Effects of accelerated aging on fiber damage thresholds

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ABSTRACT

Laser-induced damage mechanisms that can occur during high-intensity fiber transmission have been under study for a number of years. Our particular interest in laser initiation of explosives has led us to examine damage processes associated with the transmission of Q-switched, Nd:YAG pulses at 1.06 μm through step-index, multimode, fused silica fiber. Laser breakdown at the fiber entrance face is often the first process to limit fiber transmission, but catastrophic damage can also occur at either fiber end face, within the initial "entry" segment of the fiber, and at other internal sites along the fiber path. Past studies have examined how these various damage mechanisms depend upon fiber end-face preparation, fiber fixturing and routing, laser characteristics, and laser-to-fiber injection optics. In some applications of interest, however, a fiber transmission system may spend years in storage before it is used. Consequently, an important additional issue for these applications is whether or not there are aging processes that can result in lower damage thresholds over time. Fiber end-face contamination would certainly lower breakdown and damage thresholds at these surfaces, but careful design of hermetic seals in connectors and other end-face fixtures can minimize this possibility. A more subtle possibility would be a process for the slow growth of internal defects that could lead to lower thresholds for internal damage.

In the current study, two approaches to stimulating the growth of internal defects were used in an attempt to produce observable changes in internal damage thresholds. In the first approach, test fibers were subjected to a very high tensile stress for a time sufficient for some fraction to fail from static fatigue. In the second approach, test fibers were subjected to a combination of high tensile stress and large, cyclic temperature variations. Both of these approaches were rather arbitrary due to the lack of an established growth mechanism for internal defects. Damage characteristics obtained from fibers subjected to each of these aging environments were compared to results from fresh fibers tested under identical conditions. A surprising result was that internal damage was not observed in any of the tested fibers. Only breakdown at the fiber entrance face and catastrophic damage at both end faces were observed. Fiber end faces were not sealed during the accelerated aging environments, and thresholds at these faces were significantly lower in the aged fibers. However, most fibers transmitted relatively high pulse energies before damaging, and a large fraction never damaged before we reached the limits of our test laser. The absence of any observable effect on internal damage thresholds is encouraging, but the current results do not rule out the possibility that some other approach to accelerated aging could reveal a growth mechanism for internal defects.

Keywords: fiber optics, fiber damage, accelerated aging, static fatigue, CO₂-laser conditioning, diffractive optics

1. INTRODUCTION

Previous studies have addressed the various damage mechanisms that can occur during the transmission of Q-switched, Nd:YAG laser pulses through step-index, multimode, fused silica fiber.¹⁻³ As indicated in Fig. 1, internal damage can occur within the initial "entry" portion of the fiber or at other sites farther along the fiber path. Entry damage results from beam focusing within the initial fiber segment, and depends upon the specific laser characteristics and the design and alignment of laser-to-fiber injection optics.² Internal damage well beyond this initial segment results from local focusing within the core due to bends in the fiber path. For example, strong focusing can occur following a transition from an initially straight path to a curved path, particularly if the local mode power distribution within the straight segment is relatively narrow.¹ Focusing within the core can also occur if the fiber path is forced to experience a small bending radius locally, as will occur when a fiber is routed around a thin post or a sharp edge with some axial tension present. Fiber buffer layers and jacketing can minimize this effect. Regardless of the specific focusing mechanism, internal damage occurs at a site where the combination of local laser intensity and local defect absorption is sufficiently severe.

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Fig. 1 Breakdown and damage processes in a high-intensity fiber transmission system

In some applications involving the transmission of high-intensity laser pulses, the lifetime of a fiber system is defined in terms of how many laser pulses can be transmitted successfully before breakdown or damage occurs. End-face contamination during use is the most obvious factor that can affect this lifetime, but a more subtle possibility is laser-induced growth of internal defects. Studies of cumulative effects on bulk damage thresholds in fused silica found that significant reductions from single-pulse thresholds could occur after a sufficient number of pulses (>1000 for a $1.06\text{-}\mu\text{m}$ wavelength laser).⁴ In other applications of interest, the required lifetime is defined in terms of the number of years the system can stay in storage and still function successfully. An important assumption is that the fiber end faces can be hermetically sealed so that end-face contamination is not a factor in determining this lifetime. This leaves the issue of whether or not internal defects can grow in time in some fashion analogous to laser-induced growth.

For fibers transmitting relatively low intensities, the expanding needs of the telecommunications industry have resulted in extensive studies of aging processes that can limit fiber lifetime. The principal aging process is typically referred to as static fatigue (or stress corrosion).^{1,5} This process involves the slow growth of flaws that inevitably result from fiber fabrication processes and from handling. Most of these flaws are surface microcracks, although bulk defects are also present. When subjected to tensile stress, surface microcracks will grow into the fiber interior due to stress-enhanced chemical reactions occurring at crack tips. Atmospheric water is the principal corrosion agent, and crack growth rates are sensitive to relative humidity and temperature. Sudden fiber failure occurs when a critical crack size is reached, as described by basic fracture mechanics in brittle materials. Standardized testing methods have been developed to establish fatigue parameters used by common models for crack growth. The current understanding of the static fatigue process is sufficiently mature that engineering predictions are routinely made for minimum fiber lifetimes in a variety of applications.

In our studies we have always used step-index fiber having a fluorine-doped, fused silica cladding surrounding a pure fused silica core. Cladding thicknesses have varied from 17.5 to $20.0\text{ }\mu\text{m}$. Surface flaws that will grow in time under tensile stress are certainly present at the outer cladding surface. However, calculations of microcrack growth have shown that crack dimensions increase very slowly over most of the expected lifetime, with significant changes only occurring close to the time for catastrophic fiber failure.¹ In addition, special proof testing on our fiber has ensured that the surface flaws in fresh fiber have dimensions $\leq 0.1\text{ }\mu\text{m}$. Consequently, surface flaws will be confined to the outer region of the cladding for nearly the entire fiber lifetime relative to static fatigue failure. Because these flaws will not approach the fiber core region where high laser intensities can be present, static fatigue is not a process that can lead to lower internal damage thresholds over time.

Because of the particular manufacturing processes used for our fibers, it is quite likely that they have significant internal defects in the core at the core/cladding interface. The preforms used for drawing our fiber have the same core/cladding structure as the final fiber. However, these preforms were originally homogeneous rods of pure fused silica. The fluorine-doped silica cladding is added to these rods through a chemical plasma deposition process.⁶ Consequently, the resulting core/cladding interface in our fibers can still have "surface" flaws associated with the original silica surface in the rods, modified in some way by the both the plasma deposition and drawing processes. Strong evidence for such defects appeared

in a previous study in which internal damage consistently occurred in a bend where strong beam focusing was likely.¹ Examination of damaged cross sections showed that the damage consistently originated near the core/cladding interface in the core region that corresponded to the highest laser intensities. In order for these defects to have a time-dependent effect on local damage thresholds, however, a mechanism for their growth must be possible. In terms of a stress-enhanced chemical process similar to static fatigue, high tensile stresses can certainly be present but the availability of a corrosion agent such as atmospheric water is not obvious. Whether or not the high concentration of hydroxyl radicals in the core of "high OH" silica fiber (up to 1200 ppm in the fiber we have typically used) can be a factor in defect growth is not known. In fact, the lack of an established growth mechanism for these defects represents a serious challenge to experimental efforts aimed at identifying whether or not internal defect growth is a concern for long lifetime applications. Without knowledge of an aging process and its controlling parameters, there is no way to ensure predictable acceleration of the process within a reasonable time for observation. In the present study, high tensile stresses and large, cyclic temperature variations were chosen somewhat arbitrarily in an attempt to stimulate internal defect growth.

In an earlier study on possible aging effects, conclusions were frustrated by inconsistent test configurations (leading to different damage mechanisms being dominant) and by the use of fibers from different production lots that apparently had different initial strength and fatigue characteristics.¹ We have addressed these difficulties in the present study by using a fixed, optimized test configuration and by only using fibers from a single, well-characterized production lot. The next section describes the two approaches to stimulating internal defect growth that were used in the current study. The following section describes the experimental procedures that were used for damage testing both fresh and aged fibers. Subsequent sections present and discuss the data obtained with these fibers.

2. ACCELERATED AGING APPROACHES

The first approach to stimulating internal defect growth was to subject a collection of test fibers to high tensile stresses for a time sufficient for some fraction to catastrophically fail due to static fatigue. Figure 2a shows the standard method that we used for generating predictable stresses in these fibers. The central portion (85 cm) of each 1-meter long test fiber was carefully wrapped around a smooth mandrel having a very uniform diameter. The resulting stress distribution at any fiber cross section varies linearly from a compressive stress at the inner radius to an equal tensile stress at the outer radius. The peak stresses are a simple function of the mandrel radius, the radius of fused silica in the fiber, Young's modulus for fused silica, and the thicknesses of the buffer layers present.⁷ In order to keep track of the fiber region experiencing tensile stresses, each fiber was marked after wrapping as shown in Fig. 2b. The static fatigue behavior of our test fibers had been

Fig. 2a Wrapped mandrel technique

Fig. 2b Stress indexing of wrapped fibers

characterized previously over a range of stresses and relative humidity conditions. Figure 3 shows typical measurements of failure time as a function of peak tensile stress. The stresses are quite high in order to produce failure times within reasonable observation periods. Stresses in our applications of interest due to fixturing and bends in the fiber path are considerably lower (< 100 kpsi) than shown in this figure, resulting in predicted lifetimes based on static fatigue of

hundreds of years. The straight-line fits correspond to a simple power-law model for crack growth. Included in this figure are failure time measurements obtained initially for the accelerated aging condition. For these measurements, aluminum mandrels having a diameter of 7.94 mm were used to generate peak stresses of 545 kpsi in the fibers. Relative humidity was held at 23% by enclosing the wrapped mandrels in a sealed chamber that contained a saturated solution of potassium acetate.⁸ Fibers were preconditioned at this relative humidity and at zero stress for several days prior to wrapping. Failure times were obtained for ten unpolished test fibers in order to obtain a fit for the cumulative probability of failure as a function of time assuming Weibull statistics. The resulting fit showed a 10% probability of failure at these conditions after 7.0 days, and a 50% probability after 10.0 days. For the accelerated aging of fibers to be damage tested, polished test fibers were preconditioned and then wrapped on the same mandrels and held at the same relative humidity. Fatigue aging was stopped after 5.8 days when 27% (9 out of 33) had surprisingly failed. The more rapid fatigue behavior in these fibers probably resulted from the presence of larger surface flaws following the handling and fixturing required for polishing the end faces. During the accelerated aging the fiber end faces were enclosed in soft tubing for mechanical protection, but were not hermetically sealed.

The second approach to stimulating internal defect growth was to subject a collection of fibers to a combination of high tensile stresses and large, cyclic temperature variations. A 430 kpsi tensile stress was achieved by wrapping polished test fibers on mandrels having a diameter of 10.03 mm, with fused silica used for the mandrels to minimize thermal expansion effects. Temperature cycling was achieved by placing the wrapped mandrels in a programmable, vented oven. Figure 4 shows the temperature variation used during this accelerated aging process. The test fibers were subjected to thirty cycles

Fig. 3 Static fatigue behavior of test fibers

Fig. 4 Temperature cycles used for accelerated aging

from -54°C to $+74^{\circ}\text{C}$ over a total period of 45 hours. Relative humidity was not controlled, and varied from approximately 2% to 100% during each temperature cycle. None of the test fibers failed during this aging condition. The end faces were again enclosed in soft tubing for mechanical protection but were not hermetically sealed.

In preparation for the testing required in the current study, we had obtained a large quantity of polished test fibers from an external source. All of the fibers were from a single production lot which had been specially characterized and proof tested by the manufacturer. The fiber end faces had been mechanically polished using a sequence that ended with colloidal silica. Unfortunately, we found that on some fibers the source had let the final polishing slurry dry and crystallize on the polishing pad, resulting in scratches on the end faces. On other fibers the source had let a layer of the final polishing slurry dry on the fiber end faces, resulting in irregular deposits strongly adhered to the surface. In either case the fibers could not be tested as received, and time constraints prevented re-polishing all of the fibers. Consequently, we chose to add a surface conditioning step to every fiber end face using a CO_2 laser. The conditioning schedule was essentially the same as we had developed previously for mechanically polished fibers.² Because of concern over possible adverse affects on the end faces by the accelerated aging environments, the CO_2 -laser conditioning was performed after the aging processes were completed. The end faces of baseline fibers to be used for comparison were also conditioned in the same manner.

3. PROCEDURES FOR FIBER DAMAGE TESTING

The experimental configuration used for testing baseline and aged fibers is shown schematically in Fig. 5. The test laser is a compact, oscillator-only, multimode Nd:YAG (Laser Photonics Model YQL-102) operated at the fundamental wave-

Fig. 5 Experimental configuration for fiber damage testing

length ($1.064\ \mu\text{m}$) in a Q-switched, single-shot mode. A larger Nd:YAG rod and flashlamp had been installed in this particular laser by the manufacturer to provide higher output energy (200 mJ instead of 150 mJ). As with previous test lasers we have used, the cavity alignment was adjusted to produce nearly Gaussian near-field and far-field profiles while preserving the laser's full multimode output energy. The average pulsewidth obtained in a 100-shot measurement was $10.2 \pm 0.3\ \text{ns}$ (FWHM), which is slightly shorter than the pulsewidths of previous test lasers. All the fibers used in the present study were from the same production lot as had been tested previously.^{2,3} This lot is type FG-365-UER fiber from 3M, having a $365\text{-}\mu\text{m}$ diameter core of high- OH^- fused silica, a $17.5\text{-}\mu\text{m}$ thick cladding of F-doped fused silica (resulting in a numerical aperture of 0.22), a $15\text{-}\mu\text{m}$ thick TECS™ coating, and a $150\text{-}\mu\text{m}$ thick Tefzel® buffer.

The laser was injected into test fibers using a combination of a custom diffractive optic and a lens having a very short focal length (15 mm). The diffractive optical element has been described in detail previously.^{3,9} This combination results in a number of attributes, such as a low ratio of peak fluence to incident energy at the fiber entrance face due to the mitigation of hot spots in the laser beam. Light is injected at angles approaching the maximum acceptance angle of the fiber, resulting in the population of higher-order fiber modes. A skew angle is added to the incident light, preventing focusing along the fiber axis within the "entry" segment of the fiber. These last two attributes also minimize downstream focusing due to bends in the fiber path. Previous use of this injection system resulted in significantly higher thresholds for internal damage.³ Because the diffractive element successfully avoids putting light into the zero (undiffracted) order, one drawback is that the lowest-order fiber modes are relatively unpopulated. The fiber path used in our previous testing (straight except for one 15-cm diameter, 360° loop) did not mix modes very effectively, and this injection system produced near-field laser profiles at the fiber exit face having reduced fluences near the fiber centerline. A uniform fluence at the fiber exit face gives higher thresholds for exit-face damage and is desirable in most applications, so some means of additional mode mixing was necessary. To achieve this in our current testing configuration, an additional 360° loop having

a diameter of 6.0 cm was added to the test fiber path. The effects of this additional loop on the exit face profile are shown in Fig. 6.

Fig. 6a Laser profiles at the exit face of a test fiber without the additional 6-cm diameter loop

Fig. 6b Laser profiles at the exit face of a test fiber with the additional 6-cm diameter loop

Approximately twenty fibers were tested for the baseline case and for each approach to accelerated aging. A machined plastic fixture was used to define the entire fiber path. When one of the aged fibers was placed in this fixture, the indexed side of the fiber that had experienced peak tensile stresses (Fig. 2a) was again placed so that it experienced the peak tensile stresses generated by the two 360° loops. Although these stresses were comparatively small, this placement exposed this fiber region to the highest possible fluences due to the internal focusing occurring within these loops.¹ The test procedure used for each fiber was similar to that used in previous studies. After alignment using a very low pulse energy, a fiber was subjected to a series of single laser pulses in which the energy was increased with each successive pulse. A total of 24 pulses were used to reach the maximum output energy from the laser (corresponding to a peak transmitted energy of at least 100 mJ), assuming that no breakdown or damage occurred. The testing was halted when a breakdown or damage event did occur on a given shot. The maximum energy transmitted before this shot, together with the attempted and actual transmitted energies during this shot, were then recorded. The measured values for highest transmitted energy before breakdown or damage were then used to generate cumulative damage probability distributions. To represent the results in this fashion, the individual fiber data were first ordered from lowest to highest transmitted energy E_i before damage, then assigned a corresponding rank R_i , where $R_i = 1, 2, 3, \dots, N$ (N is the total number of fibers in the particular case being tested). The cumulative probability for damage F_i at energy E_i was then assigned according to:¹⁰

$$F_i = (R_i - 0.5)/N$$

A Weibull distribution in terms of the variable E_i was obtained from a least-squares fit to the function:

$$F(E) = 1 - \exp(-E/E_0)^m$$

where m is the Weibull slope and E_0 is a scale parameter. If a fiber did not damage by the end of the test sequence, no value of E_i was used in the distribution fitting but the fiber was included in the total count N .

4. RESULTS

The baseline results obtained by testing fibers that had not experienced any aging environment are shown in Fig. 7. Of the twenty fibers tested, eleven had not damaged when the maximum output of the test laser was reached. Of the fibers that damaged, one fiber still transmitted over 80 mJ and the rest transmitted over 90 mJ. Three of these fibers damaged at the exit face, three fibers experienced front-face breakdown, and three fibers damaged at the front face. The Weibull probability distribution found from fitting this data is also shown in Fig. 7. This testing represented the first time that the current injection optics had been used on mechanically polished fibers that had subsequently been conditioned with a CO₂ laser,

thereby producing end faces that have demonstrated the highest thresholds for breakdown or damage.² For comparison, Fig. 8 shows the best results obtained previously using fibers with this optimum end-face processing. These results were

Fig. 7 Test results from baseline fibers

Fig. 8 Best previous test results (Ref. 2)

obtained using a simple lens injection system (50 mm focal length) followed by a mechanical mode scrambler early in the fiber path to generate a broader mode power distribution.² Laser characteristics were very similar to those of the current test laser. The earlier results show a broader range of maximum transmitted energies before damage, as reflected in the smaller scale parameter and the much smaller slope for the Weibull probability distribution. Of the 22 fibers tested in the previous study, only two did not damage. Fourteen of these test fibers damaged within the 15-cm diameter 360° loop or within the “entry” segment. This comparison emphasizes the fact that the current injection optics significantly raises thresholds for internal damage.

The results obtained with fibers that had experienced severe static fatigue are shown in Fig. 9. Although six of the fibers did not damage, the remaining fibers showed a much broader range of maximum transmitted energies. However, no internal damage was observed. Six of these fibers experienced front-face breakdown, six fibers damaged at the front face, and one fiber damaged at the exit face. The results obtained with fibers that experienced large temperature cycles while at high stress are shown in Fig. 10. These results are similar, with six fibers that did not damage but a broad range of maximum transmitted energies for the fibers that did damage. Again, no internal damage was observed. Five of these fibers damaged at the exit face, three fibers experienced front-surface breakdown, and three damaged at the front face.

Fig. 9 Test results using fibers subjected to severe static fatigue

Fig. 10 Test results using fibers subjected to temperature cycling

5. DISCUSSION

The present study considered the possibility of fiber aging mechanisms that could lead to lower thresholds for internal damage as a result of internal defect growth. Although the manufacturing processes used for our test fibers strongly suggest that core defects (particularly near the cladding interface) are present, an obvious challenge to this study is the lack of an established growth mechanism for such defects. Two aging environments were used in the present study in an effort to obtain experimental evidence that would indicate the existence of such a growth mechanism. Without prior knowledge of what controlling parameters may be important, however, our choices of high stresses and large, cyclic temperature variations were somewhat arbitrary.

The experimental configuration used for fiber testing included injection optics that had been designed to minimize most laser-induced damage mechanisms. In particular, previous use of this injection system had shown that it was very effective in minimizing internal damage. The results from testing baseline fibers (Fig. 7) showed the highest damage levels that we have obtained with practical injection optics in our various studies, with all damage in these fibers occurring at the fiber end faces. In hindsight, a test configuration so insensitive to internal damage mechanisms could have hindered observation of internal damage effects introduced by the aging environments. As stated earlier, damage occurs at a site where the combination of local laser intensity and local defect absorption is sufficiently severe. Lower local intensities within bends where focusing occurs means that larger defect changes are necessary for damage to occur at a given pulse energy. A test configuration that enhances intensities near the outer core/cladding interface within a bend¹ might have been a better choice. However, such a configuration also results in lower thresholds for "entry" damage.

Changes in internal damage thresholds were not apparent in the aged fibers, as none of these fibers experienced internal damage. Unfortunately, damage thresholds for fiber end faces were lower in the aged fibers, thereby limiting the peak intensities experienced internally within some of these fibers (Figs. 9,10). We had chosen to condition all end faces with a CO₂ laser after realizing that our fiber source had experienced problems with colloidal silica polishing. The excellent results obtained with the CO₂-laser conditioned baseline fibers indicates that this conditioning process was successful in eliminating the effects of these polishing problems. For the end faces of fibers to be aged, we were concerned over possible adverse surface effects occurring during the aging environments. Some atmospheres in chambers that use saturated salt solutions to maintain constant relative humidity have been found to be reactive,¹¹ and outgassing from organic materials present during the temperature cycles could be a contamination source. Rather than address the complexity of hermetically sealing all the end faces, we chose to perform the CO₂-laser conditioning after the aging processes were completed. The reduced damage thresholds observed for the end faces indicate that adverse surface effects did occur, and that the CO₂-laser conditioning was not successful in eliminating these effects. In an earlier study using cleaved end faces, we found that CO₂-laser conditioning would not produce the desired resistance to entrance-face breakdown and damage unless exceptional means were taken to eliminate contaminants from the end faces prior to conditioning.³

Twelve of the fibers tested in the current study experienced catastrophic front face damage. Most of these fibers showed a damage mechanism that we had not observed previously. In past studies, we distinguished between front face breakdown and front face damage on the basis of whether or not small pits in the surface were formed. No pits are observed after a breakdown event, and the subtle modifications to the surface may leave the surface more resistant to further breakdown events.¹² If small pits are formed, however, subsequent laser pulses always enlarge these pits and create additional damage sites. The new mechanism observed in the current study was associated with a subsurface damage site near the core/cladding interface. Figure 11a shows the front face of a fiber that experienced this damage process. Bright field illumination was used on the microscope to obtain this image of the 365- μ m diameter core. On this fiber a small crack can be seen in the cladding, although no surface features were apparent in most of these damaged fibers. Figure 11b shows the same fiber face under backlighting with the microscope focused down into the core. A large fractured area appears to have originated at a site near the cladding interface. This new damage process could have resulted from a combination of two features of the current testing. The diffractive optical element used in the injection optics results in a distribution of relatively high fluence spots over much of the fiber entrance face, including fairly near the cladding interface.³ In addition, the entrance faces had been conditioned with a CO₂ laser following a schedule that results in significant rounding of the surface over a region that includes the cladding and extends into the core.² Previous use of this injection system had been with cleaved fiber faces that had much less rounding following either a lighter CO₂-laser conditioning schedule or flame

polishing.³ Therefore, in the fibers that experienced this subsurface damage, the local contouring of the fiber face could have resulted in internal focusing of high-fluence spots near the core/cladding interface.

Fig. 11a Front surface of damaged fiber viewed under bright field illumination

Fig. 11b Subsurface fracture in same fiber viewed by focusing down into the fiber and backlighting

The fact that no internal damage was observed in fibers subjected to the two accelerated aging environments means that the current study produced no evidence of a mechanism for the growth of internal defects. While this result is encouraging for high-intensity, long-lifetime applications, it is certainly not conclusive. Should such a mechanism exist, its controlling parameters may not be sensitive to the accelerated aging environments used in this study. In terms of high static stresses, the current results suggest that such stresses could lead to catastrophic failure due to static fatigue before any degradation occurs in internal damage thresholds. In terms of cyclic temperature variations, the conditions used in the current study may not have been sufficiently aggressive. More extreme temperature limits, many more temperature cycles, and more time spent at the highest temperature may be worth pursuing in a future attempt to stimulate the growth of internal defects. By wrapping fibers on mandrels made from a material with a larger coefficient of thermal expansion than fused silica, some stress cycling could be introduced during temperature cycling. The current study showed the need to protect polished end faces during an aging environment, or to age unpolished fibers and then polish the end faces after the aging environment is concluded. If an aging mechanism for internal defect growth actually does not exist, however, additional attempts at accelerated aging will simply provide the same null result as the present study.

6. ACKNOWLEDGMENTS

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