An indentation method for creating reproducible proof-stress level flaws in commercial optical fiber

G. Scott Glaesemann*, Donald A. Clark, James J. Price
Corning Incorporated

ABSTRACT

A technique was developed for obtaining proof-stress level flaws in commercial optical fiber with low variability in strength. It involves a novel method for stabilizing and protecting the round fiber prior to indentation. Indentation was performed in an automated fashion using a nano-indenter equipped with a cube-corner indenter. A Weibull modulus of 50 was achieved with a value of 100 over the lower portion of the distribution. This method will be useful in static fatigue testing of fiber with proof test level flaws.
Keywords: Optical fiber, reliability, strength, indentation

1. INTRODUCTION

It is common for researchers to use artificial flaws when studying the strength and fatigue behavior of proof-stress level flaws on optical fiber. This is due to the infrequency of flaws with strength near the proof-stress level on standard telecommunications fiber. Two on-draw abrasion techniques have been used for creating artificial flaws on fiber: contacting the glass surface with another fiber before the coating is applied[1] and passing fiber over an abrasive pulley before applying the coating.[2] Particle abrasion after removal of the coating has been employed and is fashioned after a standard ASTM test method.[3] Strength distributions of draw-abraded and particle abraded fiber are shown in Weibull fashion in Figure 1. The resulting Weibull modulus for both methods is near 20, at the upper end of what is typical for mechanical abrasion methods.[4]

Vickers indentation is a common method for creating proof test level flaws in fiber. Here one removes the coating and presses a Vickers micro-hardness indenter against the fiber surface to create surface flaws.[5,6] Flaws produced by this method resulted in a strength distribution with a Weibull modulus of around 8.[5]

The usefulness of artificial flaws in studying the fatigue behavior of optical fiber is limited by the ability to reproducibly create these flaws. Ritter et al.[7] showed how the statistical confidence of fatigue parameters relates to the strength distribution. For example, with dynamic fatigue testing the measured fatigue strength, σ_f, is related to the initial as-abraded strength distribution, S_i, by $\sigma_f \propto S_i^{\frac{n-2}{n+1}}$; and therefore, variability in measured fatigue strength is close to that of the initial strength distribution. In this case, a Weibull modulus of 25 will produce sufficient confidence in the fatigue parameter, n, provided the sample size for each stressing rate is 15 to 20 specimens.

Static fatigue testing requires m values on the order of 50 to 100. The time to failure, t_f, relates to the initial strength as $t_f \propto S_i^{n-2}$. Thus, a small change in strength results in a large change in the time to failure. The inability to achieve high Weibull modulus has limited the usefulness of static fatigue testing of abraded or indented optical fiber. For a demonstration of this point, see reference 8.

* glaesemags@corning.com; (607) 524-6737; http://www.corning.com/opticalfiber; Corning Incorporated, SPTD022, Corning, NY , USA 14831

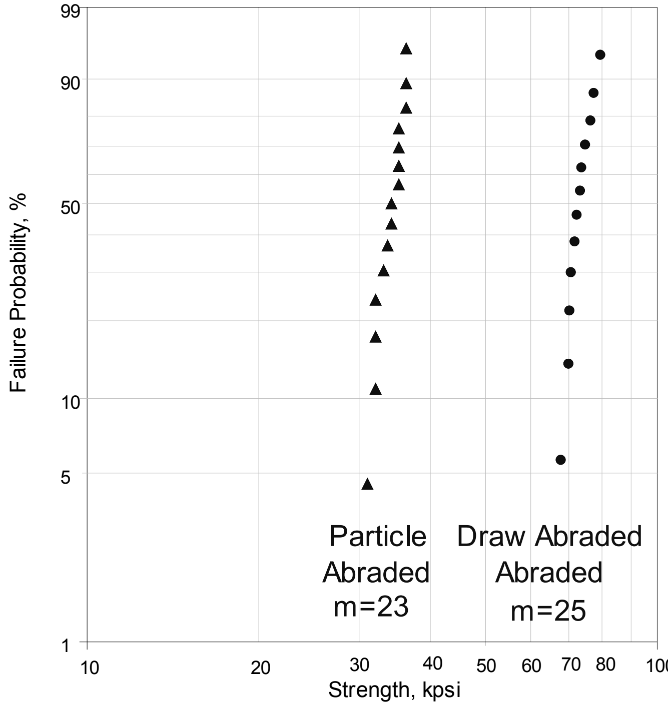

Figure 1. Strength distributions of abraded fiber.[1]

The reproducibility of indentation flaws is difficult and has been the subject of a several investigations.[9,10,11] Factors that affect the reproducibility of Vickers indentation flaws in glass are the repeatability of the indentation loading cycle, the presence of secondary cracking (lateral and Hertzian), the crack initiation threshold, fatigue during indentation and loading to failure, as well as specimen and indenter cleanliness.[12,13,14,15] Lin and coworkers[16] suggest that the difficulty inherent in positioning an indenter tip directly over the curved surface of an optical fiber is also a key factor. If slightly off center, the indenter tip can slip to one side making flaw reproducibility difficult. To facilitate indentation they used fiber with flat sides. This fiber was created by first cutting two parallel flats on opposite sides of a glass blank and then drawing the blank into fiber at a low temperature. Figure 2 is an image of such a fiber.[11] Semjonov et al.[11,17] indented flat fiber with a cube-corner low-load indenter and achieved Weibull moduli ranging from 30 to 70. Note that a cube-corner indenter displaces three times as much volume for a given contact area than a Vickers indenter, and thus, generates greater contact residual stress. They found that radial cracks were present down to 0.2 gram indentation loads. With their combination of flat fiber, low-load indentation machine, cube-corner indenter, and sample preparation procedure they were able to create proof stress level flaws with less variability in strength than those created by Vickers indentation or mechanical abrasion. It has yet to be shown that such strength distributions can be achieved on standard commercial fiber. The purpose of this paper is to describe and demonstrate an experimental method for creating a well-controlled distribution of proof-stress level flaws on commercial optical fiber.

2. EXPERIMENTAL APPARATUS

2.1 Fiber preparation

The fiber used in this study was a standard 125 micron diameter single-mode silica-clad fiber. A 25 mm length of polymer coating was removed from the middle of a meter long length of fiber. The coating was removed by exposing the fiber to 180 to 200°C sulfuric acid for 10 to 20 seconds. The fiber was then rinsed in distilled water for 5 seconds before indentation. If done properly, this method of coating removal is known to cause little or no degradation of the glass surface.[18]

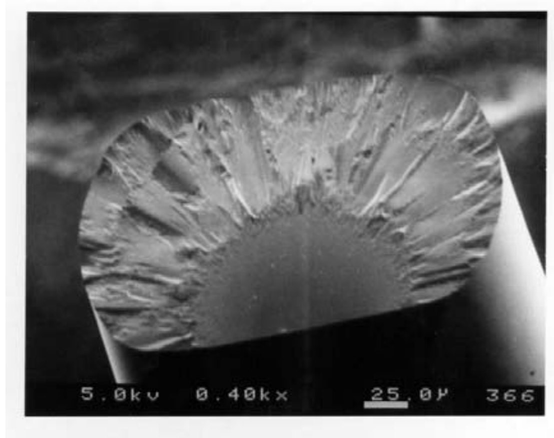

Figure 2. Flat fiber has been used in an effort to facilitate the indentation process.[11]

2.2 Preparation for Indentation

Of particular importance in indenting circular fiber is the method of stabilizing the fiber during the indentation process. If the fiber moves, the indenter is prone to slide to one side of the fiber. Grooved glass substrates are commonly used in aligning fiber for optical measurements. Schematics of one such substrate,[α] optimized for 125 micron diameter optical fiber, is shown below.

Side View Top View

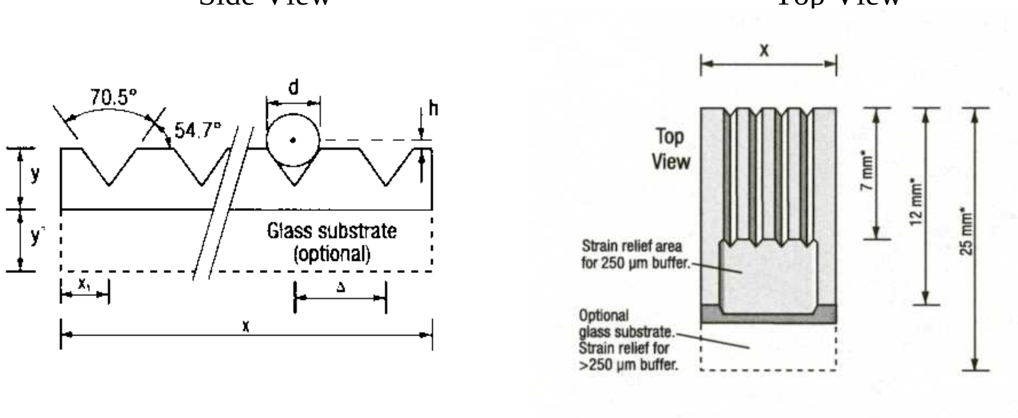

Figure 3. Schematics of grooved substrates used to stabilize commercial fiber.

A low viscosity uncured polymer coating was applied to the grooved surface of the substrate with a small brush. This provides a soft, clean cushion for the bare glass fiber. The thickness of this layer was sufficient to cover the grooves in the substrate.

The center-stripped section of fiber was manually placed in a groove in the substrate while being viewed through a stereo-microscope. Note that the uncured coating was not allowed to wick over the top of the fiber during this process and care was taken not to contact the bare glass surface prior to placement in the groove. The polymer cushion was then cured by exposure to ultra-violet light, thereby, securing the bare

[α] Wave Optics, Mountain View, CA

fiber in place. A picture of fiber aligned in the substrate is shown below. The procedure was repeated until 20 grooves in the substrate were filled with fiber.

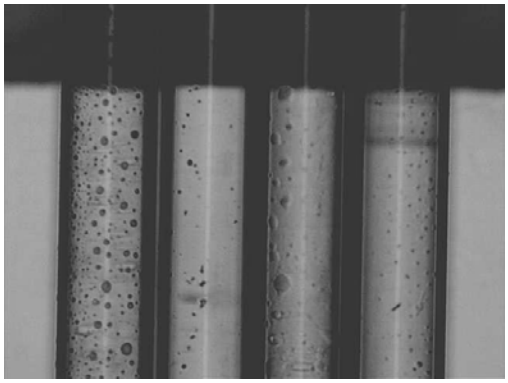

Figure 4. Bare fiber is shown to be well aligned in the grooved substrate. A soft polymer layer beneath the fiber stabilizes the fiber in the groove while keeping the underside from being damaged.

2.3 Indentation

A nano-indentation apparatus[γ] equipped with a cube-corner indenter was used to create proof-stress level flaws on the fiber. This highly instrumented device is designed to operate at low indentation loads with precise control over the indentation process. More detailed descriptions of the instrumentation can be found in the literature.[19,20] A schematic of this instrument is shown in Figure 5.

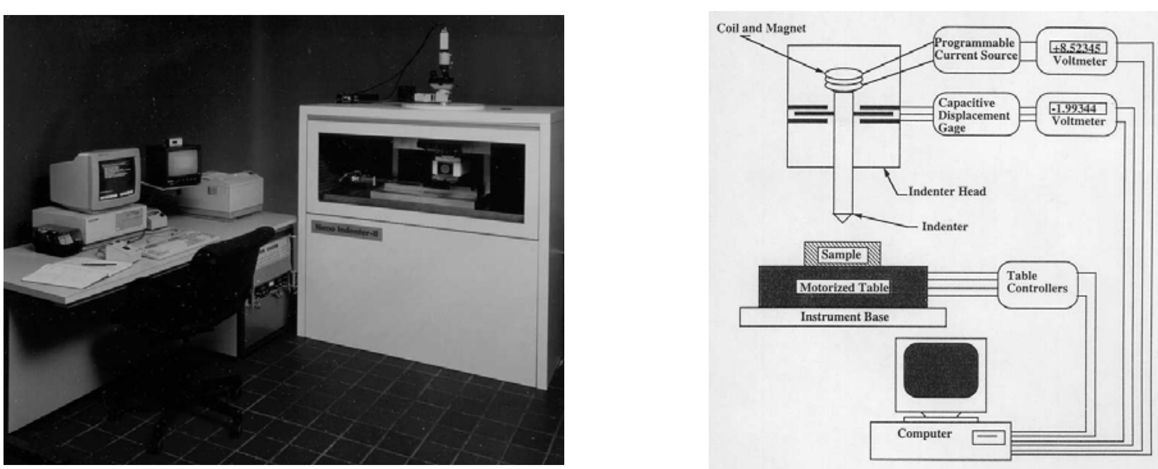

Figure 5. Image and schematic of the nano-indenter used in this study.

[γ] Nano-Indenter II, MTS Systems Corp., Eden Prarie, MN

This indentation instrument allows the position of the indentation site to be selected using reflected light microscopy with differential interference contrast (DIC) capability. By focusing on the transversely mounted fibers at a magnification from 100 – 1700X, one can precisely determine the location of the apex of the fiber circumference. Once the indentation location is selected for each fiber, the grooved substrate containing the specimens can then be translated, via precision stages, to a position aligned with the indenter tip. Calibration of the precision stages permitted indents on optical fiber to be located within one micron of the selected location.

A diamond cube-corner indenter was used to create surface flaws. The cube-corner indenter has the configuration of a three-sided pyramid with an angle of 35.26° between the axis of symmetry and the faces of the indenter. This indenter geometry was selected because of its ability to generate well-defined crack systems at low loads. Also, the small contact area is well suited for indenting a small region like the apex of a fiber. Since strength testing of the fiber was to be done in uniaxial tension, the cube-corner indenter was oriented such that one of the indenter corners pointed perpendicular to the fiber axis.

For this work, the load was applied using a constant loading rate (P') per unit load (P) of $P'/P = 0.025\ s^{-1}$. Upon reaching a peak load of 9.8 mN (1 gf), the indenter was unloaded at a rate of P' = 0.220 mN/s. The load and displacement were monitored and recorded continuously throughout the process. After indentation of one fiber, the stage automatically indexes to the predetermined indentation location for the next fiber. A typical load-displacement response curve from the indentation cycle is shown below. Figure 7 is an SEM micrograph showing a typical cube-corner indent on the surface of the fiber. The size is approximately 5 to 6 μm.

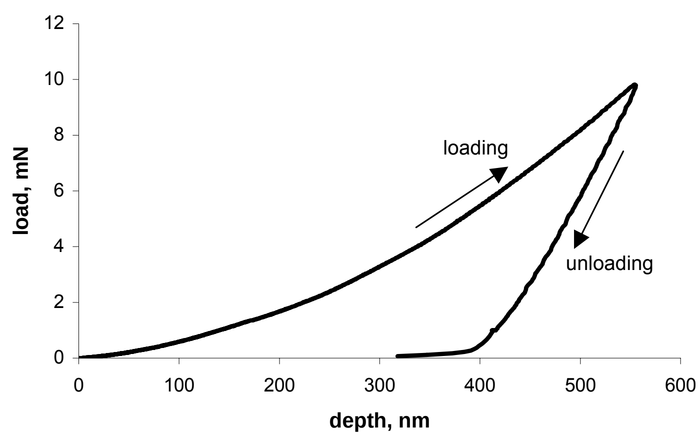

Figure 6. Load displacement curve for a cube-corner indentation load of 1 gram.

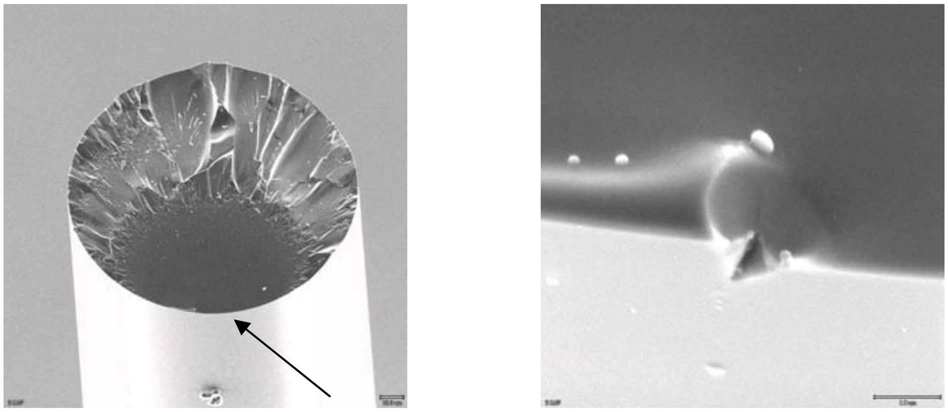

Figure 7. Fracture surface of fiber with a cube-corner indent (left). The plastic impression generated by the indentation event is shown on the right.

Tensile testing was performed on a universal testing machine[β] at a crosshead speed of 10 mm/min. This resulted in a stressing rate of approximately 14 kpsi/sec (0.1 GPa/s). The specimens were preconditioned in the test environment and all testing was performed under controlled lab ambient conditions.

The resulting strength distribution is plotted in Weibull fashion in Figure 8 and is compared to the cube-corner indentation of flat fiber from reference 11. A Weibull modulus, m, of 52 was obtained for all data

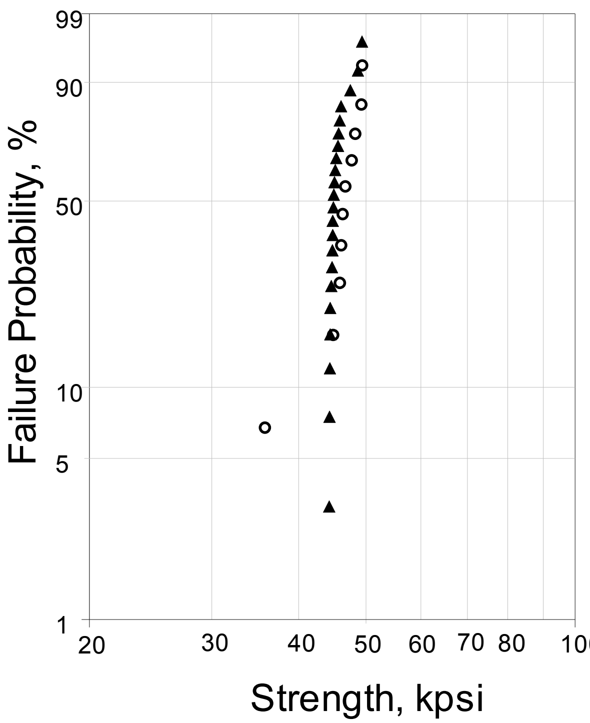

Figure 8. Strength distribution of cube-corner indented commercial fiber compared to that of flat fiber from reference 16. ▲– this study, o – from ref. 11.

[β] Instron Corp., Canton, MA

points. The strongest data points departed slightly from a straight line. The weakest strengths are most important in static fatigue testing, as it is common to suspend testing once half the specimens have failed. The Weibull modulus for the weakest half of the specimens is close to 100. This is nearly equal to that of pristine fiber. With this indentation procedure it is possible to artificially create proof stress level flaws with Weibull moduli close to that of pristine fiber. This makes these flaws particularly suitable for static fatigue testing.

In Figure 9 the data from cube-corner indentation of commercial fiber is plotted along with the abraded fiber from reference 1. Whereas the draw abraded and post-draw abraded flaws more closely represent those created during manufacturing and handling, the indentation flaws created in this study have the advantage of a narrow strength distribution.

The indentation method used here focused on four indentation events that can be controlled; namely, the use of a "sharp" cube-corner indenter, a well controlled indentation loading cycle using a nano-indenter, a rigorous specimen alignment procedure, and protective support of the fiber specimen beneath the indenter. It is not known which of these factors is most important.

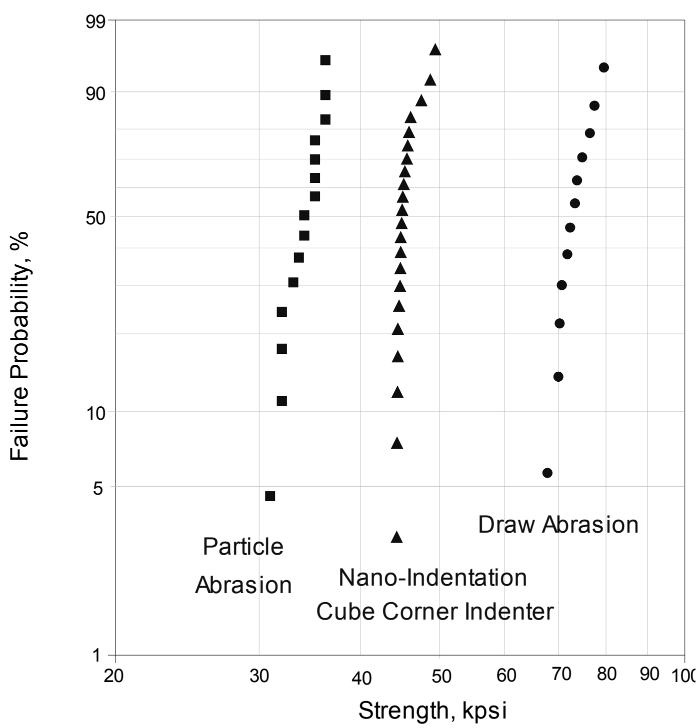

Figure 9. Strength distribution of cube-corner indented fiber compared to that of abraded fiber from reference 1.

3. SUMMARY

Building on previous success with flat fiber,[11] a technique was developed for obtaining proof stress level flaws in commercial optical fiber with low variability in strength. It focused on four events under the operator's control. A novel method for stabilizing and protecting the round fiber prior to indentation was developed. Specimen alignment was carefully controlled. Indentation was performed with a nano-indenter capable of a highly repeatable loading cycle and a "sharp" cube-corner indenter was used to produce the flaws.

A Weibull modulus of 50 was achieved with a value of 100 over the lower portion of the distribution. This method is useful for static fatigue experiments of fiber with proof test level flaws as well as fundamental studies of fiber fracture from lower strength flaws.

Flaws created by abrasion or contamination can closely replicate actual flaw inducement events during manufacturing and handling, and therefore, have an advantage over indentation flaws. However, this study shows that indentation flaws created by nano-indentation with a cube-corner indenter can have significantly reduced variability in strength. Until such strength distributions can be achieved with abrasion methods, these indentation flaws are well suited for static fatigue studies.

REFERENCES

[1] G.S. Glaesemann, "The Mechanical Behavior of Large Flaws in Optical Fiber and Their Role in Reliability Predictions," *Proceedings of the 41st International Wire and Cable Symposium*, 689-704, Reno, NV, 1992.

[2] S. Sakaguchi and Y. Hibino, "Fatigue in Low-Strength Silica Optical Fibres," *J. Mat. Sci.* **19**, 3416-3420 (1984).

[3] ASTM D968-93 01-Nov-1993, Standard Test Method for Abrasion Resistance of Organic Coatings by Falling Abrasive.

[4] G.S. Glaesemann, "Advancements in Mechanical Strength and Reliability of Optical Fibers," *Proc. Soc. Photo-Opt. Instrum. Eng.* **CR73** 1-23 (1999).

[5] T.P. Dabbs and B.R. Lawn, "Strength and Fatigue Properties of Optical Glass Fibers Containing Micro-Indentation Flaws," *J. Am. Ceram. Soc.* **68** 563-569 (1985).

[6] K. Jakus, J.E. Ritter, Jr, S.R. Choi and T.J. Lardner, "Failure of Fused Silica Fibers with Subthreshold Flaws," *J. Non-Cryst. Solid* **102** 82-87 (1988).

[7] J.E. Ritter, Jr., N. Bandyopadhyay, and K. Jakus, "Statistical Reproducibility of the Dynamic and Static Fatigue Experiments," *J. Am. Ceram. Soc.* **60** [8] 798-806 (1981).

[8] T. Svensson and A. Breuls, "Strength and Fatigue of Different Kind of Weak Spots from the Manufacture of Optical Glass Fibres," *SPIE* **2290**, 211-219 (1994).

[9] J. Gong, Y. Chen, and C. Li, "Statistical Analysis of Fracture Toughness of Soda-Lime Glass Determined by Indentation," *J. Non-Cry. Solid.* **279** 219-223 (2001).

[10] G.S. Glaesemann, K. Jakus, and J.E. Ritter, Jr., "Strength Variability of Indented Soda-Lime Glass," *J. Am. Ceram. Soc.* **70** (6) 441-444 (1987).

[11] S.L Semjonov and C.R. Kurkjian, "Strength of Silica Optical Fibers with Micron Size Flaws," *J. Non-Cry. Solid.* **283** 220-224 (2001).

[12] B.R. Lawn and R. Wilshaw, "Indentation Fracture: Principles and Applications," *J. Mat. Sci.* **10** 1049-1081 (1975).

[13] K. Jakus, J.E. Ritter, Jr., S.R. Choi and T.J. Lardner, "Failure of Fused Silica Fibers with Subthreshold Flaws," *J. Non-Cry. Solid.* **102** 82-87 (1988).

[14] S. Lathabai, J. Rodel, T. Dabbs, and B.R. Lawn, "Fracture Mechanics Model for Subthreshold Indentation Flaws," *J. Mat. Sci.* **26** 2157-2168 (1991).

[15] B. Lin and M.J. Matthewson, "Inert Strength of Subthreshold and Post-Threshold Vickers Indentations on Fused Silica Optical Fibres," *Phil. Mag. A* **74** (5) 1235-1244 (1996).

[16] B. Lin, A.P. Stanzeski, and M.J. Matthewson, “Modeling of Extrinsic defects in Silica Fibers Using Vickers Indentation,” *Proc. Soc. Photo-Opt. Instrum. Eng.* **2611** 122-128 (1995).
[17] S.L. Semjonov, G.S. Glaesemann, C.R. Kurkjian and M.M. Bubnov, “Modeling of proof test level flaws using cube corner indents,” *47th Int. Wire & Cable Symp. Proc.*, pp. 928-932 (1998).
[18] M.J. Matthewson, C.R. Kurkjian and J.R. Hamblin, “Acid Stripping Of Fused Silica Optical Fibers Without Strength Degradation,” *J. Lightwave Tech.*, **15** [3] 490-497 (1995).
[19] W.C. Oliver, “Progress in the Development of a Mechanical Properties Microprobe,” *MRS Bulletin*, **11** [5] 15-19 (1986).
[20] W.C. Oliver and G.M. Pharr, “An Improved Technique for Determining Hardness and Elastic Modulus using Load and Displacement Sensing Indentation Experiments,” *J. Mat. Res.* **7** [6] 1564-1583 (1992).