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Strength of silica optical fibers with micron size flaws

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Abstract

Silica fibers have been indented with a diamond having a cube-corner tip. The tensile strengths of fibers indented with loads from 0.2 to 10 g were 465–130 MPa, respectively, with Weibull m -values ~ 30 –70. Radial cracks (0.5–6.5 μm) were produced in all cases, with no transition to sub-threshold behavior at the lowest load (0.2 g) employed. © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

While the intrinsic strength of silica fibers is known to be ~ 14 GPa, most commercial proof-testing of communications lightguide fibers is carried out at 0.7 GPa. This proof-testing is done to remove the relatively infrequent flaws that develop during the manufacturing processes. Using the standard fracture mechanics equation (strength is inversely proportional to the square root of the length of a sharp crack) a crack length of 1 μm is estimated to be responsible for this strength reduction. It is generally agreed that such flaws are caused either by mechanical damage or by the interaction of the glass surface with refractory particles. These flaws occur infrequently in today's high-quality fiber and it is difficult to study them directly. In order to gain some insight

into their general behavior, two types of 'synthetic' flaws have been studied in the past: abrasion-produced flaws and flaws generated by refractory dust particles either introduced into the furnace or painted onto the preform surface. While such flaws have led to some interesting results, their size and distribution are difficult to control in detail.

An approach which has been successfully employed in the study of mechanically induced flaws is the use of static indentation. The indenter most commonly used (Vickers) unfortunately gives rise to flaws which are much larger (15–20 μm) than 1 μm . Early studies by Taylor [1] and Abe [2] showed that indentations could be produced in oxide glass plates without cracking if sufficiently low loads (25 g) were employed. In 1974 Baikova et al. [3] measured the strengths of etched silica and soda lime glass plates after indenting with a Vickers indenter. They found that the strength decreased discontinuously as the indenting load was increased. The discontinuity coincided with the appearance of cracks. Subsequently, Dabbs et al. [4] and others [5] studied this behavior in high-strength silica fibers and termed the conditions sub- and post-threshold. The exact load at which

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cracks appear depends on many experimental parameters. The condition of the silica surface, the relative humidity of the atmosphere in which the indentation is carried out, the length of time allowed for the indentation, as well as the length of time elapsed after the indentation has been completed, i.e., the pop-in time, all affect the observed behavior.

These studies are of considerable interest and importance to the understanding of the entire fracture process in silica glass and glasses in general. From the point of view of studies of light-guide fibers, however, the flaws produced by the Vickers indenter are too large to serve as a model for proof-test level flaws (~ 0.7 GPa strength and ~ 1 μm long cracks). Recent work by Pharr et al. [6], however, showed that sub-micron cracks could be produced using a cube-corner-shaped diamond if loads less than grams were employed. The use of

such an indenter then, would appear to allow the production of simple radial cracks of the desired length, and thus detailed studies of proof-test sized flaws could be made. In the present study, we employ a ‘cube-corner’ indenter for the development of proof-test size flaws directly on silica fiber.

2. Experimental procedure

Fibers roughly rectangular in shape (ranging in size from 125/250 to 400/800 μm) with two flat sides were drawn and coated in-line with an acrylate polymer especially for these experiments in order to simplify the indentation process. The polymer coating was removed with hot sulfuric acid from 2 to 3 cm of the center of the fiber sample and a single indent was made. Indentations were made using a low-load indentation instru-

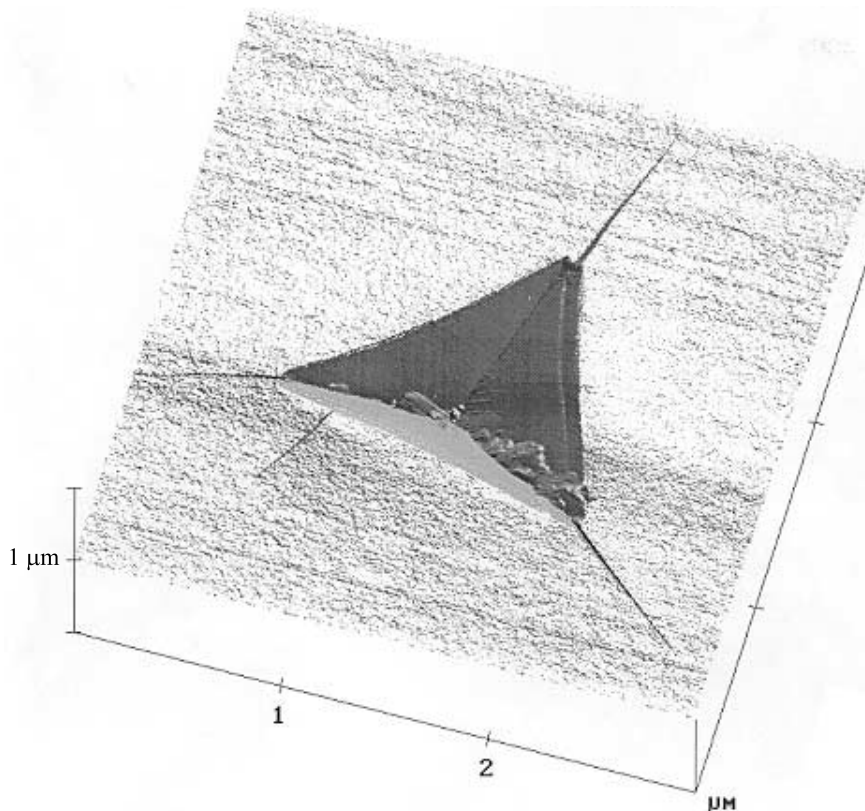


Fig. 1. AFM image of 1 g indent.

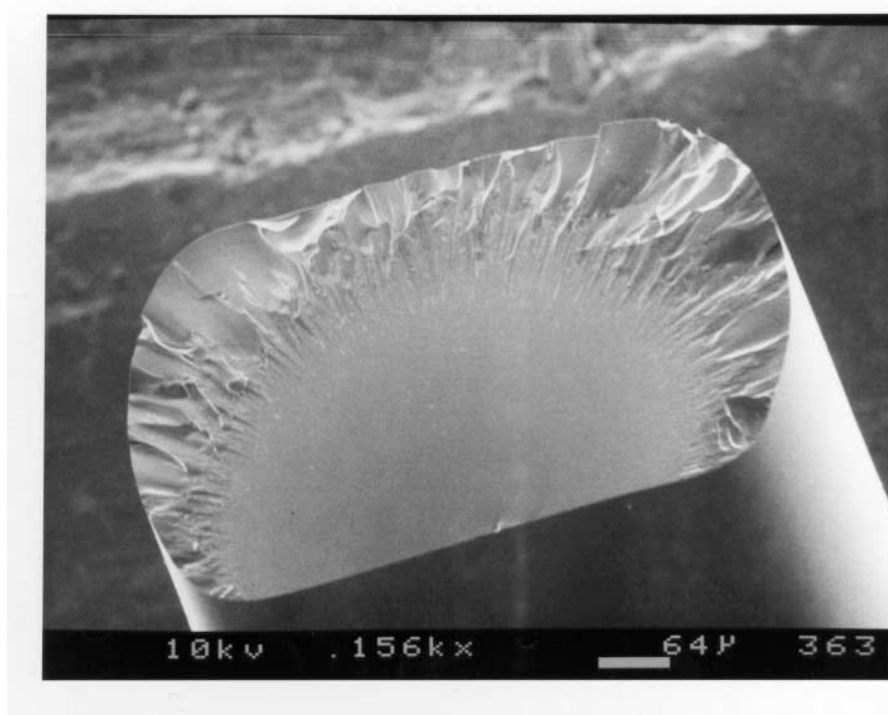


Fig. 2. SEM image of fiber indented with 10 g load.

Table 1
Strength and crack length measurements

Load (g)	Average measured crack length (μm)	Median strength (MPa)
10	6.52	132
5	5.24	164
3	3.42	180
2	2.35	208
1	1.75	269
0.5	0.98	351
0.3	0.75	378
0.2	0.55	465

ment (Leco, Model M400-G-3, St. Joseph, MI 49085, USA) and a diamond cube-corner indenter (Nano Instruments, Oak Ridge, TN 37830, USA). This indenter produces a triangular shaped impression with radial cracks extending from the three corners. A typical atomic force microscope (AFM) image of such an impression is presented in Fig. 1. Loads from 0.2 to 10 g were employed. After indenting, these fibers were tested at room temperature and ambient humidity ($\sim 50\%$) in

tension in a bench model tensile tester (Model 1130, Instron, Canton, MA 02021, USA) at a strain rate of 5% per minute. A drop of ink on the fiber surface near the indent aided in locating the indentation and ensuring that the sample was broken at that point. The indentation was made so that one of the radial cracks was perpendicular to the fiber axis. Surface traces of the cracks were measured on AFM images of untested fibers. An SEM image of a typical fracture surface is shown in Fig. 2.

3. Results and discussion

Well-defined radial cracks (as in Fig. 1) were observed by AFM for all indenting loads (from 0.2 to 10 g). Results of crack size measurements and strength testing at ambient conditions are presented in Table 1. Crack sizes estimated from the measured strengths are $\sim 1/3$ those shown in this Table. This results from the measurement of the

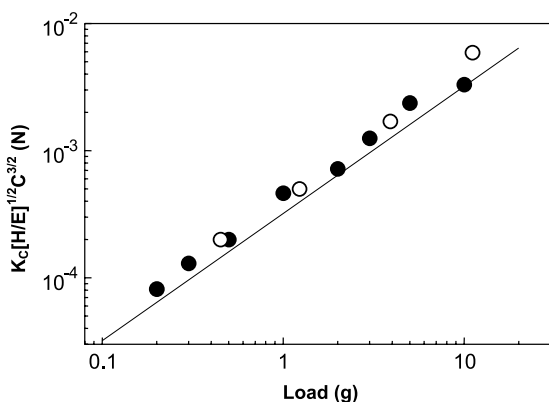


Fig. 3. Dependence of crack size on indentation load: (●) current data, (○) data from Pharr et al. [6], solid line – according to Eq. (1) and parameters from Pharr et al. [6] ($K_C = 0.58 \text{ MPa m}^{1/2}$, $E = 72 \text{ GPa}$, $H = 8.9 \text{ GPa}$, $\alpha = 0.32$).

surface trace rather than the actual propagating crack as well as the fact that crack growth occurs during the strength measurement at room temperature.

Pharr et al. [6] showed that crack radius c and indenting load P could be related in the following way:

$$K_C = \alpha \left(\frac{E}{H} \right)^{1/2} \left(\frac{P}{c^{3/2}} \right), \quad (1)$$

where K_C is a critical stress intensity factor, E the Young’s modulus, H the hardness, and α is an empirical constant depending on geometry of indenter. For silica glass and a cube-corner indenter, they used the following constants: $K_C = 0.58 \text{ MPa m}^{1/2}$, $E = 72 \text{ GPa}$, $H = 8.9 \text{ GPa}$, $\alpha = 0.32$. Fig. 3 demonstrates good agreement of our results with those obtained by Pharr et al. [6].

From the Griffith or fracture mechanics equations,

$$\sigma \sim \frac{1}{\sqrt{c}}. \quad (2)$$

Consideration of Eq. (1), results in

$$\sigma \sim P^{-1/3}. \quad (3)$$

Fig. 4 shows that our results are in good agreement with this relation. The linearity shown here

demonstrates that there is no sub-threshold to post-threshold transition in the load region investigated here. This is in contrast to the behavior found with Vickers indents. The load for a given behavior (that is, cracking and strength) is two orders of magnitude smaller in the case of the cube-corner indenter. This behavior has been explained by Pharr et al. [6], by noting that for a given contact area, the cube-corner indenter displaces three times more volume than does the Vickers indenter. Since the stress that is generated scales with the volume displaced, the cube-corner indenter will generate higher stresses for a given load.

The $-1/3$ power relation between the indenting load and the fiber strength means that the strength

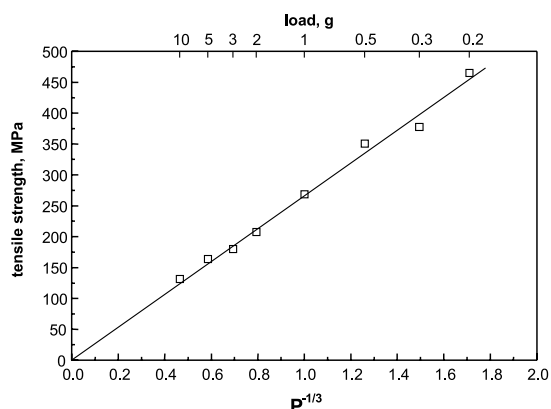


Fig. 4. Dependence of median strength of indented fibers on indentation load.

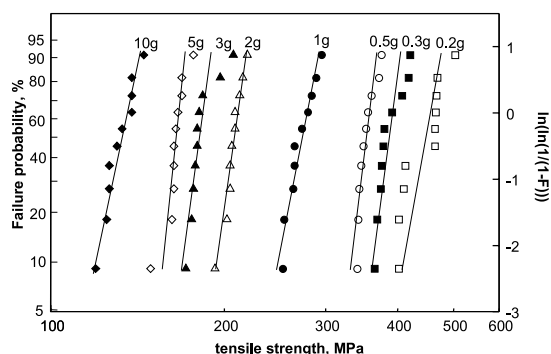


Fig. 5. Ambient strength of indented fibers at different indentation loads.

varies slowly with the indenting load. As a result, narrow strength distributions are obtained for samples indented at the same load (Fig. 5). Weibull m -parameters are in the range of 30–70. Samples with a narrow strength distribution and specific placement of the defect are very attractive for study of mechanical properties of proof-test level flaws. Using such fibers, at least one type of flaw that has proof-test strength can be characterized in an effort to more completely understand their behavior. Additional studies of the strength as a function of time and strain rate (i.e., fatigue), and at low-temperature (77 K) as well as after aging, are being carried out in order to understand the detailed behavior of these ‘proof-test’ flaws [7].

4. Summary

We have shown that by using a cube-corner indenter, strengths of the order of normal proof-

test levels (~ 700 MPa) can be obtained with tight distributions (Weibull parameter ~ 30 –70). No sub- to post-threshold transition was observed with indentation loads from 0.2 to 10 g.

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