White paper

Hot Water Aging Performance of Silica Optical Fiber with Single Coating Characteristics

Author
Sudipta Bhaumik

Strip force and curing of primary coating were found to have a significant impact in terms of an optical fiber's ability to withstand corrosion and delamination problems

Introduction

Silica optical fiber is widely used in long-haul telecommunication systems. In these applications the fibers are frequently exposed to corrosive environments that can cause strength loss. However, optical fiber can be corroded chemically due to moisture attack, which is known as "zero-stress aging" and makes unreliable prediction of expected lifetime.[1] Optical fiber consists of two coating namely inner and outer coating. The inner coating, which is softer, protects the glass for improved bend sensitivity. The outer coating, which is harder, helps to reduce the handling damage on the fiber. Another function of this protective dual polymer coating applied to the fiber during the drawing process should be to protect against such corrosion.
Zero stress aging, in terms of strength degradation and appearance of delamination at glass/coating interface in dual coated fiber, is caused by surface dissolution of silica [2], low degree of curing of coating and a change in other coating performance attributes like adhesion promoters, water absorption, water extractable, gel fraction, glass peel strength, glass transition temperature (T_g) and volatile matters [3,4,5,6,7,8]. Matthewson.J et al showed addition of nanosized particles of fumed silica to coating increases the resistance to strength degradation by zero stress aging [9]. In this paper, the influence of coating related parameters like strip force, degree of curing of primary and secondary coating and the coating geometry of unaged fiber to hot (85°C) water aging is investigated without changing liquid coating characteristics/chemistry.

Experimental Process

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. Table. 1 shows the typical values of different cured characteristics that were proven to have impact on hot water aging performance of the primary and secondary coating materials.
A total of eight test fibers were drawn with different process parameters and hardware. The proof-tested fibers were collected and tested for coat geometry, two-point bend strength, and degree of curing of primary & secondary coating and strip force. Loose coils of approximately 160 mm diameter were prepared for each test fiber and aged under de-ionized water in glass bowls at

85°C temperature. Each fiber was aged for a predetermined amount of time (e.g. 1,3,7,14,21 and 30 days). Upon removal from hot water, the samples were preconditioned in laboratory environment (23±2°C and 50±5% RH) for minimum 12 hours prior to testing.

Strip Force

The strip force was measured according to the Telecommunication Industry Association Fiber Optic Test Procedure FOTP-178. A minimum of 5 specimens for each sample was measured in a customized strip force-testing machine mounted with a commercial stripping tool of diameter 180µm. The stripping length was 50 mm with a stripping rate of 500-mm/min. The strip force of both aged and unaged fiber was measured after conditioning in a laboratory environment. The median value of 5 readings was reported as strip force.

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). A minimum of 15 specimens for each sample was measured in a commercial two-point bend test apparatus. The median value for those 15 readings was reported as the two-point bend strength. All calculations were based on the nominal glass diameter of 125µm and actual coating diameter. The initial faceplate spacing was 10 mm and the plate velocity was 100µm/sec.

Optical Microscopy

An optical microscope was used to examine each unaged fiber and fibers after each aging interval. The samples were immersed in an index-matching oil to locate the interfaces between the clad (glass)/ primary (inner) coating and primary (inner) / secondary (outer) coating using transmitted light illumination. The aged fibers were examined after conditioning in a laboratory environment for minimum of 12 hours and dried of surface water.

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).

Degree of coating curing

A Fourier Transform Infra Red (FTIR) Spectrometer was used to determine degree of curing of primary and secondary coatings. IR-spectroscopy analyses the resonance vibrations of functional groups. Depending on the functional groups, typical bands (peaks) may be observed in characteristics regions of the infrared spectrum. In the course of curing of UV-curing coating the vibrational bonds at ~1406cm^{-1} diminish but the bond at ~1520cm^{-1} (or ~1510cm^{-1}) remains unchanged throughout the curing process. Therefore, the extent of reduction of peak area at ~1406 cm^{-1} with respect to peak area at ~1520cm^{-1} (or ~1510cm^{-1}) are taken as a means of judgment for the extent of curing achieved. The peak at ~1406cm^{-1} and ~1520cm^{-1} (or ~ 1510cm^{-1}) are known as absorbance or analytical peak and reference peak respectively. Fig. 1 & 2 show IR spectrum of primary and secondary coating (both liquid gel and fiber) with the indicated absorbance and reference peak. The locations of reference and absorbance peaks in the IR spectra of coating are depending on the coating formulation and chemistry. Most commonly, to

determine degree of curing (DOC), the bands to be analyzed are integrated and specific figures in “percent” are obtained upon the application of
following equation:

$$DOC\ (\%) = \left[1 - \frac{(A_{AF} / A_{RF})}{(A_{AL} / A_{RL})}\right] \times 100$$

A_{AF} = Area under the absorbance peak (at ~1406cm-^{1}) of fiber;
A_{RF} = Area under the reference peak (at ~1520 cm-^{1} and ~1510 cm-^{1} for secondary and primary coating respectively) of fiber; A_{AL} = Area under the absorbance peak (at ~1406cm-^{1}) of liquid coating; and A_{RL} = Area under the reference peak (at ~1520 cm-^{1} and ~1510 cm-^{1} for secondary and primary coating respectively) of liquid coating.
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 degree of cure 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 degree of curing of primary coating. Portions, ~2 cm, of both coatings were cut together along the length of the fiber by blade and then the glass (clad) portion was taken out to expose the primary (inner) coating inner surface. The sample was placed (one sided coating only) on the diamond crystal of UATR by keeping the primary coat portion down. It was pressed to get a good contact with crystal surface and the IR spectrum of the primary coating was taken. Care had to be taken to avoid contact between the secondary coating and the diamond crystal. However, the primary and secondary coating spectrum can be distinguished by the reference peak position as shown in Fig. 1 & 2.

Results and discussion

Coating related parameters of unaged test fibers

The unaged (initial) coating related parameters of eight test fibers are shown in Table.2. The unaged strip force of test fiber is directly proportional to %DOC of primary coating, %DOC of secondary coating and the secondary coat thickness and is inversely proportional to the primary coat diameter. The strongest corelation (R-value: 0.75) is observed between strip force and the primary coat diameter (see Fig. 3). The decrease in the lower modulus primary coating diameter resulted in an increase in the strip force. A higher degree of curing of coatings enhances adhesion between the glass & coating and subsequently increases strip force value.

Hot water aging behavior of test fibers

Table.3 shows the changes in two-point bend strength for hot water aged fibers at different day intervals. The relationship between the strength degradation and unaged strip force is presented in Fig.4. A strong relationship exists between these two parameters. A higher unaged strip force resulted in lesser strength degradation. The co-relation factor (R-value) for different day interval is also presented in Table. 3. High R-values at all day intervals signify unaged strip force plays an important role to control strength degradation for both short and long period aging processes. An increase in strength value on aging was observed for Fiber# 8, which also shows the highest strip force.

Table.4 shows the changes in strip force for hot water aged fibers at different day intervals. The strong relationship between change in strip force on aging and degree of curing of primary coating is presented in Fig.5. A higher degree of curing of the primary coating resulted in lesser degradation of the aged strip force value. The co-relation factor (R-value) for different day interval is presented in Table. 4. Higher R-values at longer aging period signifies that higher %DOC of primary coating resists strip force degradation during the long-term aging process. But there is no correlation between degradation of strength and the strip force of same fiber. Test fibers # 4,5,6 and 7 have shown an increase in strip force and decrease in strength as the aging process progresses. The opposite phenomenon is observed for fiber#8.

Table 5 shows percentage de-lamination observed between the glass and primary coating of all test fibers at different aging day interval. Fig. 6, 7 and 8 show optical micrograph of aged fiber #4 at day 7, aged fiber#5 at day 7 and aged fiber#6 at day 30. Fig . 6 & 7 show presence of de-lamination. The extent of de-lamination in fiber#5 is more compare to fiber#4. Fig. 8 shows no delamination after 30 days aging. From Tables 2 & 5, it can be concluded that a higher strip force, degree of curing of primary & secondary coatings and a lesser primary coat diameter increases de-lamination resistance. As de-lamination observed only in between the primary coating and the glass interface, the %DOC of the primary coating is the most important controlling parameter to resist delamination.
A higher degree of curing reduces amount of unsaturated acrylate, which can be extracted by aging and hence prevents de-lamination. From Table 1 it can be seen that the primary coating contains more volatiles and water extractable compared to the secondary coating. During hot water aging these volatile matter and water extractable come out from the coating causing debonding between primary coat and glass interface. Thus, a reduction in the primary coating diameter decreases the total amount of the extractable and subsequently reduces de-lamination. From Tables 2 & 5, it can be concluded that from unaged strip force, degree of curing of primary and secondary coating and primary coat diameter, an idea can be generated about the de-lamination behavior of fibers on hot water aging.

Conclusion

Unaged strip force is directly proportional to %DOC of the primary coating, %DOC of the secondary coating and secondary coat thickness and inversely proportional to primary coat diameter. The strongest co-relation is observed between the strip force and the primary coat diameter.
A higher unaged strip force resulted in lesser strength degradation on hot water aging both for short & long aging period.
A strong relationship between the change in strip force on aging and the degree of curing of primary coating is observed. A higher degree of curing of the primary coating resulted in lesser degradation of the aged strip force for longer period aging. There is no correlation between the degradation of strength and the strip force of same fiber on aging.
A higher strip force, degree of curing of primary & secondary coating and a lesser primary coat diameter increase the de-lamination (between glass and primary coating) resistance of fiber on hot water aging.
Hot water de-lamination behavior of silica glass optical fiber with single coating characteristics can be predicted from unaged strip force, degree of curing of primary & secondary coating and primary coat diameter.

Acknowledgments
The author wish to thank Pankaj Majumdar, Ravi Shankar and Pranab Kr. Dash for their help in to carrying out the necessary testing.

REFERENCES

1. J.T.Krause, " Zero stress strength reduction and transitions in static fatigue of fused silica fiber lightguides", J. Non-Cryst.Solids, 38 & 39, 497 (1980)
2. M.J. Matthewson and C.R.Kurkjian, "Environmental effects on the static fatigue of silica optical fiber", J. Am. Cram Soc., 71,17 (1988)
3. Rolf.A.Frantz, Barry J.Keon, Eva.M.Vogel, Trevor.N.Bowner and Haken.H.Yuce "The affects of optical Fiber Coating and Ink materials on the corrosion of the Glass Surface", IW&CS, P 742(1994)
4. Akira Murata, Keija Oohashi and Shinji Araki, "Reliability of the adhesion of the Glass- Primary Coating Interface on Optical Fiber", IW&CS, P: 323(1996)
5. Tomoyuki Hattori, Akira Urano, Nobuhiro Akasaka and Yasuo Matsuda "Investigation on influence of Coating Materials to zero Stress Aging of optical Fiber," IW&CS, P: 865(1995).
6. H.Hyuce, I.M.Pitz, R.A.Frantz and M.Andrejco "The affects of Coating Cure on the Mechanical Characteristics of optical Fiber", IWCS, 715 (1990).
7. N.Akasaka, T.Hattori, T.Nanoka, K.Oishi and Y.Matsuda"Design of Optical Fiber Coating", Proceeding of the !9th ACOFT, 375(1994)
8. US 6,470,128
9. M..John Matthewson, Vincenzo V.Rondinella and Charles R..Kurijan, "Coating Additives for Improved mechanical reliability of Optical Fiber", J.Am.Ceramic Soc, 77(1)73-80(1994)

Table. 1 : Characteristics of the Primary and Secondary Coating

Cured Characteristics	Primary Coating	Secondary Coating
Tensile Modulus at 25°C	0.9 MPa	30.7 MPa
Glass Transition Temp.(T_g)	- 22°C	92°C
Glass Peel Strength	13 gm -	
Water Absorption at 25°C Water Extractable at 23-25°C	0.78% 1.78 %	0.82% 0.24%
Water Extractable at 85°C	3.2%	2.8%
TGA Volatiles	5.7%	4.3%

Table 2: The unaged coating related parameters of the eight test fibers

Test Fiber No.	Unaged Strip Force (in N)	%DOC Secondary Coat	%DOC Primary Coat	Primary Coat Diameter (in	Secondary Coat µm)	Secondary coat Diameter (in µm) thickness (in µm)
Fiber#1	0.823	96.4	92	188	245	29
Fiber#2	1.136	97.8	90	195	244	24
Fiber#3	1.200	98.9	91	185	245	30
Fiber#4	1.254	94.5	92.4	184	244	30
Fiber#5	1.372	96.7	96.8	186	245	30
Fiber#6	1.500	99.7	98.5	183	242	29
Fiber#7	1.607	98.9	97	180	247	34
Fiber#8	1.746	99.7	96	178	244	33

Table. 3 Changes in two-point bend strength for hot water aged fibers at different day intervals

	Normalized Two-Point Strength value						
	Day0	Day1	Day3	Day7	Day14	Day21	Day30
Fiber#1	1	0.85	0.82	0.77	0.65	0.55	0.46
Fiber#2	1	0.83	0.79	0.73	0.77	0.76	0.78
Fiber#3	1	0.87	0.84	0.73	0.88	0.80	0.78
Fiber#4	1	0.9	0.86	0.80	0.73	0.74	0.74
Fiber#5	1	1	1.00	0.92	0.89	0.88	0.85
Fiber#6	1	1	0.95	0.93	0.89	0.94	0.88
Fiber#7	1	1	0.99	1.04	0.96	0.98	0.98
Fiber#8	1	1.03	1.01	1.02	1.00	1.02	1.07
R-value with Un-aged strip force	-	0.88	0.84	0.87	0.92	0.98	0.97

Table. 4 Changes in strip force for hot water aged fibers at different day intervals

	Normalized Strip Force value						
	Day0	Day1	Day3	Day7	Day14	Day21	Day30
Fiber#1	1.000	1.02	1.08	1.11	0.75	0.70	0.65
Fiber#2	1.000	1.00	0.98	0.95	0.98	0.96	0.82
Fiber#3	1.000	1.00	0.98	0.94	0.86	0.82	0.80
Fiber#4	1.000	0.9	0.85	0.89	1.04	1.08	1.11
Fiber#5	1.000	1	1.01	1.01	1.19	1.10	1.10
Fiber#6	1.000	1.1	1.14	1.20	1.42	1.42	1.38
Fiber#7	1.000	1.03	1.02	1.05	1.05	1.19	1.20
Fiber#8	1.000	1.01	0.98	0.93	0.94	0.95	0.99
R-value with %DOC of Primary coat	-	0.5	0.45	0.5	0.73	0.75	0.82

Table. 5 De-lamination percentages observed between the glass and the primary coatings of all test fibers at different aging day intervals

	Percentage De-lamination					
	Day1	Day3	Day7	Day14	Day21	Day30
Fiber#1	0	0	100	100	100	100
Fiber#2	100	100	100	100	100	100
Fiber#3	0	0	100	100	100	100
Fiber#4	0	0	10	10	50	100
Fiber#5	0	5	100	100	100	100
Fiber#6	0	0	0	0	0	0
Fiber#7	0	0	0	0	0	0
Fiber#8	0	0	0	0	0	0

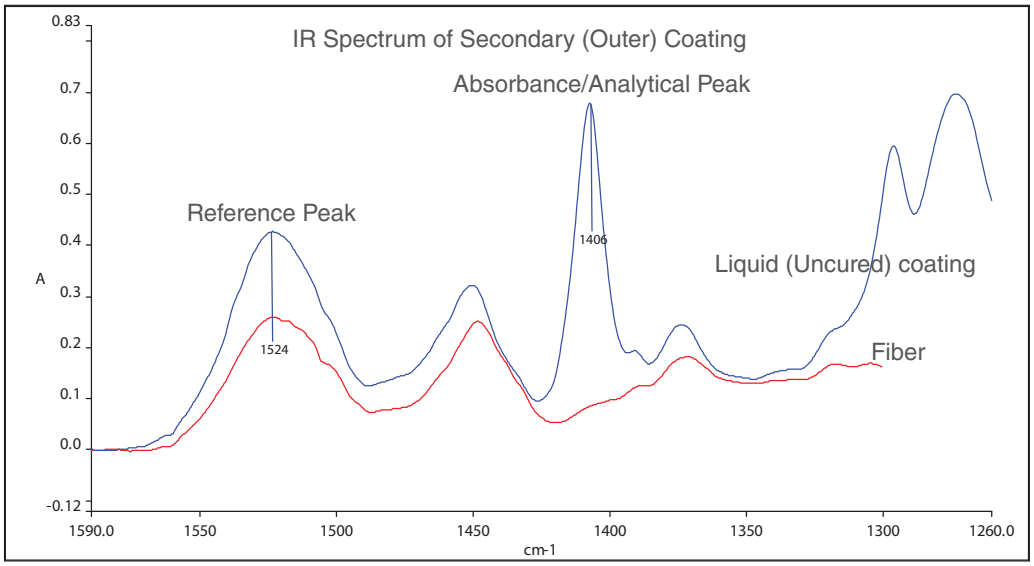

Fig.1 IR spectrum of secondary coating of both liquid and fiber show diminishing of absorbance peak at ~1406 cm-1 compare to reference peak at ~1520 cm-1

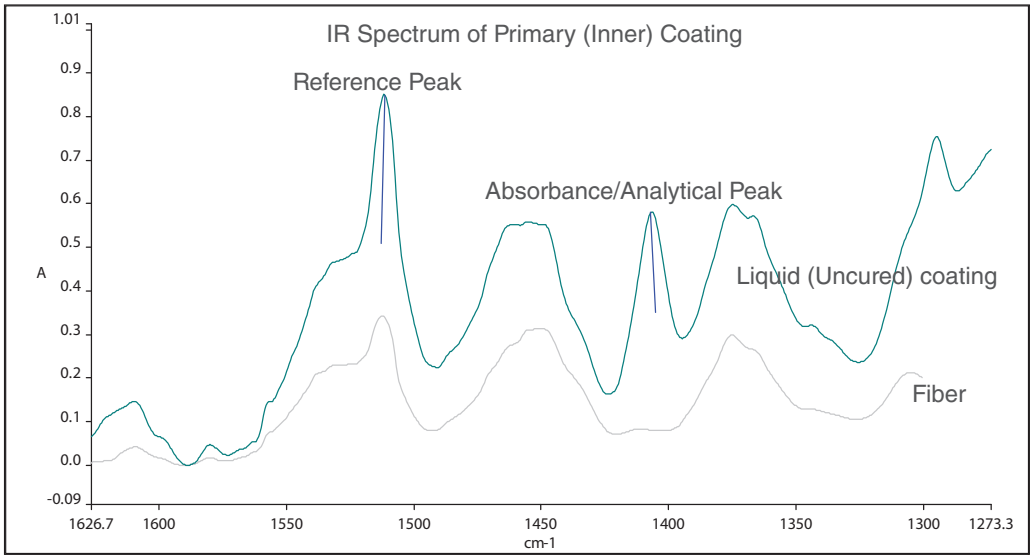

Fig.2 IR spectrum of primary coating of both liquid and fiber show diminishing of absorbance peak at ~1406 cm-1 compare to reference peak at ~1510 cm-1

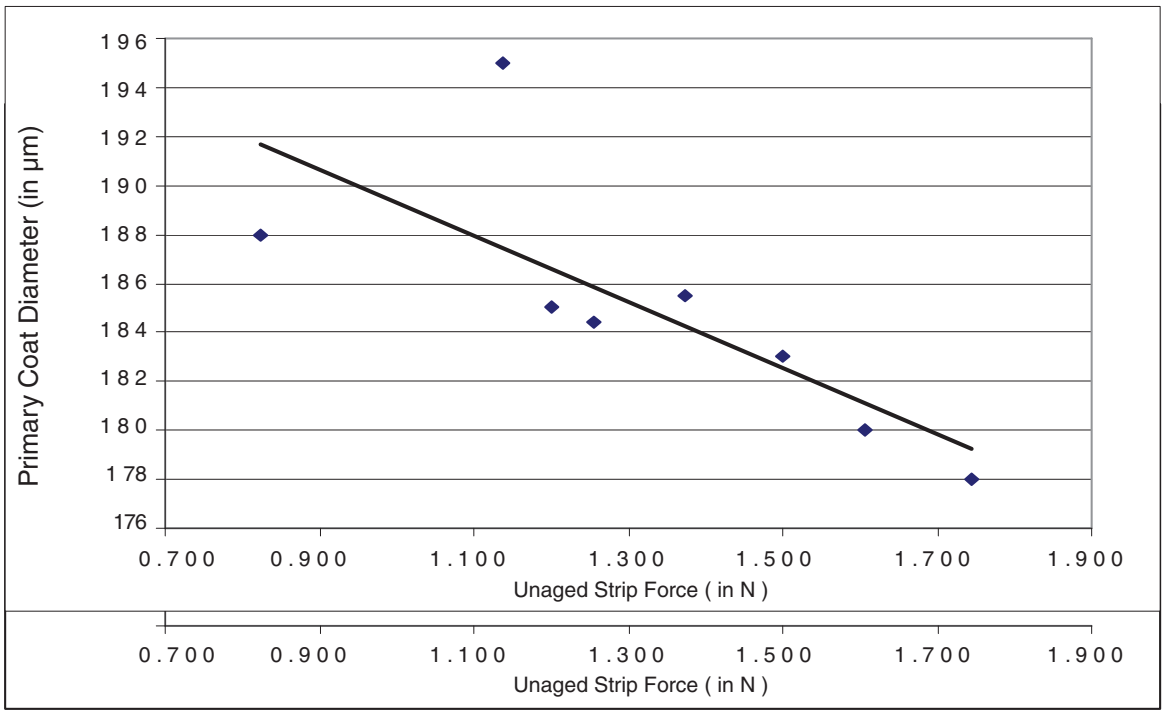

Fig. 3 Co-relation between Primary coat diameter and the unaged strip force

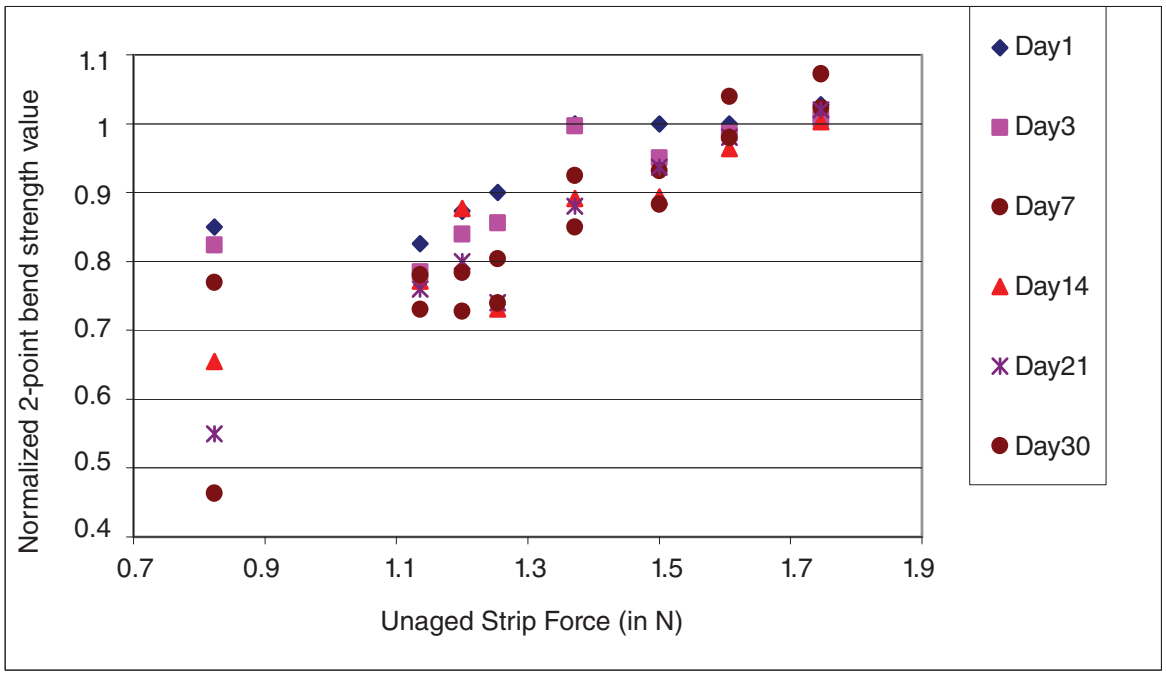

Fig. 4 Relationship between the unaged strip force and the normalized aged two-point strength value at different day intervals

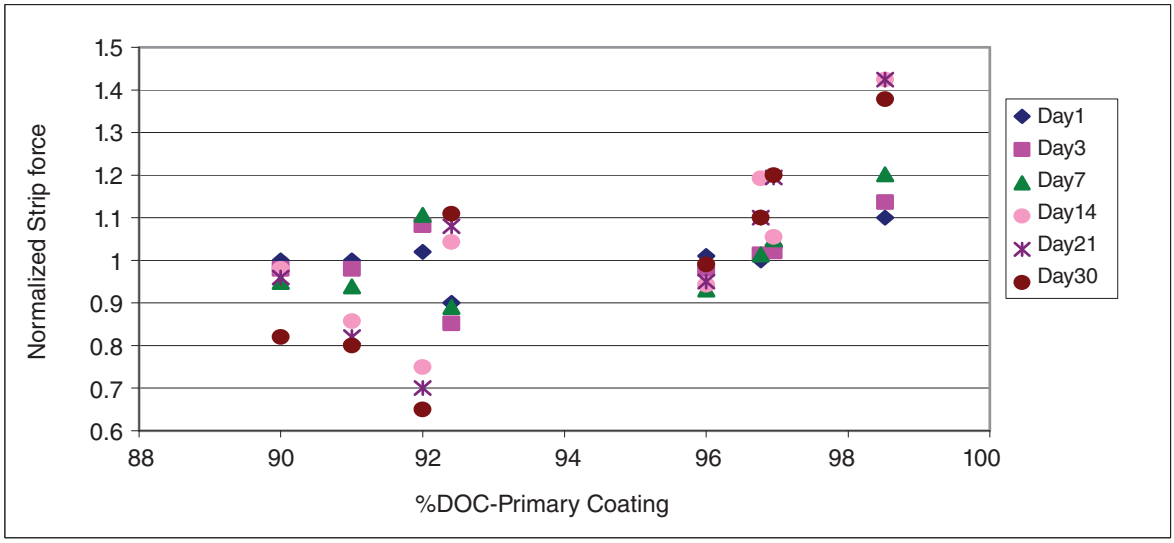

Fig. 5 Relationship between the %DOC primary coating and the normalized aged strip force value at different day intervals

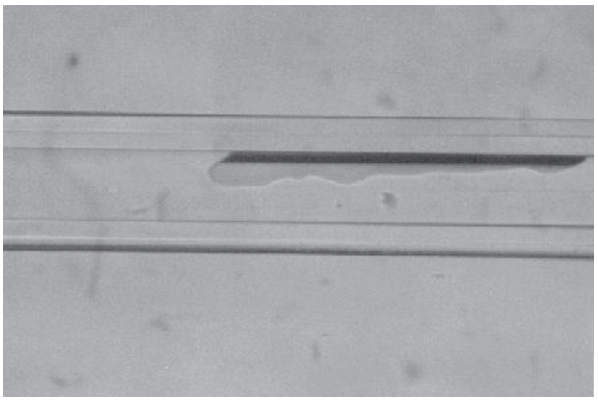

Fig. 6 Optical Micrograph of Fiber#4 after 7days of aging

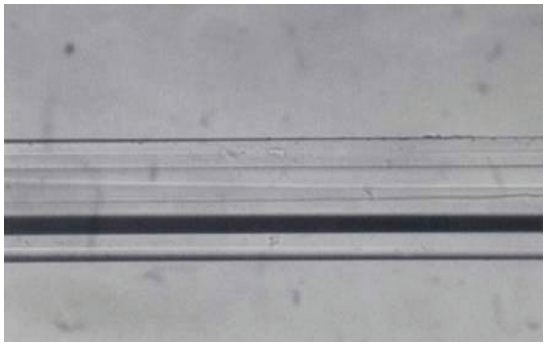

Fig. 7 Optical Micrograph of Fiber#5 after 7days of aging

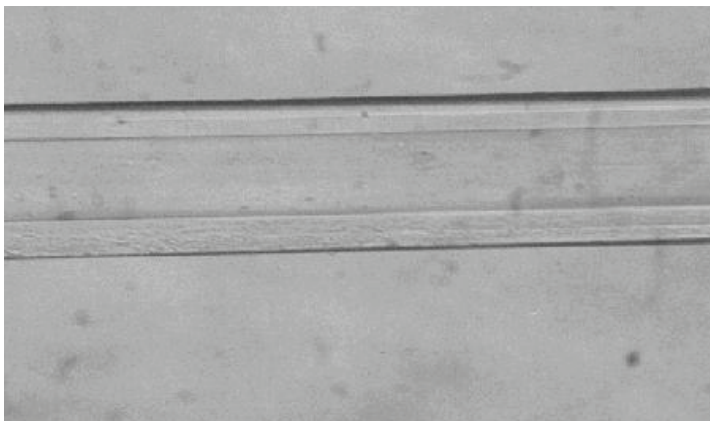

Fig. 8 Optical Micrograph of Fiber#6 after 30days of aging

About the Author

Sudipta Bhaumik is a Manager at Sterlite Optical Technologies Limited, Aurangabad, India. He joined Sterlite in 1998 as process development engineer. He received his bachelor's degree in ceramic engineering from Calcutta University, India and master's degree in ceramic engineering from Banaras Hindu University, India. He has been engaged with quality and reliability issues of optical fiber and development of analytical techniques.

He has received Urbain J.H.Malo medal (by Wire Association International, Boston, USA) award for best technical paper in Electrical/Fiber Optics division in 2002 and 2003.