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AUTHOR(S): Peter B. Lyons and Larry D. Looney

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Radiation-Induced Attenuation of High-OH Optical Fibers After Hydrogen Treatment in the Presence of Ionizing Radiation

P. B. Lyons and L. D. Looney[1]
Los Alamos National Laboratory
Los Alamos, New Mexico, 87545
Telephone 505-667-3899
FAX 505-667-0407

***Abstract*—High purity, high-OH, optical fibers were irradiated in a hydrogen atmosphere to explore hydrogen binding into defects created by the ionizing radiation. Significant improvements in subsequent measurements of radiation-induced attenuation were observed.**

I. INTRODUCTION

For more than two decades, hydrogen has been identified as a significant factor in controlling the performance of silica materials. Early experiments [1,2] demonstrated that radiation-induced attenuation in bulk silica samples was reduced if hydrogen was impregnated into the materials. Some of the earliest observations of hydrogen effects in fibers involved the ease by which molecular hydrogen could diffuse through the small dimensions of optical fibers, coupled with concerns and measurements demonstrating that increases in hydrogen concentration within the fiber core could lead to increased attenuation at certain wavelengths [3]. A number of papers considered the importance of fiber cabling designed to minimize the generation of hydrogen in proximity to the fibers [4,5], especially in very long distance undersea applications of optical fibers. These concerns also encouraged development of a range of technologies to hermetically seal the surface of optical fibers to significantly reduce diffusion of hydrogen into the silica material [6].

In optical fibers, improvement in performance in environments with ionizing radiation due to hydrogen treatment was first noted by [7], where it was observed that hydrogen permeation into the fiber either prior to or following irradiation suppressed radiation-induced absorption. Dramatic improvements were seen in the visible region near the 2 eV draw-induced defect in these demonstrations [8]. These and other papers have thoroughly shown that hydrogen can bind into various defects. Special attention has been given to fibers with extremely low-OH concentrations, since these fibers are frequently more sensitive to radiation than high-OH materials, and low-OH fibers are required for long wavelength communication applications [9,10]. Beneficial effects of hydrogen have also been recently observed in measurements of UV transmission in optical fibers [11]. A complete review paper has been published [12].

The present work employed a variation of most previous hydrogen treatments, in that hydrogen was maintained within a high-OH fiber <u>during</u> Co^{60} pre-irradiation. Absorption centers created during this first irradiation were then available for reaction with the permeated hydrogen. The goal was to use hydrogen reactions to "heal" weaker bonds that were broken during the pre-irradiation, such that subsequent irradiations would occur in a fiber with fewer major defects and, ideally, demonstrate improved radiation resistance. (This treatment process is most similar to that used in [8] where the reported data focused on the 2 eV visible band.) High-OH core material was chosen for these tests since this material has usually, although not exclusively, demonstrated superior resistance to increases in attenuation caused by exposure to ionizing radiation, and further improved resistance remains essential for some applications.

Motivation for the present approach stems from two observations. Measurements of Co^{60}-induced attenuation in high-OH fibers have usually produced strongly nonlinear dependence between increased attenuation and radiation dose. For example, in Fig. 3 of [13], wherein attenuation increases

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[1] Work performed under the auspices of the U.S. Department of Energy.

were studied during 10 krad exposures, more than 90% of the radiation-induced attenuation was created with less than the first 10% of the total dose. In transient radiation-induced measurements, observations of the increased attenuation at early times, for example, in Fig. 4 of [14], measured on the basis of unit absorbed dose, demonstrate strong nonlinearities. In both these cases, it can be speculated that the behavior could be explained by some class(es) of pre-existing defect(s) in the drawn fiber that is easily damaged at low doses, but whose effects become much less significant at higher doses (perhaps as the number of pre-existing defect sites become saturated). If the number of pre-existing defects can be reduced through a combined radiation exposure (to create a site), and hydrogen exposure (to bind into the site), significant improvement in radiation resistance should result.

2. EXPERIMENTAL PROCEDURE

Two different batches of test fiber were specially prepared for these measurements. For each batch, a Fluosil [15] fluorosilicate clad preform with a high-OH Suprasil core was drawn at Polymicro Technologies, Inc. into a 100 µm core diameter fiber. Clad diameter was 110 µm and a thin polyimide buffer layer was applied, resulting in a final diameter of 125 µm. [This buffer layer has been shown in previous efforts [14,16] to result in improved radiation resistance.] Each batch of test samples was cut from a single length of optical fiber drawn from one preform.

In the first series of measurements, the fiber was prepared as described above. However, severe instabilities were noted in the measurements with that fiber, which were hypothesized to be traceable to large and highly variable microbending losses (variations in attenuation) in the multilayer test coils of fiber utilized in the radiation tests. The second batch of fiber was prepared as described above, but an additional acrylate buffer layer was added to increase the fiber diameter from 125 µm to 250 µm, and reduce microbend sensitivity. This second batch demonstrated stable attenuation in subsequent tests.

Measurements with the first series of fibers demonstrated significant benefits to be realized from the simultaneous hydrogen concentration in the fiber and exposure to ionizing radiation. However, only data from the second batch of fiber will be reported in this paper.

The long single fiber was cut to lengths of 200 m. Several of these shorter fibers were exposed to a 55 psi hydrogen atmosphere at 107°C for 100 hours. This time duration should be ample to guarantee a hydrogen concentration within the fiber close to equilibrium with the external hydrogen environment. After hydrogen permeation, the fibers were packed in dry ice to retard out-diffusion of the hydrogen and transported to a Co^{60} source. After warming to room temperature, some fibers were promptly exposed to Co^{60} doses at several dose levels. Details of fiber treatment are given in Table I. All fibers were then stored at room temperature for 7 days prior to subsequent tests. Most residual interstitial hydrogen gas, but not all, would have escaped during this 7 day period. A quantitative measurement of residual hydrogen in the fibers during the final measurement was not available.

TABLE I
FIBER TREATMENT PARAMETERS

Fiber #	H_2 Exposure	Co^{60} pre-irradiation
1	no	none
1H	yes	none
2	no	1 krad
2H	yes	1 krad
3	no	10 krad
3H	yes	10 krad
4	no	50 krad
4H	yes	50 krad

Radiation induced attenuation was measured with a Co^{60} source under the conditions specified in the NATO Nuclear Effects Task Group procedure [17] A Laser Precision AP-4200 Stabilized Fiber Optic Light Source with an 850 nm AP418 laser output unit provided the illumination into a multimode pigtail. That pigtail was connected to the test fiber. The output of the test fiber was connected to a Hewlett Packard 81000JA Detector Head. The detector was coupled to a Hewlett Packard 8152A Optical Average Power Meter whose output was measured with a Hewlett Packard 3457A Multimeter. The multimeter output were sampled on one second intervals under computer control and recorded. Per the specification in the test procedure [17], input power to the test fiber was adjusted to be 1 μW. Output powers from the test samples were about 0.5 μW.

Test fibers were wound on a 10 cm diameter test spool and placed 38 cm from a 13 kCi Co^{60} source. Source output was measured with an EG&G, Inc. NIST-traceable 1 cc air ionization chamber whose output was corrected for pressure and temperature. Dose in rads was obtained using 33.7 eV/ion pair in air and gamma ray attenuation coefficients for both SiO_2 and air [18]. The resulting correction factor from Roentgen to rad(SiO_2) was 0.869. [All doses in this paper are in units of rad(SiO_2).] Dose at the center of the fiber bundle was 21.66 rad/s (1300 rad/min) after correction for spool and fiber attenuation. Fibers were irradiated to a 10 krad total dose.

3. DATA

Attenuations are listed in Table II for several dose levels, including the final 10 krad level, and actual data follow in Fig. 1-4. Previous measurements [14] of similar fibers have frequently shown a peak in the radiation-induced attenuation at low dose (a few hundred rads) with an improvement (i.e., decrease) in attenuation at higher doses. That phenomenon was observed in some of the present measurements and this peak attenuation is denoted by a_{pk} in Table II.

4. DISCUSSION AND CONCLUSIONS

In Fig. 1, data from fibers 1 and 1H are intercompared. If all the hydrogen diffused into sample 1H had diffused back out, these two fibers should have yielded equivalent performance. The significant difference demonstrates that out-diffusion has not been completed and that residual hydrogen is still present. It may be speculated that the improvement in performance of fiber 1H at late time is due to that residual hydrogen healing defects that were created during the irradiation. At early times, where fibers 1 and 1H are comparable, it is probable that the residual hydrogen has not yet had time to diffuse to the defect centers being created.

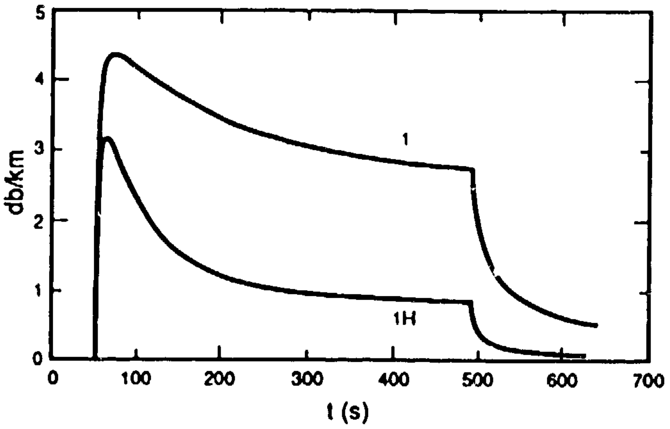

Fig. 1. Radiation-induced absorption for fibers 1 and 1H. Performance of the control fiber (1) is similar to that observed for comparable fibers (PM2 and PM3) in [14] where a_{pk} of 4.23 and 2.91 db/km and attenuation at 10^4 rad(S_iO_2) of 2.26 and 2.25 db/km were noted, respectively.

Table II
RADIATION-INDUCED ATTENUATION DATA
(db/km)

	Dose [rad(SiO_2)]				
Fiber #	100	200	500	10^4	a_{pk}
1	3.25	4.06	4.35	2.73	4.36
1H	2.51	3.09	2.99	0.83	3.17
2	2.32	3.04	3.34	2.39	3.35
2H	0.73	0.85	0.88	0.74	0.87
3	1.27	1.68	1.98	2.35	-
3H	0.57	0.67	0.70	0.64	0.70
4	1.29	1.87	2.58	3.25	-
4H	0.47	0.55	0.63	0.61	0.63

Pre-irradiation of optical fibers frequently leads to a somewhat improved performance in a subsequent radiation exposure, as noted in [14]. This could occur for several reasons. For example, radiation-induced annealing may be occurring as defects are effectively healed through relaxation of the neighboring lattice atoms into configurations resulting in stronger bonds. In these high-OH fibers, the radiation may itself be releasing hydrogen, which can diffuse to and bond into broken bonds. Other explanations may also be appropriate. For the first two pre-irradiation levels of 1 and 10 krad, significant improvement is noted in Fig. 2 where the four fibers, which were not exposed to hydrogen, are intercompared.

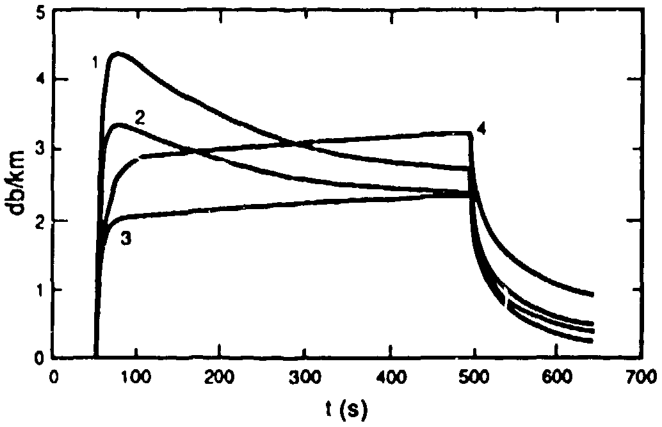

Fig. 2. Radiation-induced absorption for the control fiber (1) and the three pre-irradiated fibers (2-4).

In Fig. 3, extremely dramatic improvement in radiation-induced attenuation is noted where the control fiber is compared to the three fibers that were pre-irradiated while significant levels of molecular hydrogen were within the fibers. This improvement is further clarified in Fig. 4 where the same four fibers are intercompared at early times. Large ratios between treated and untreated samples are evident.

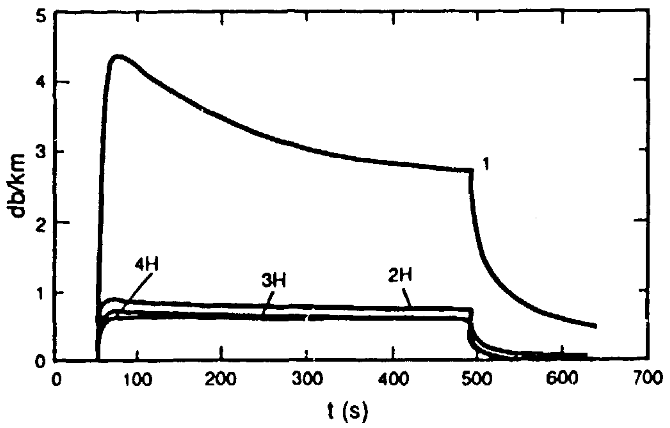

Fig. 3. Radiation-induced absorption for the control fiber (1) and the three fibers (2H, 3H, 4H), which were exposed to radiation while hydrogen was present, prior to the attenuation measurements shown here.

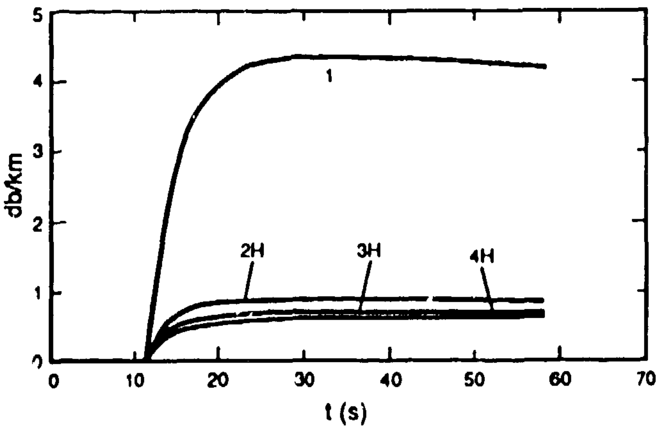

Fig. 4. Data of Fig. 3, repeated to illustrate behavior at early times (low doses).

In fibers of this type, without pretreatment, about 90% of the final attenuation is realized before more than 5-10% of the dose is delivered. The original motivation for the current study was a hypothesis that the extremely rapid rise in attenuation of untreated fibers at low doses may be due to a class of readily damaged pre-existing defects. It was hypothesized that the hydrogen gas might bind into those sites in the presence of radiation, thereby decreasing the damage probability for that site. If this had occurred, the fibers treated with hydrogen would not have displayed the rapid initial increase in attenuation, and ideally would have demonstrated a linear dependence of attenuation on dose. In the observed data, however, the attenuation observed in all the samples, whether hydrogen treated or not, was still very nonlinear.

Nevertheless, the hydrogen plus pre-irradiation treatment has demonstrated extremely beneficial results, yielding about 8x less attenuation at early times and few 100 rad doses (cf. Fig. 4). Thus, some classes of defects have been partially (or totally) healed by the hydrogen treatment, but clearly not all them. Other choices of pre-irradiation conditions (higher doses, for example) need to be further examined.

Additional work is clearly required to optimize this process. The three pre-irradiation dose levels chosen herein suggest that still higher doses may be more beneficial. The stability of the hydrogen bound into the created defects needs to be studied, although it would be anticipated that this hydrogen should be very robustly bound into those sites. Additional studies should be made when a larger fraction of the hydrogen has diffused out of the fiber. Further studies could also explore hydrogen treatment of the fiber preform, before fibers are drawn, as was demonstrated to significantly improve UV performance of similar fibers [11]. Transient radiation-induced attenuation measurements need to be completed to see if the large differences noted herein, approaching a decade improvement in radiation-induced attenuation, can be duplicated under pulsed exposure conditions.

6. ACKNOWLEDGEMENTS

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