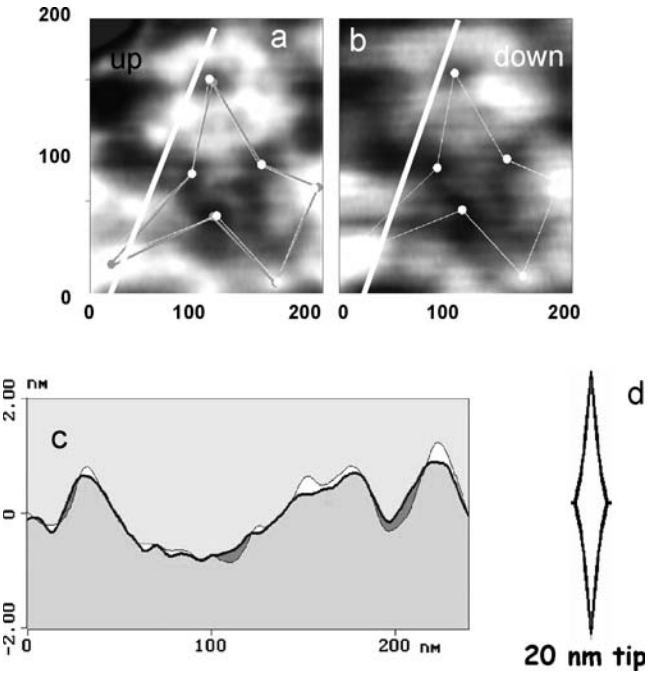

Fig. 2. Profile made from two opposing fracture surfaces, (a) and (b). Directions on the original image have been reversed so that on this kind of plot, the images will have the same value of height shading. The diamond like figure in (c) shows what a cavity, 20 nm by 5 nm should look like if present on the fracture profile shown in (d). The profile is taken along the white lines in (a) and (b). In the images are from soda lime silicate glass tested in air at a velocity of about 1×10^{-2} m s^{-1}. Taken from Ref. [9].

like if located at the fracture surface [10]. In other words, the mismatch between the two fracture surfaces would have to be the size of the diamond if cavities 20 nm by 5 nm were generated during fracture. The scale on the diamond schematic, Fig. 2d, is identical to Fig. 2c on its left. Based on the analyses of many images like these, we concluded that there are no 20 nm by 5 nm cavities formed in either soda lime silicate glass or silica glass [9, 10].

Arguments have been made that the kinds of section comparisons shown in Fig. 2 could have occurred by random chance between fracture surfaces that were otherwise uncorrelated, and therefore, such figures are not evidence that the surfaces are cavity-free. We find this argument hard to accept in the view of the fact that images such as those in Fig. 2 show a high degree of correlation between opposing surfaces. Figures such as Fig. 2 have been obtained from at least 10 other fracture surfaces that we have examined, with similar correlations [9–11]. Furthermore, profiles made in different directions on the same image have consistently shown the fracture surfaces to be cavity-free in the same sense as in Fig. 2.

Nevertheless, we also performed another kind of comparison to show that the two surfaces match one another and that there are no cavity remnants located at the fracture interface. An image comparison program was used to align opposing silica fracture surfaces and to minimize the distance between them [11]. The minimization procedure is carried out on tens of thousands of points distributed over the surfaces. After minimization [11], a histogram of the distances between the two surfaces is plotted, Fig. 3. A negative distance on Fig. 3 means that the two surfaces overlap; a positive distance means that the two surfaces do not touch. The histogram in Fig. 3 compared 204 472 points.

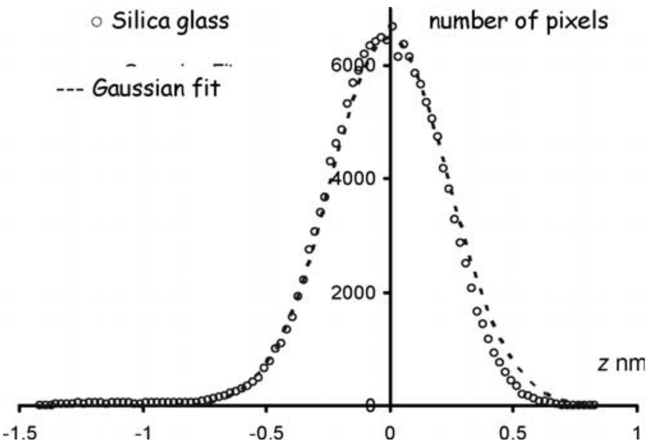

Fig. 3. A histogram of the mismatch distance between two fracture surfaces (open circles) in silica glass. The distances follow a Gaussian distribution (dashed curve) with a mean value of zero and a standard deviation of 0.22 nm. Although the plateau of data between approximately −1 and −1.5 indicates non Gaussian behavior, the data was analyzed no further. The analysis involved 204 472 points of comparison; the bin size was 0.0227 nm.

The measurable distances ranged from −1.42 nm to 0.877 nm; no distances were obtained outside of this range. A Gaussian fit to the data, the dashed curve in Fig. 3, yields a mean value of zero and a standard deviation of 0.22 nm, which means that 95 % of all of the data lay between −0.44 nm and 0.44 nm.

If cavities of order 125 nm by 25 nm form at crack tips in silica glass [7], then the remnants of these cavities should be observable in Fig. 3 as a peak lying somewhere between 10 nm and 25 nm, depending on how the cavities form and what sort of remnants are present on the surface. The fact that nothing is detected at distances larger than about +1.5 nm is strong evidence that cavities of the size reported in Ref. [7] are not present at the tips of cracks in silica glass. In the original discussion of Fig. 3 it was argued that, if present, cavities in the silica glass had to be smaller than 6 nm by 0.44 nm [11], which also excludes cavities of the size reported in Ref. [5]*. Smaller size cavities could not be detected in our experiments, as they would be lost in measurement noise. Thus, the nm size cavities discussed in molecular dynamics simulations of the fracture of silica [12, 13] could not be observed using our techniques, even if they were in the glass.

1.4. How to explain the observations of Ref. [5]

In view of the AFM experiments to search for cavity remnants on fracture surfaces, the observations made by Célarié et al. [5] must have been the result of some other physical process, not associated with cavity formation. In the remainder of this paper we explore this possibility. First, we present the results of crack growth data obtained by AFM. A "before" and "after" trace of a propagating crack suggests that the features reported in Refs. [5] and [7] appeared behind and not in front of the moving crack. Then, through a simulation of the crack motion, we show that images very similar to those shown in Fig. 1 can occur as a consequence

* The $x-y$ resolution is limited to greater than 6 nm, while 0.44 nm is at the two standard deviation boundary of the histogram in Fig. 3.

of the roughness of both the stress-free glass surface and the fracture surface of the glass. While other factors might contribute to our observations, we believe the primary cause of the features reported in Refs. [5] and [7] to be surface roughness, and not cavity formation at crack tips.

2. Experimental procedure

2.1. Crack growth

Crack growth studies were made on soda lime silicate glass microscope slides. It is well known that cracks formed by a Vickers diamond indenter in soda lime silicate glass continue to propagate long after the diamond indenter has been removed from the glass surface as a consequence of residual stresses associated with the indentation [14]. We used 20 N loads to propagate cracks from the indentation. A field of 16 indentations was arranged in a square array and the cracks so formed were examined for their suitability for study under the AFM. The cracks have to be approximately normal to the specimen surface. If a crack was not normal to the surface of the microscope slide then a shear lip formed near the crack tip, which made it unsuitable for examination because one side of the crack would be higher than the other. Using an optical microscope to examine the microscope-slide surface permitted us to establish the fact that the crack was narrow and nearly normal to the surface of the glass. Normality was established when the crack did not change position on focusing into the glass. Of the approximately 60 cracks that were formed and examined, approximately 6 were suitable for examination by AFM.

In order to be smooth enough for our purposes, the glass surfaces were first flame polished. As noted by Gupta et al. [15], such surfaces have about one-half the roughness of surfaces that are mechanically polished. This has also been our experience. Over a square area 0.5 µm on a side, the rms roughness (root-mean-square roughness)* of the surface was approximately 0.3 nm. This surface was comparable to the polished surfaces used in Ref. [5].

Once the indentations were put into the glass, cracks propagated, rapidly at first, then more slowly. The cracks propagated at a slow enough velocity, 10^{-11} m s^{-1} to 10^{-10} m s^{-1} by the second day to be used in our study. The slides had to be stored in a desiccator over night to avoid water condensation within the crack; otherwise condensation of water and precipitation of $CaCO_3$ left small particles on the surface of the glass, which made the specimen useless for examination by AFM. Specimen surfaces were examined using contact mode tips (DNP tips, Veeco, Santa Barbara, CA)** with a probe tip radius of 20 nm, reported by the manufacturer. Our study used the contact mode because the scans could be done faster with the contact mode than with the tapping mode. Also, the contact mode tips tended to sweep the surfaces clean of debris inadvertently located within the scanning area.

For the present experiment, the AFM area scanned was located initially in front of the moving crack. As the crack propagated, it eventually passed through the scanned area. The position of the frame was held constant primarily against piezoelectric drift, which required adjusting the scanning area so that characteristic regions (high points for example) within the scanning area stayed fixed. Thus, only the crack moved from frame to frame. This procedure permitted us to scan the area once every few minutes. In this way a motion picture of the crack movement could be compiled. For the present paper, we are only interested in comparing the appearance of the scanning area as the crack started to pass through the area being scanned and as the crack finished its passage. So, only two frames of the area being scanned will be shown in this paper.

2.2. Simulation of crack motion

Summarizing our simulation, surface features similar to the ones shown in Fig. 1 were obtained by first generating a surface with a roughness similar to the one shown in Ref. [5], using surface generation routines similar to those described in literature on fractal geometry, see for example Ref. [16]. Then, a crack was propagated through the surface in the same way as shown in Fig. 1. The crack had a roughness typical of that measured in Ref. [11]. The crack opening displacement increased with distance from the crack tip, as expected for a crack in a brittle solid [17]. As the crack propagated through the "rough surface", the surface was scanned by a probe with a spherical tip, and the rise and fall of the probe was monitored. The family of curves giving the position of the probe defined the surface topography as the crack grew through the surface. Thus, the surface displacement due to the passage of a crack was computed.

The surface that we generated represented the stress free surface of the Double Cleavage Drilled Compression (DCDC) specimen containing the emerging crack tip that was scanned in Refs. [5–7], see Fig. 4. Using the techniques described in Ref. [16], the surface was randomly generated to have a roughness exponent H of about 0.5 [16]. Then, both a high- and a low-pass filter were applied to eliminate small and high wavelength features from the surface. Finally, the surface was scaled to have a given rms value. The parameters were chosen so that the surfaces looked "right" in the sense that they resembled the images reported in Ref. [5]. In the simulations used in our study,

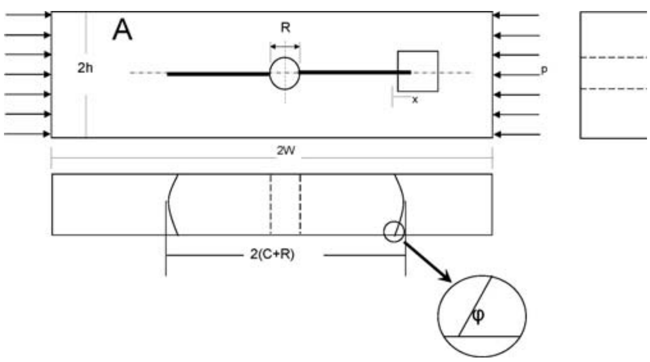

Fig. 4. Schematic diagram of test specimen. The crack lies perpendicular to the large surface of the specimen, **A**, and the crack front makes an angle, φ, with that surface. Displacements are measured within the square surrounding the crack tip, perpendicular to surface **A**.

* This is the standard deviation of the height of the surface about a mean plane passing through the surface.
** The use of commercial designations is for purposes of identification only and does not imply endorsement by the National Institute of Standards and Technology, nor does it imply that the items mentioned are the best ones for the intended application.

$H = 0.5$, $r_{highpass} = 20$ nm, $r_{lowpass}$ varied from 3.5 nm to 6.5 nm, where $r_{highpass}$ and $r_{lowpass}$ were the high and low pass wavelengths, and the rms varied from 0.14 nm to 0.18 nm. The $r_{lowpass}$ filter controlled the average size of the surface "bumps", and the rms roughness controlled their depth. To match Célarié's results, the same color map was used with a 2 nm height range, and the area was taken as a 75 nm × 75 nm square (250 × 250 pixels).

A path through the surface representing the crack edge position was generated by means of a random walk generator. The generator used was tuned to produce a positively correlated walk with little contribution from higher frequency components. This produced a rather smooth shape for the curve (fractal dimension between 1.05 and 1.1), which was furthered by applying a low-pass filter with a kernel radius varying from 4.5 nm to 8.5 nm. The curve was then horizontally centered on the simulated image, and its rms value was established to between 1.5 nm and 3.5 nm by scaling with an appropriate constant. The final shape for the crack edge is a smooth, spike-less curve that meanders with small amplitude as compared to the edge length.

Some parameters are needed to match the images published by Célarié et al. [5]: $K_I = 0.43$ MPa · $m^{1/2}$; probe tip radius = 10 nm; $W = 20$ mm (half height of the DCDC sample). We use the elastic solution for the surface displacements (A in Fig. 4) near the crack front [18]. The crack surface was normal to A and the crack front intersected A at an angle of $\varphi = 60°$, Fig. 4. In the simulation, the crack tip begins 18 nm to the left of the image and advances 4.5 nm between frames. The only other parameter needed is the applied stress p, which is a proportionality-constant for the displacements. Once collected, the images can be assembled to form a motion picture of the crack growth. In the results section of this paper, we present three such images for comparison with the images obtained in Ref. [5].

3. Results

3.1. Crack growth in glass

In the course of this study we spent many hours observing the motion of the tips of emerging cracks in soda lime silicate glass. The start and finish of one set of these observations is shown in Fig. 5. Figure 5a is the start of the crack motion. Marked on the figure is the crack tip depression, which can be distinguished from other dark regions by the fact that it moves relative to the rest of the image as the crack grows. This depression of the free surface is a consequence of a Poisson contraction along the crack front close to the free surface. The crack behaves as if it were applying a line tension to the surface at the point at which the crack tip emerges from the body of the glass. It is important to note in Fig. 5a that there exists no shallow linear zone of depression in front of the crack tip. The surface in front of the crack tip exhibits random highs and lows, typical of a surface with an rms roughness of about 0.3 nm. These highs and lows remain fixed to the image as the crack moves, but may be affected by a general depression in the surface as the crack passes them.

In Fig. 5b, the crack has grown completely across the image. Now the deep depression is at the top of the image. A shallow, linear depression trails behind the depression that characterizes the crack tip. This linear depression is in fact the crack. Note that the depth of the linear depression does not appear to be constant along its length. Some regions are deeper, whereas others are shallower. The apparent range of depths is a consequence of surface roughness – those areas that were deep on the original surface augment the apparent depth of the crack once it has formed. The converse is true of areas on the surface that were high relative to the original surface. Clearly, the roughness of the surface plays an important role in what the crack looks like after a crack tip has passed through the surface.

3.2. Role of roughness in affecting the appearance of a crack

To illustrate the importance of roughness to a crack image, we simulated the passage of a crack tip through a rough surface, as described in Section 2.2. In Fig. 6a, the crack has already propagated completely across the image. The deep trough (the band of dark blue going from the left side of the image to the right) arises for two reasons. First, the depressed region near the emerging crack tip extends for some distance back from the crack tip. Second, as the crack opens, the scanning probe tip enters into the open crack indicating a depressed zone. Initially, as the crack first opens, it is too narrow to register strongly on the AFM probe. Most of the depression is due to the elastic displacement field near the crack tip, at the free surface of the specimen. As the crack moves on, the displacement field vanishes and the probe senses the crack by penetrating into it as much as the curvature of the probe tip will allow. Since the crack

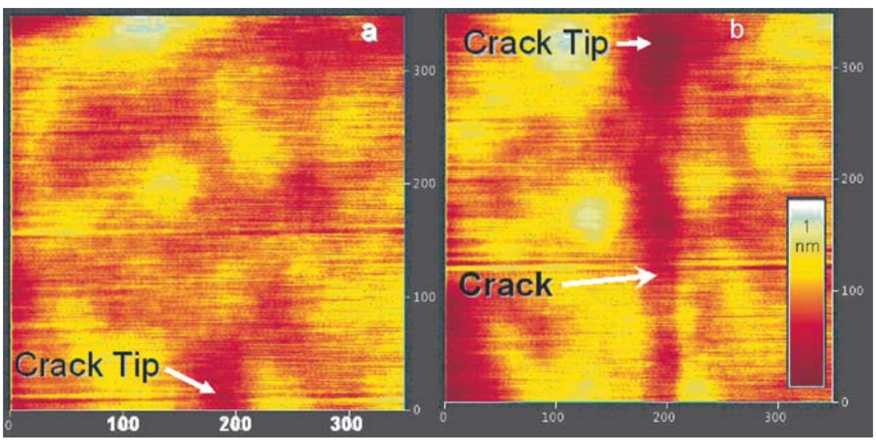

Fig. 5. AFM scan of a growing crack, height image: (a) start of growth; (b) end of growth. The crack tip is characterized by a deep depression. Following the crack tip is a shallow, linear depression that distinguishes the portion of the crack just after the crack tip has passed. Note the shallow depression is not of equal depth. The crack in this figure propagated from the bottom to the top of the figure.

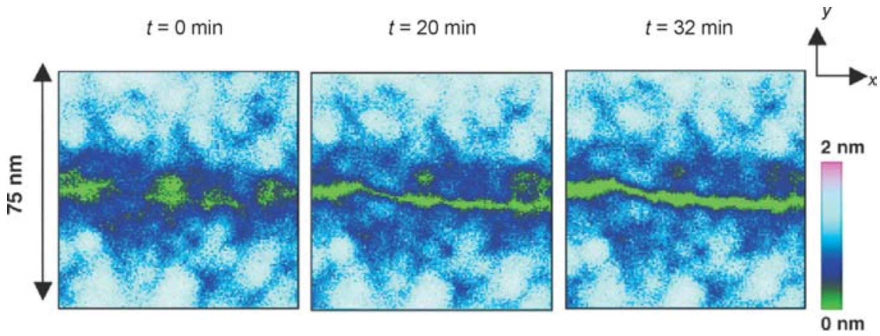

Fig. 6. Simulated images of a crack propagating through a glass surface. The surface of the image and the fracture surface have about the same roughness as the surfaces used in Ref. [5]. The time sequence between frames is almost the same as those in Ref. [5]. Compare this figure with Fig. 1. The crack growth direction is from left to right.

width is small compared to the probe tip size, the tip does not go very far into the crack. Rather, it sinks a certain amount; the wider the crack is opened, the more it sinks.

Superimposed upon these displacements are displacements due to the surface roughness. The trough, which is normally deeper than the average height of the surface, will appear to be even deeper where the crack has crossed a low point on the surface. This gives rise to the dark green spots that have been interpreted as cavities [5]. In a similar way, points on the original surface that were high will appear to be high after the crack has passed through them. As a consequence of the surface roughness, alternating light and dark features along the crack length then give the impression of cavities opening within the glass. With propagation, the crack widens and the "apparent cavities" join up assisting the crack growth.

The appearance of the trough is a little more complicated than implied above. The depth of the trough depends on the shape of the displacement field and, ultimately, on the choice made for the value of the applied stress intensity factor. Its magnitude is about the same or lower than the magnitude of the surface roughness. Furthermore, the depth of the trough diminishes as the crack tip goes by and the effect of the displacement field vanishes. When the crack tip is far enough away from the particular site being investigated, Fig. 6c, for example, the "width" of the trough depends only on the tip radius and the height of the stress-free surface through which the crack penetrates.

4. Discussion

Several experimental studies of crack motion in glass concluded that cracks move by the nucleation and coalescence of nanometer size cavities [5–7]. In those studies, moving cracks were imaged with an atomic force microscope that showed a trough extending beyond the crack tip; within the trough was a series of depressions that looked very much like growing cavities. These apparent cavities seemed to grow larger with time and eventually to join up to form the open crack.

Those crack growth studies suggested other studies that could be used to characterize or to qualify the original study. If cavities appear near the surface of the glass, and if cavitation is the main mechanism of crack growth, then cavity remnants should also appear on the fracture surface after the crack has passed, as they do for metals [8] and metallic glasses [19]. With this in mind Guin and Wiederhorn formed fracture surfaces in glass at controlled velocities and compared the two fracture surfaces using an atomic force microscope [9–11]. The conclusion of these studies is that, within the resolution of the experiment (6 nm in the plane of the fracture surface and 0.15 nm normal to that surface), there are no cavities formed when cracks propagate through glasses like silica or soda lime silicate glass. Fracture occurs in an entirely brittle manner.

An alternative explanation to the observation made in Refs. [5–7] is proposed. In the current study, a crack growth experiment and a fracture simulation were carried out. In the experiment, we followed the motion of indentation cracks propagating in soda lime silicate glass using the atomic force microscope, much in the same way as in [5–7]. Crack propagation was observed for extended periods of time (up to 7 h) and the position of the crack tip was recorded every minute or two. In these studies the crack tip could be identified by a surface depression that extends outwards from the crack tip. This surface depression moved with the crack from frame to frame. What is clear is that the shallow trough previously attributed to a plastic zone in front of the crack tip [5–7], actually follows the crack tip, Fig. 5b. This being said, we suggest that the "plastic zones" reported in Refs. [5–7] were not plastic zones at all, but portions of the open crack that were misidentified.

The simulation, Fig. 6, illustrates that the features observed in Refs. [5–7] could be duplicated by the displacements caused by the crack and the roughness of the specimen surface. When scanning a surface containing a crack, the probe tip will sense the open crack and be displaced in a negative direction as it passes over it. The amount of displacement will depend on the radius of the probe tip and the size of the crack opening. Superimposed on this displacement will be the roughness of the specimen surface. High points on the surface will result in shallow displacements as the probe passes over the crack. Low points on the surface will show up as deep displacements. The deeper displacements appear to be the cavities in Fig. 6. Thus, the observations that the shallow displacement trough always follows the crack tip, coupled with the fact that the simulation can duplicate observations in [5–7], support the suggestion that these observations are a consequence of a sharp crack and the roughness of the specimen surface.

One may conclude that our study is consistent with the argument that crack growth in glass occurs by processes that are entirely brittle. We see no evidence of nanoscale ductility or the formation of cavities at crack tips in glass.

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(Received August 2, 2007; accepted September 10, 2007)

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DOI 10.3139/146.101583
Int. J. Mat. Res. (formerly Z. Metallkd.)
98 (2007) 12; page 1170 – 1176

ISSN 1862-5282

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