White paper

An Investigation on Effect of Time Aging on Mechanical Behavior of Optical Fiber with Partially Cured Coating

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Abstract
The effect of time aging on mechanical behavior of optical fiber with partially cured coating is investigated experimentally. Fiber samples were drawn with different primary and secondary coating curing level. Fiber samples were exposed to fluorescent light and stored in a controlled laboratory environment. Tensile strength, two-point bend strength, primary & secondary coating diameters and curing of primary and secondary coatings were measured at various time intervals after draw upto 180 days. Dynamic fatigue of the samples was measured just after drawing and after 120 days of time aging. Increase in strength & fatigue, degree of curing of both primary & secondary coatings and decrease in coating diameters with time aging were observed. Weibull parameter (M-value) was also increased with time aging for the under-cured fiber. However, the increasing rate was different for various fiber samples and dependent on coating shrinkage and increase in curing level. It is found that mechanical properties of optical fiber have a strong relation on time aging and coating curing.

Key Words
Optical Fiber; zero stress aging; Coating Curing; Strength; Fatigue

Introduction
Zero stress aging has been a topic of research and interest in understanding the mechanical behavior of optical fiber during the expected lifetime. Several studies have been carried out to understand the effect of aging on fiber strength properties. The aging behavior of fiber in terms of strength degradation, coating de-lamination and surface roughness in harsh environment have been correlated with various properties of fiber like coating chemistry, coating curing, dissolution of silica etc[1,2,3] . Reduction in strength value was reported for partially cured coating, coating with high moisture diffusion and less adhesion to glass. However, it was also reported that the fiber strength increases with aging time for bulk silica glass. The increase in strength for abraded silica glass was attributed to conversion of sharp flaw to blunt flaw on aging[4]. Dynamic fatigue, which is

one of the predictors of lifetime of optical fiber, were found to increase on aging due to dispersion of strength values.[5, 6] Relationship between mechanical strength and coating shrinkage with time aging has also been investigated[7]. It was found that the coating shrinkage is an important factor to increase the mechanical strength of the optical fiber. This paper is intended to investigate the effect of time aging in normal laboratory environment on mechanical behavior of optical fiber with different levels of coating curing.

2. Experimental Process

2.1 Samples and Aging

Single mode silica optical fibers (125µm) were drawn with dual coating application (wet on wet) system. Commercially available coating materials were used. Three fiber samples were drawn with different curing level of primary and secondary coating. The proof-tested fibers were collected and tested for coat geometry, tensile strength, two-point bend strength, and degree of curing of primary & secondary coating. The tested values of the fiber samples just after drawing are presented in Table 1. The curing levels of the samples were kept below than the ideal range of 92-96% other than secondary coating of sample 3. All samples were exposed to fluorescent light and stored in a constant 25±2°C and 50±5% relative humidity environment. Tensile strength, two-point bend strength, primary & secondary coating diameters and curing of primary and secondary coatings were measured at various time intervals after draw upto 180 days. Dynamic fatigue of the samples was measured just after drawing and after 120 days of time aging.

Table 1 Test values of fiber samples just after drawing

Parameters	Sample No. 1	Sample No. 2	Sample No. 3
%DOC Secondary Coating	90	87	96
%DOC Primary Coating	61	78	88
Secondary Coat Diameter (µm)	245.1	242.8	244.8
Primary Coat Diameter (µm)	189.9	85.4	181.7

2.2 Dynamic Tensile Strength

This test was conducted as per FOTP- 28C (EIA/TIA) with a 0.5m sample length and 25 mm/min extension rate at 25(±2)°C temperature and 50 (±5)% relative humidity. Mean of five readings were recorded as tensile strength. However, 30 readings of each sample were taken just after drawing and after 180 days of time aging to plot Weibull probability distributions.

2.3 Two-Point Bend Strength

The two-point bend strength in dynamic two-point bending was measured according to Telecommunication Industry Association standards (ITM-13/ TSB62-13). In the two-point bend apparatus, the fibers are bent between two faceplates that are brought together until a fiber breaks. An acoustic transducer detects the break. Grooved faceplates are used to locate the fiber accurately. Bending stress develops at the tip of the fiber sample placed in between two faceplates. The length of the region under bending stress is approximately 2 mm. A minimum of

15 specimens for each sample was measured in a commercial twopoint bend test apparatus in a laboratory environment. All calculations were based on the nominal glass diameter of 125µm and actual coating diameter. The initial faceplate spacing was 10 mm and the faceplate velocity was 100µm/sec. Mean value of 15 readings was recorded as two-point bend strength.

2.4 Dynamic Tensile Fatigue

Dynamic fatigue by tensile loading was measured as per IEC 60793-1-31. Four different extension rates were chosen between 0.1, 1.0, 10 & 100 percent/min. The gauge length was 0.5 meter. A sample size of 15 per extension rate for each fiber sample was used. Dynamic fatigue or stress corrosion parameter (N_d) had been calculated from the slope of linear plot of log of failure stress vs. log of the stress rate.

2.5 Coating Geometry

The diameter of the primary and the secondary coatings were measured by a geometry Analyzer by ray traced side view method as per International Telecommunication Union standards (ITU G.650).

2.6 Degree of curing (%DOC)

A Fourier Transform Infra Red (FTIR) Spectrometer was used to determine percentage degree of curing or percentage reacted acrylate unsaturation (%RAU) of primary and secondary coatings.

The instrument set-up and sample preparation techniques are different for secondary and primary coatings. *Secondary (Outer) Coating* - A Horizontal Attenuated Total Reflection (HATR) accessory was used to measure the %DOC at the surface of the secondary coating to a depth of 1-3µm. The IR spectrum was taken after placing ~10cm pieces of fibers side by side on the ATR (Zinc Selenide) crystal.

Primary (Inner) Coating -Universal Attenuated Total Reflection (UATR) Diamond (3 reflections) accessory was used to measure the %DOC of primary coating. Sample preparation and calculation of degree of curing from FTIR spectrum were presented in previous publication[3].

3. Results and Analysis

Changes in primary and secondary coating diameters with time aging after draw are shown in Fig. 1 & 2 respectively. Coating diameters of both primary and secondary coatings were decreased with time aging. Changes in degree of curing of primary and secondary coatings with time aging after draw are shown in Fig. 3 & 4 respectively. Curing level of all partially cured samples was increased with time aging except secondary coating of sample 3, which was in the ideal range of curing. Post-draw curing resulted shrinkage of coating.

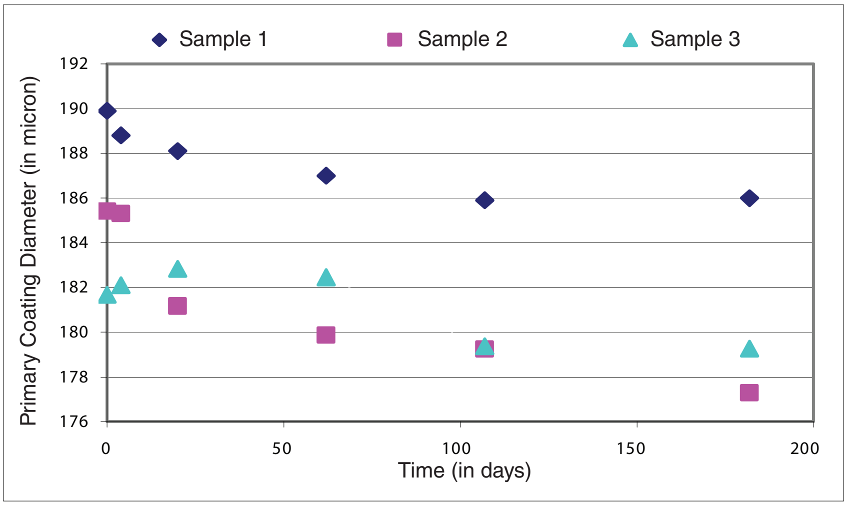

Figure 1 Change in primary coating diameter with time aging after fiber drawing

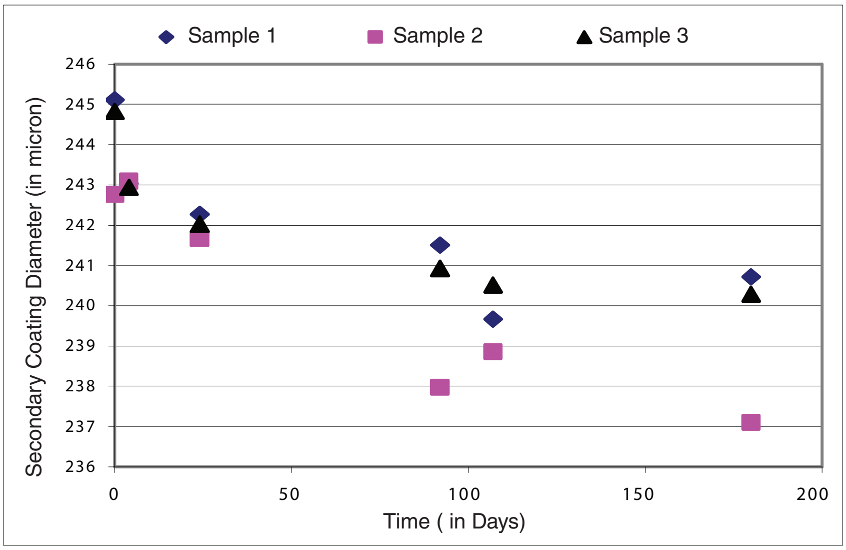

Figure 2 Change in secondary coating diameter with time aging after fiber drawing

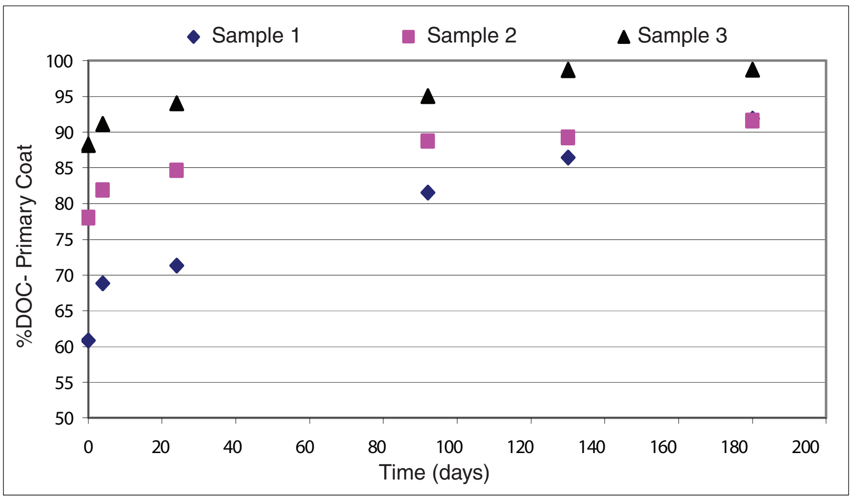

Figure 3 Change in %DOC- primary coating with time aging after fiber drawing

Fig.5 & 6 shows change in tensile and 2-point bend strength with time aging respectively. Increase in strength values were observed for both the modes of loading. However , increase in tensile strength was more compare to 2-point bend strength particularly for sample 1 & 2 , which were under-cured originally. On time aging, curing level of the samples were increased resulted shrinkage of coating diameters which built-up axial stress on the fiber and caused increase in tensile strength. Extent of increase in curing level and tensile strength were more for sample 1 & 2 compare to sample 3. Curing level and strength values were highest for sample 3 and little changes were observed for those parameters on time aging. Similar type of phenomenon was reported in previous study[7], where shrinkage in primary coating diameter was found to be a major contributor in increase in strength base on Lame formula.

Table 2 Weibull shape parameter of 2-point bend strength values

Day	Sample 1	Sample 2	Sample 3
0	12	104	146
23	61	154	125
37	68	161	155
106	71	132	203
180	133	152	230

Table 2 shows shape parameter calculated from Weibull probability distribution of 2-point bend strength values. Shape parameter is calculated from the slope of the Weibull plot. Higher shape parameter signifies less variation in the strength values. Fig 7 & 8 show Weibull distribution of tensile strength values of all the three sample just after drawing and after 180 days of time aging respectively. Shape factor of all three samples were increased on time aging. From Table 1 & 2 , we can see higher curing level resulted higher shape parameter. Same phenomenon was observed for tensile strength as well. Table 3 shows dynamic fatigue measured in tensile mode before and after 120 days of time aging. Under-cured sample 1 & 2 shows much less fatigue, which increased on time aging significantly. Under-cured coating caused non-uniform distribution of axial stress resulted wide variation of strength values and low shape parameter. Un-cured coating were postcured slowly with time aging under fluorescent light and other environmental conditions of storage. Previous studies[3,6] showed increase in dynamic fatigue values of fiber on hot water aging due to blunting of large flaws. In this study we observed increase in fatigue value due to slow post-curing of coating.

Table 3 Dynamic fatigue parameter

	Sample 1	Sample 2	Sample 3
Day 0	15	12	18
Day 120	18	17	21

4. Conclusions

The behavior of mechanical strength and fatigue of partially cured fibers was investigated. Post-draw coating curing leaded to coating shrinkage and subsequent reduction in diameters and increase in strength and fatigue values. Under-cured samples showed high variation in strength values and thus low Weibull shape parameters. Significant increase in shape parameter was observed on post-draw curing during time aging under fluorescent light.

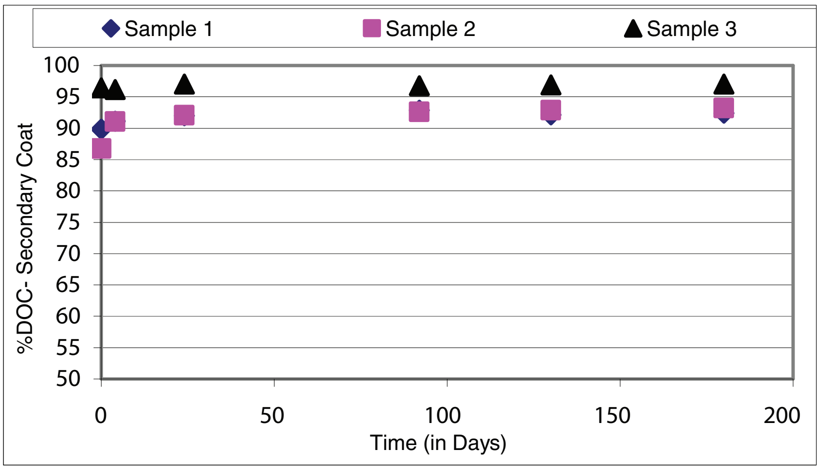

Figure 4 Change in %DOC- secondary coating with time aging after fiber drawing

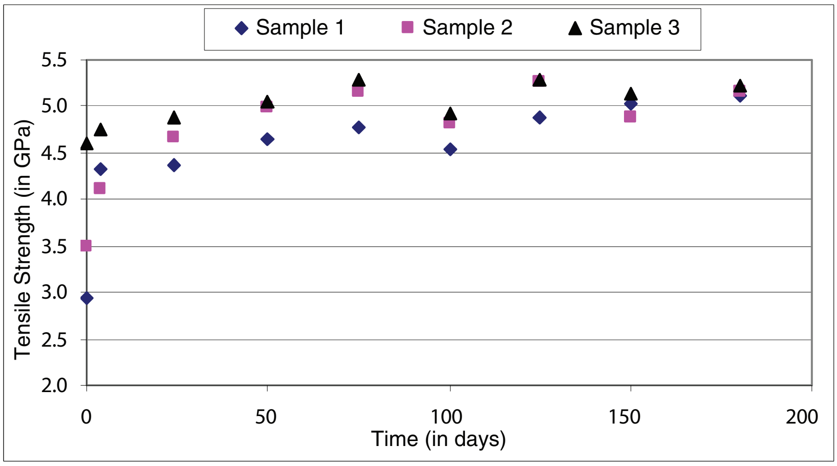

Figure 5 Change in tensile strength with time aging after fiber drawing

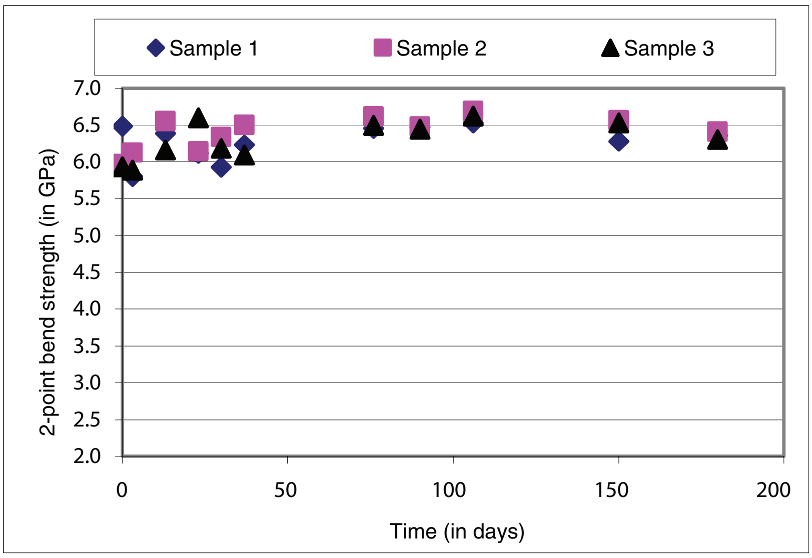

Figure 6 Change in two-point bend strength with time aging after fiber drawing

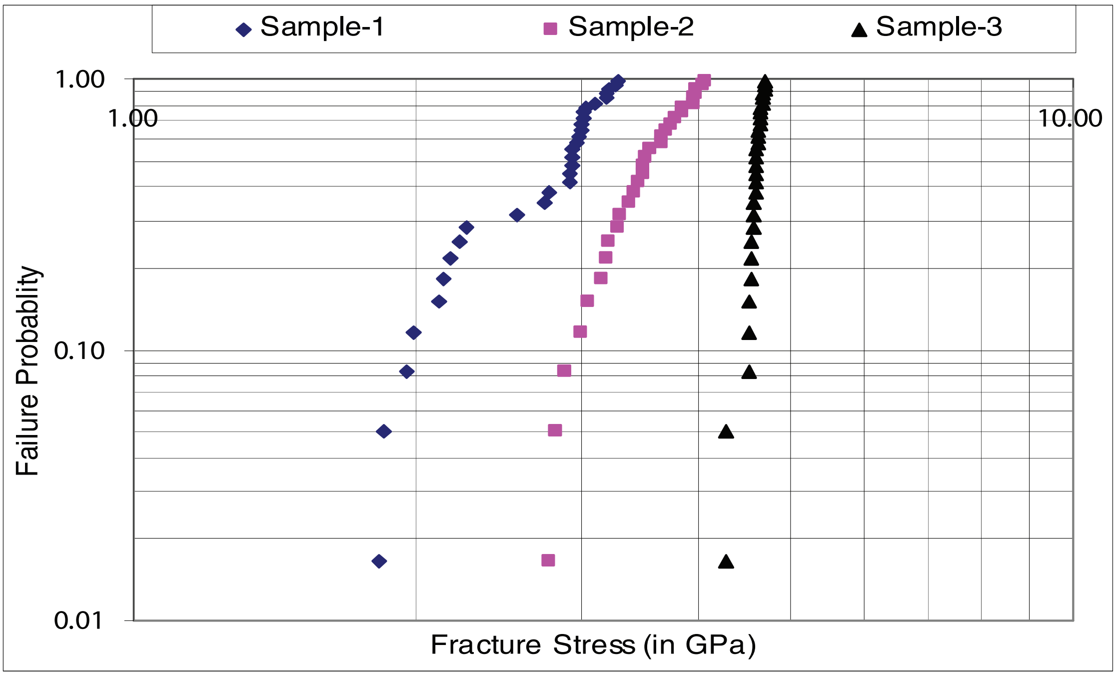

Figure 7 Weibull probability plot of tensile strength values at day 0

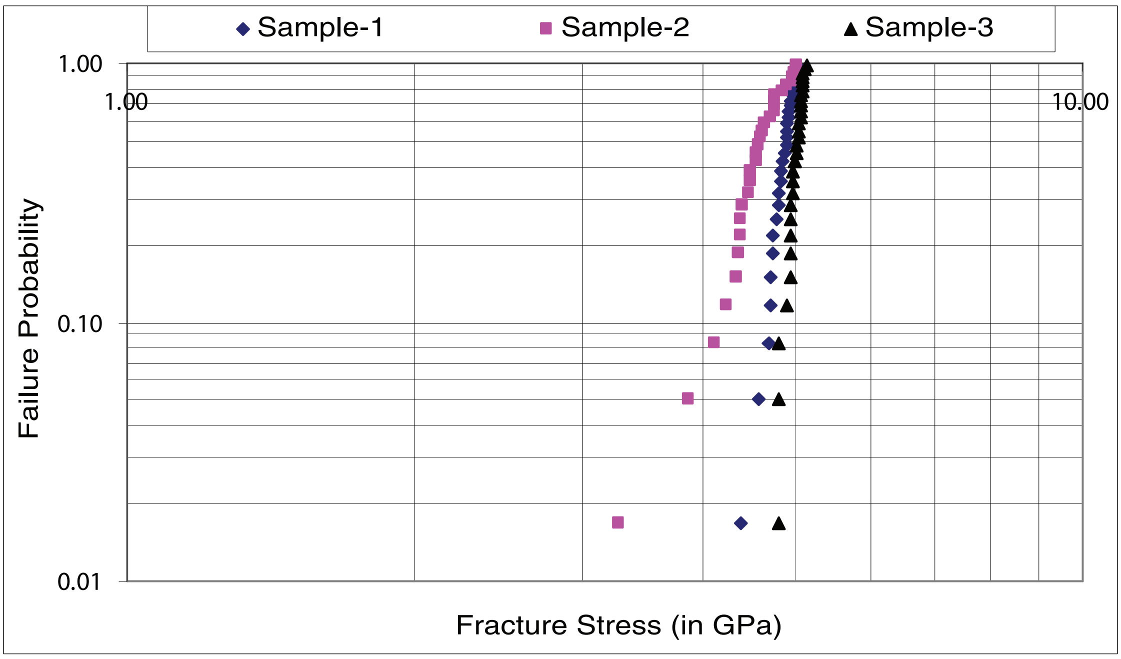

Figure 8 Weibull probability plot of tensile strength values at day 180

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