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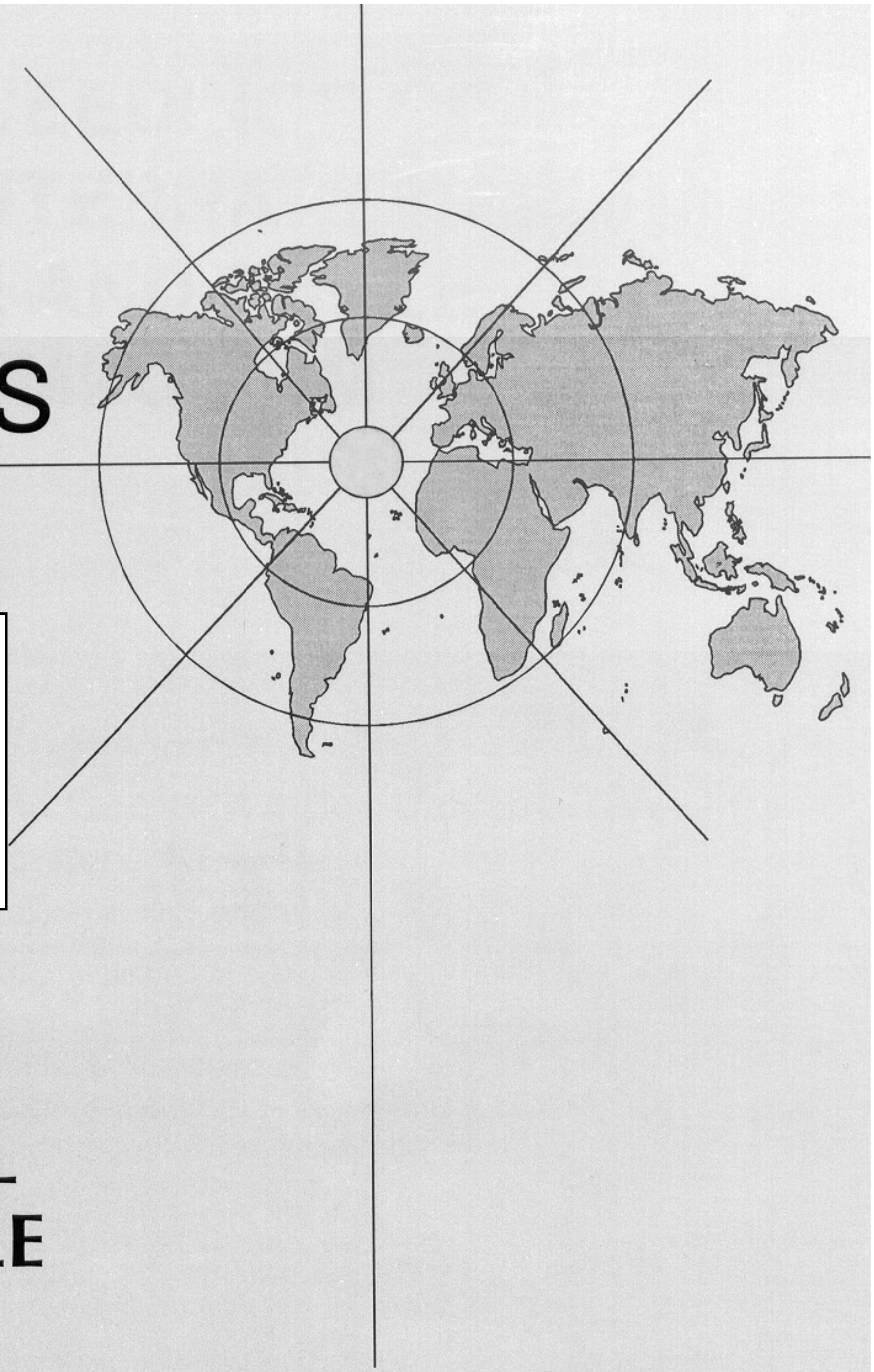

The Fatigue Behavior of Vickers Indentations in Fused Silica Optical Fibers

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THE FATIGUE BEHAVIOR OF VICKERS INDENTATIONS IN FUSED SILICA OPTICAL FIBERS

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ABSTRACT

Catastrophic fatigue failure of optical fiber in the field is expected to occur at the occasional weak defects. The behavior of these defects is different from that of high strength pristine optical fiber, and therefore it is desirable to study them directly rather than by extrapolating from the behavior of the pristine material. Because the probability of encountering a weak defect is very low, alternative means of studying these defects have been developed. We have used Vickers diamond pyramid indentations to introduce controlled flaws under controlled environmental conditions into fused silica of three geometries: 1000 μm diameter flat fiber, 1000 μm diameter cylindrical fiber, and 2 mm thick sheet glass. The resulting crack morphologies were studied using optical microscopy and SEM. Implications of these results to fiber optic reliability will be discussed.

INTRODUCTION

The behavior of weak fiber has often been modeled by deliberately damaging fiber by abrasion, or other techniques(e.g. ref. 1). Such studies give little detail of the nature and behavior of the flaws responsible for failure. In this study we use Vickers indentation to introduce a single dominant flaw of a predetermined size, shape, and location. In this way, the flaw can be examined before, during and after exposure to corrosive environments.[2] This provides information about the possible behavior of real weak defects which have key similarities, such as the presence of residual stress.

The Vickers indentation technique utilizes a diamond ground in the shape of a square based pyramid with an included angle of 136° between opposite faces. The indentation depth is about 1/7 the length of the diagonal, *i.e.* the indents are shallow. The indentation diagonal length, 2*a*, shown in Fig. 1, is used to measure the hardness, while the indentation radial crack length, 2*c*, is a measure of the material toughness.

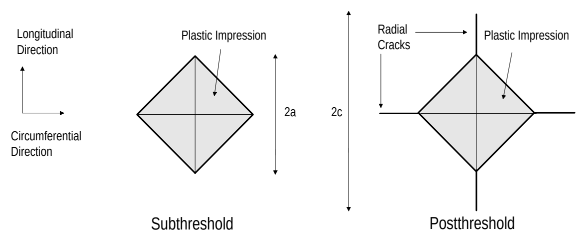

Fig. 1. Top view of indentation.

It is well established that a threshold load for radial crack formation exists. Low load indentations create crack-free plastic impressions and are termed "subthreshold" while higher load indentations with radial cracks are called "postthreshold".[3] Radial crack "pop-in" may occur some time after indentation resulting in a subthreshold to postthreshold transition with a corresponding loss in strength due to an increase in the effective flaw size. The pop-in of these strength-controlling radial cracks occurs over a range of loads which depends on the indentation environment, duration of indentation contact, surrounding environment after indentation, and the length of time after indentation. The pyramidal indenter causes plastic deformation in the glass which leaves a residual stress field upon unloading and acts as the driving force for radial crack pop-in. "Real" flaws, such as particles which adhere to the fiber surface during drawing, are likely to also have a residual stress field associated with them due to thermal expansion mismatch upon cooling. Abrasion damage will also have residual stress due to plastic deformation.

The purpose of this study is to examine the behavior of the Vickers indentation as a model flaw and to study the post-indentation pop-in behavior under various environmental conditions both during and after indentation.

EXPERIMENTAL PROCEDURE

All indentations were performed using a Leco M-400-G3 microhardness tester with loads ranging from 2 mN to 20 N (0.2 g-f to 2 kg-f). The peak indentation load was held for 10 s throughout. Indentations were performed inside an environmental chamber held at 100%, 50%, or ~ 0% humidity. Special experimental procedures were used in the case of the dry indentation experiments. Although not precisely measured, the lowest humidity was obtained using drierite in the chamber while flushing with dry N_2 gas, and finally, by blowing this gas directly onto the surface of the material at the indentation location. The amount of water in the N_2 gas was between 5-10 ppm. The chamber was equipped with an optical microscope for cursory evaluation after indentation. Samples were then placed in a vacuum desiccater while inside the chamber to avoid contact with environmental moisture during transport to either an optical microscope equipped with a camera or a scanning electron microscope.

Before indentation, the fiber specimens were stripped of their polymer coating by immersing in hot sulfuric acid (~ 200°C) for about 30 s followed by rinsing with water and acetone. This stripping method does not degrade the strength of the fiber.[4] 50 indentations were performed for each load on flat fused silica fiber. The evolution of the crack morphologies was studied with respect to aging time in 90°C pH 7 buffer solution.

Given the intricacy of the experimental techniques and the large numbers of specimens required for strength testing to obtain statistically significant results, several approaches have been used to speed data acquisition. While cylindrical fiber was used early in this work, we have also used flat fiber, which was fabricated by drawing from a preform whose sides have been ground flat and flame polished, as seen in Fig. 2. This fiber geometry has several advantages; indentations are much easier to produce on the flat surface since the Vickers indenter tip does not need to be accurately aligned with the apex of a curved surface. Furthermore, aligning the indenter tip with the fiber diameter is not necessary for flat fiber because the distance from the neutral axis during bending is the same across the flat. Also, when bent, the fiber automatically bends in the direction of minimum second moment of area, *i.e.*, the neutral axis is always parallel to the flats on the fiber. The indentation therefore automatically aligns on the surface of maximum tension. This obviates the need for careful azimuthal alignment of the indentation. However, there is a possibility that the residual stress fields of an indent on a flat fiber will be different from those on a cylindrical fiber due to the difference in geometry. Both specimen geometries have been found to give similar strength and fatigue results.[5,6]

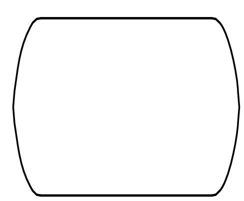

Fig. 2. Cross section of flat fiber.

A length of fiber was broken into 1000 specimens. Samples for any given experiment were then selected randomly from this batch in order to avoid any systematic variations along the fiber length. After stripping, each specimen was indented on the flat surface between two ink dots. The indentation diagonal, 2*a*, and radial crack length, 2*c*, (if present) were measured. The ink dots are used to identify the location of the indent during subsequent testing. For longer aging times an additional high load indent was applied at one end of the fiber to act as a marker for easy identification of the side of the fiber containing the indent of interest. This was necessary since the marker dots sometimes washed off after prolonged aging time in 90°C pH 7 buffer. Fibers were protected from contacting each other during aging by placing in a holder that keeps them separate. After aging, each fiber was rinsed with water and acetone to clean the surface and then re-examined under an optical microscope to observe if radial cracks had popped-in during aging. The fiber was then broken in a 4-point bend apparatus with the indent on the tensile side of the bend.[7,8] Finally, fracture surfaces of the two fragments were evaluated and then preserved for future reference. An example of a fracture surface for an indented flat fiber is shown in Fig. 3.

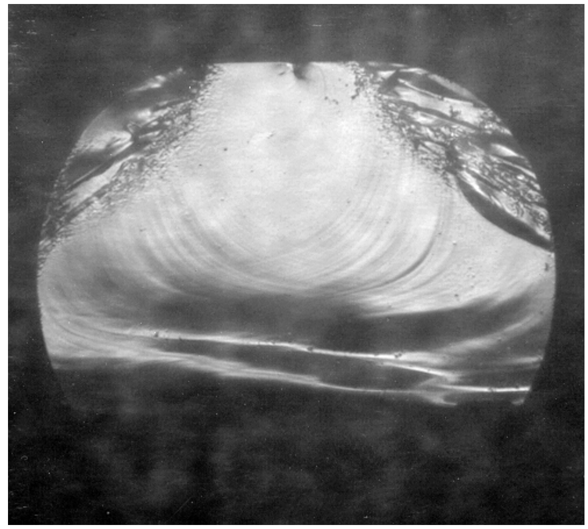

Fig. 3. Fracture surface of indented flat fiber.

RESULTS

The post indentation behavior for a 5 N load indentation performed while blowing 25°C dry N_2 onto the indent is drawn in Fig. 4. Upon unloading, a simple plastic impression is observed, as seen in Fig. 4a. If the dry N_2 is turned off, allowing some moisture to reach the indent, edge cracks appear at the indentation contact edge, as seen in Fig. 4b, and continue to grow over the next ~ 15 s, but do not reach the indentation corners, as seen in Fig. 4c. Much later (hours), radial cracks develop, as seen in Fig. 4d. These cracks emanate from near the corners and are therefore called secondary radial cracks.[9] They are different from the primary radial cracks which emanate from the corners and extend to longer lengths.

In comparison, in a 100% humidity, 25°C, environment, a 5 N indentation results in edge cracks that are immediately apparent after unloading and extend along the whole contact edge all the way to the corners. Thus, a 5 N indentation in fused silica is typically referred to as postthreshold. The typical apparent threshold load is between 2 N and 3 N for fused silica.[2,5] However, the severity of the radial crack pop-in is dependent on the environmental history during and after indentation. Indentation in a dry N_2 gas environment shows that apparent postthreshold indents are in fact subthreshold indents which have popped-in due to attack by ambient moisture for loads up to 20 N. Others have seen some similar results for inert conditions,[10] Also, the dry N_2 gas enables the kinetics of crack pop-in to be slowed for microscopic observation. Edge cracks, whose existence was speculated in a fracture mechanics model for indentation behavior[11] have been observed to form after unloading at the indentation contact edge.

The comparison of indentations made in dry N2 gas and humid environments give different crack morphologies. Even after the dry indent is exposed to moisture, the cracks that develop are principally secondary radial. This means that there is not a single type of defect for a given indentation load.

An example of typical delayed radial crack pop in behavior is shown in Figs. 5 and 6. Fig. 5 shows a 1000X optical micrograph of a 3 N indent performed in 50% humidity and 25°C. Some, but not all the edge cracks reached the indentation corners. This sample was then aged for 1 week in 90°C pH 7 buffer solution. The resulting radial cracks are shown in Fig. 6. All the radial cracks formed from the ends of the edge cracks, close to their initial position as shown in Fig. 5.

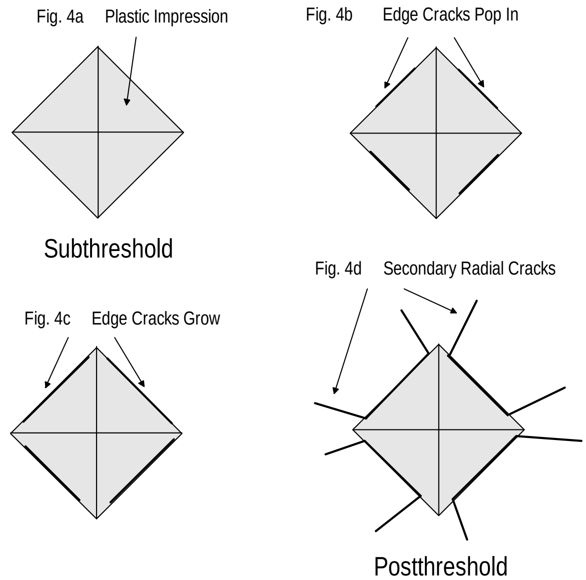

Fig. 4. Pop in sequence for dry N_2 indentation.

The radial crack pop-in behavior and the development of additional crack systems has been observed as a function of zero stress aging time in 90°C pH 7 buffer. 50 indentations for each load ranging from 5 mN to 3 N were placed on flat fused silica fiber. Each indentation was inspected after indentation, and after various aging times. The probability of radial crack pop-in as a function of aging time is shown in Fig. 7. The reported threshold load of 2 N to 3 N quickly produces radial cracks. However, at loads below the threshold, radial cracks are seen to pop in, even at loads as low as 50 mN. The consequence of this behavior may be significant. It is possible under very aggressive conditions to induce pop-in at loads far from the threshold.

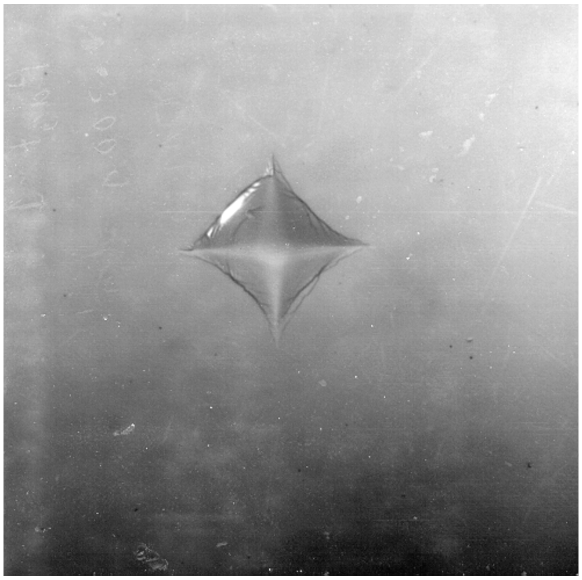

Fig. 5. 3 N indentation performed in 50% humidity on flat fused silica fiber (1000X).

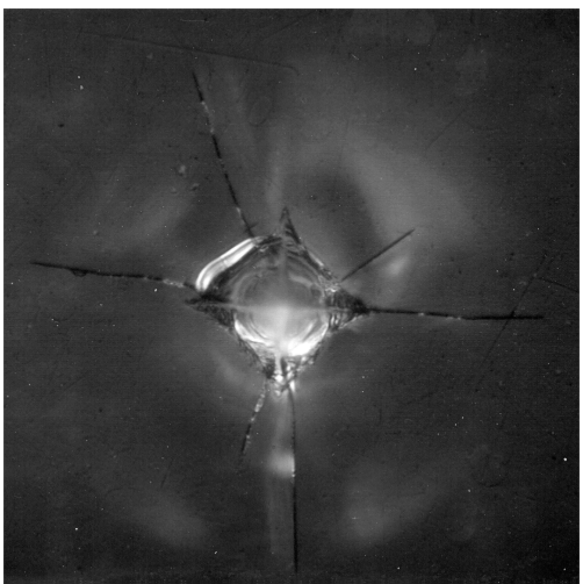

Fig. 6. Same 3 N indent after 1 week aging in 90°C pH 7 buffer environment (1000X).

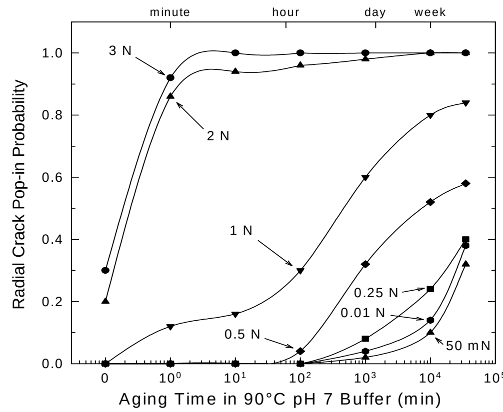

Fig. 7. Radial crack pop-in probability.

Except near the threshold, both subthreshold (low load) and post-threshold (high load) flaws show a strength increase with aging time.[5,6] These results are similar to observations on abraded fiber.[1] Two possible mechanisms for this increase have been proposed: crack tip blunting and reduction of residual stresses. Both of these mechanisms have been observed in this work. Upon careful examination of Fig. 6, a lateral crack, the light circular region under the indentation, can be seen. Laterals cracks were observed to pop-in some time after radial cracks, and even on indentations which never exhibited radial cracking. It was observed that these lateral cracks grew slowly with aging time thereby reducing the residual stress field associated with the plastic deformation zone.[12] This explains "weak" fiber increasing in strength with zero stress aging. However, another possibility is crack tip blunting. Figs. 8 and 9 show 8000X SEM images of 0.25 N indentations, which have been aged for 4 weeks in 90°C pH 7 buffer solution, with and without radial crack pop in. Fig. 7 shows that the probability of pop-in is around 40% and these images show examples of both behaviors. The edge cracks are etched away this aggressive environment. Additionally, the radial cracks in Fig. 9 appear, at least on the surface, blunt. An

SEM image of a 50 mN indentation at 8000x is depicted in Fig. 10. Here, the edge cracks popped in at different locations below the contact edge on the surface resulting in regions which were more aggressively attacked, or etched, by the 90°C pH 7 buffer environment.

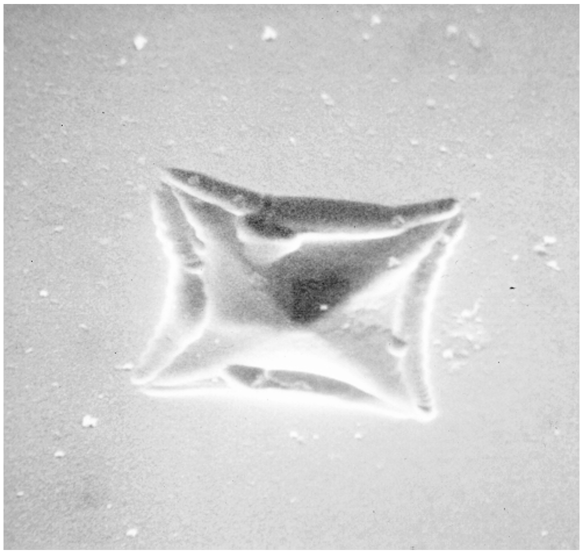

Fig. 8. SEM image of 25 mN indent aged 4 weeks in 90°C pH 7 buffer without radial cracks (8000X).

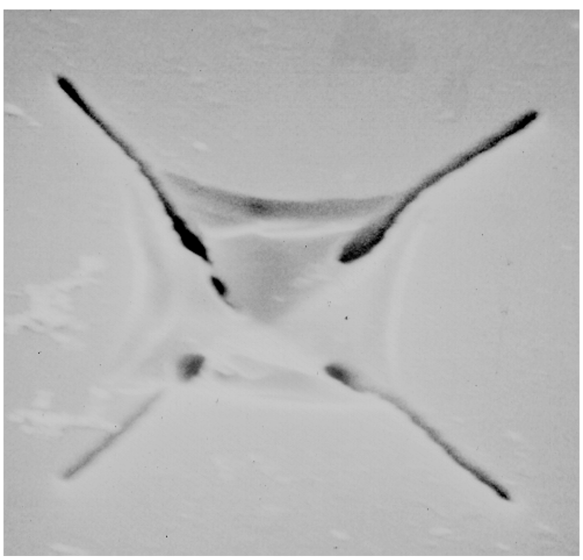

Fig. 9. SEM image of 25 mN indent aged 4 weeks in 90°C pH 7 buffer with radial cracks (8000X).

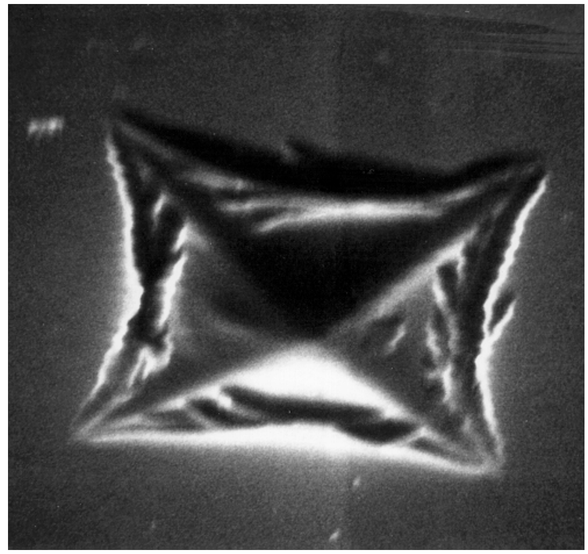

Fig. 10. SEM image of 50 mN indentation aged 4 weeks in 90°C pH 7 buffer (8000X).

DISCUSSION AND CONCLUSIONS

Contact damage on fused silica produces a complex system of cracks. The radial crack is the most important for reliability studies since it produces the greatest reduction in strength. This work shows that the initiation of radial cracks is controlled by edge cracks which form during or after indentation. The sequence of events leading to radial cracking is complex and is very sensitive to both the indentation environment and the post indentation conditions.

This work also provides direct evidence for the two possible mechanisms for strength recovery upon aging; namely residual stress relief and crack tip blunting. However, in the critical threshold region, radial crack formation, initiated by the mechanisms elucidated here, produces a discontinuous loss in strength with obvious practical implications.

The radial crack formation is sensitive to the boundary conditions of the indentation. The threshold load is slightly different for the three specimens geometries studied here. Also the indentation shape is important; a cube corner indenter initiates a 1 μm crack at loads as low as

0.02 mN.[9] The indentation defects we study here are in the strength range of typical proof loads. That is, they may model the kind of behavior exhibited by real practical defects. Clearly, the complex behavior of an indentation defect is not described by the Griffith relation for a simple stress free crack which is the basis for most optical fiber reliability models.

ACKNOWLEDGEMENTS

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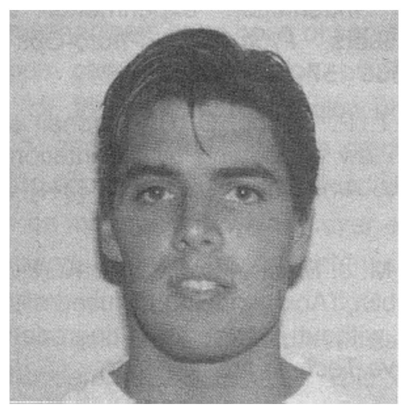

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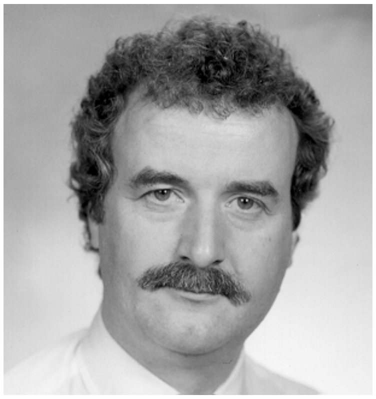

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John Matthewson received his BA in Theoretical Physics in 1975 from Cambridge University where he was a Kitchener Scholar and a Prize Scholar. He obtained MA and PhD degrees in 1978, also from Cambridge University, for his work at the Cavendish Laboratory on contact mechanics and high speed fracture. He then continued his research in this area as concurrently the Goldsmiths Junior Research Fellow at Churchill College, Cambridge and as a Science Research Council Postdoctoral Fellow. After three years as a consultant in the Cambridge University Computing Service, in 1984 he moved to AT&T Bell Laboratories as a postdoctoral member of technical staff where he worked on optical fiber strength and fatigue. From 1986 to 1989 he was an Advisory Engineer at IBM Almaden Research Center, San Jose, where he worked on reliability of magnetic recording devices and various aspects of adhesion. He is now an Associate Professor in the Fiber Optic Materials Research Program at Rutgers University where his research group is concerned with strength and fatigue of optical materials in general and oxide and non-oxide fibers in particular.