Mechanical properties of polyimide coated optical fibers at elevated temperatures

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ABSTRACT

High temperature mechanical strength and reliability of optical fibers have become important subjects as optical fibers are increasingly used for harsher environments. Theories and models of fiber mechanical properties established for traditional telecommunications applications may need to be validated for applications at elevated temperatures. In this paper, we describe the test setup for high temperature tensile strength of fiber and report initial results of dynamic tensile strength of polyimide coated optical fiber at 300 and 350ºC for different heating time intervals. The results are compared with room temperature strength data, data available in the literature, and our earlier work on thermogravimetric analysis (TGA) weight loss of the polyimide coating and the observations on surface morphology at elevated temperatures. Interesting observations are discussed and possible explanations are proposed.

Keywords: Polyimide coating, optical fiber, high temperature, fiber strength, reliability, harsh environment

1. INTRODUCTION

In recent years, optical fiber has been more broadly adopted for commercial uses in harsh environment applications, such as medical probes that are sterilized at elevated temperatures or as distributed sensors in oil and gas pipelines and wells where environmental conditions are extreme. Concern over performance and reliability needs to be addressed in order for such fibers to be used successfully at elevated temperatures. However, the current body of knowledge covering mechanical properties and lifetime predictions for silica based optical fiber has been mainly established based on the data generated from experiments conducted over a relatively narrow range of temperatures, reflecting the environment of the fiber in optical telecommunications. Theories and knowledge regarding the fiber strength and reliability are essentially proven to be applicable to the comparably benign conditions of optical telecommunications. For the harsh conditions, new knowledge and theories may be needed.

Optical fibers are typically drawn and coated with polymer to protect the pristine "as-drawn" glass surface. Conventional optical fibers are most commonly protected with acrylate materials, providing an operating temperature ranging from -40 to + 85°C, with recent materials extending the upper limit to as high as 150°C. However, fibers for harsh environment applications are frequently coated with materials that can withstand higher operating temperatures. Polyimide is commonly used as a coating for optical fibers for harsh environment applications because of its superior properties including its hardness, high heat resistance and low coefficient of friction. Polyimide coated fibers are often used at temperatures up to 300°C.

At elevated temperatures, most polymer coatings degrade due to oxidation reaction when oxygen is present in the surrounding environment. Such reaction destroys the crosslinked network of the coating, followed by loss of volatile by-products. Once the materials loss reaches a critical point, performance degradation, optically and/or mechanically, begins.

In a previous work[1], thermal stability of optical fiber coatings was studied at high temperatures using TGA, a direct method of studying material degradation. In this method, the weight loss of the coating is recorded as a function of heating temperature and duration. Quantitative data generated through such experimentation allows us to estimate long-term use temperature for optical fibers coated with materials like polyimide coating. During our experimentation, we also studied the effect of thermal treatment at elevated temperature on surface morphology of the polyimide coated fiber

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samples, observing anomalies generated and impact on the fiber strength that had been previously tested at room temperature.[2]

To conduct this study, we modified a tensile tester by adding a furnace to heat the sample while straining the sample until fibers fractured. Exposure temperature was held at 300 or 350°C throughout the mechanical testing phase through various time intervals of heating. After working to validate the test method and isolate or eliminate various causes for low fiber breaking stress, we obtained reliable and repeatable test data. The samples tested have exhibited slight strength degradation over time. We then combined and compared the high temperature tensile data with the TGA weight loss curves and added correlative comparisons with previous work on surface morphology. Our observations are discussed below.

2. EXPERIMENTAL

2.1 Experimental setup

A common Stationary Capstan Fiber Tester (SCFT) was used to test the fiber strength, as shown in Figure 1. The fiber sample was held vertically and wrapped on capstans at both ends. The fiber sample was pulled vertically by a moving capstan at a constant rate. A strain rate of 6%/min was used in this study. The breaking stress was recorded at time of fiber fracture. To heat the sample to elevated temperatures during testing, a furnace is installed as illustrated in Fig. 1. Not shown in the illustration is a computer that controls the strain rate while recording the breaking stress. The gauge length for the test was 0.85 m. The upper and lower capstans are sufficiently large in diameter to eliminate undesirable stress applied on fiber under test. The entire setup was exposed to controlled ambient environments (RH = 50 ± 5%, T = 23 ± 2°C).

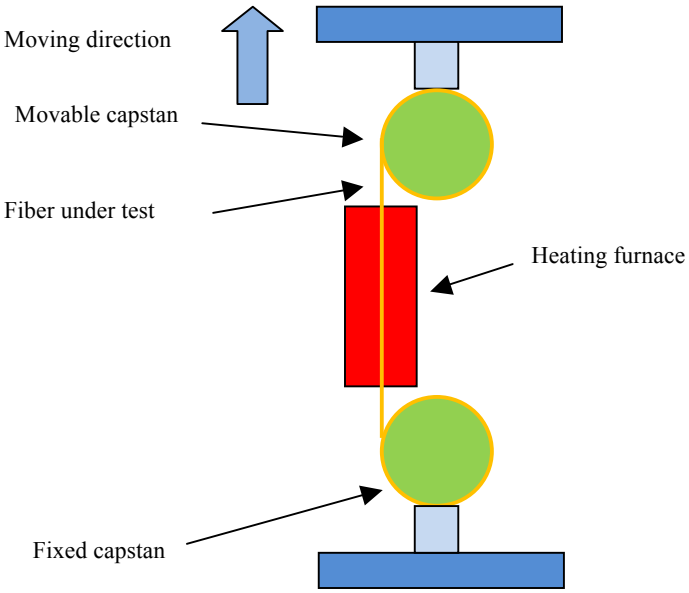

Figure 1. A schematic of a Stationary Capstan Fiber Tester (SCFT) with a heating element controlling the ambient temperature around the fiber under test.

Prior to high temperature exposure, samples of each fiber lot were evaluated at room temperature using the two-point bend technique. These tests were performed with a Fiber Sigma 2 Point Bend tester.[3] All strength testing was conducted at controlled humidity and temperature in accordance with a Telcordia GR-20 condition (RH = 50 ± 5%, T = 23 ± 2°C).[4] The samples were kept at least twelve hours at this condition before the testing, which is also required by the GR-20 standard.[4] The two-point bend tests were performed at a strain rate of 4%/min, and the median fracture stress was determined from the data.

All TGA measurements were performed using a TA Instruments TGA 2950 thermogravimetric analyzer. A more detailed description can be found elsewhere. [1]

2.2 Fiber sample and preparation

All fibers used in the study were produced by OFS and are described in table 1. By design, the polyimide coated fibers have a 125 µm silica glass cladding, and a coating diameter of 155 µm. For comparison, we also used a fiber having a commercially available dual acrylate coating. The dimensions of the acrylate coated fiber were 125 µm (silica glass cladding) and 245 µm (coating). For TGA measurements, the samples were segments of fiber < 1 cm in length placed in a platinum pan. Typical sample masses were in the range 10 – 40 mg. The sample chamber was purged using air at a flow rate of 60 cm^3/min. Since only the coating masses were of interest, the mass of the glass from the total mass measured by TGA was subtracted.

Table 1. Fiber samples used and measurements performed in the present study

Fiber sample	Silica cladding diameter	Coating diameter	Measurement
Dual-acrylate coated fiber	125 µm	245 µm	TGA
Polyimide coated fiber	125 µm	155 µm	TGA, Dynamic mechanical strength

3. RESULT AND DISCUSSION

3.1 Thermogravimetric analysis

3.1.1 Weight loss data as a function of heating temperature

TGA is used as a method of thermal analysis to study changes in weight of coated fibers, which are measured as a function of increasing temperature with constant heating rate. Fig. 2 represents a typical dynamic TGA curve collected at a heating rate of 0.5 ºC/min for polyimide coated fiber in air. The TGA data were also obtained for a fiber with a common dual-acrylate coating for comparison. As can be seen in the figure, the polyimide coating does not exhibit significant weight loss below 400 ºC, conversely, the acrylate coating starts decomposing above 250 ºC.

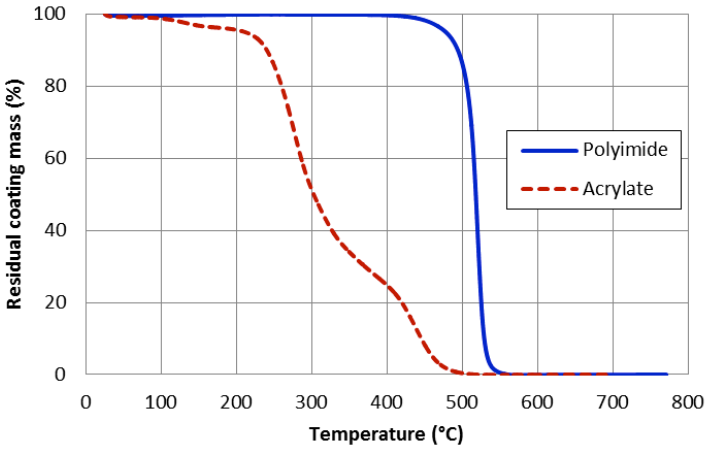

Figure 2. Dynamic TGA curves obtained for polyimide coating (blue solid line) and dual-acrylate coating (red dashed line) in air at a heating rate of 0.5 ºC/min.

3.1.2 Estimation of time to achieve 25% weight loss

The effect of coating weight loss on fiber properties may vary. For example, micro-bending induced attenuation in the fiber being heated may be observed at much lower weight loss than that for observation of degradation in fiber strength. Therefore, the extent of weight loss that is critical for different failure modes has to be determined experimentally. In this study, we selected, for the purpose of comparison, 25% weight loss as a failure criterion for coating. From the dynamic TGA experiments[1], the time to reach 25% weight loss can be calculated at any temperature. The results of such calculation are shown in Fig. 3. It can be seen that it would take many hundreds of hours to lose 25% of the polyimide coating weight at 300 and 350 °C. In contrast, for acrylate coated fibers, similar weight will be lost within several minutes.

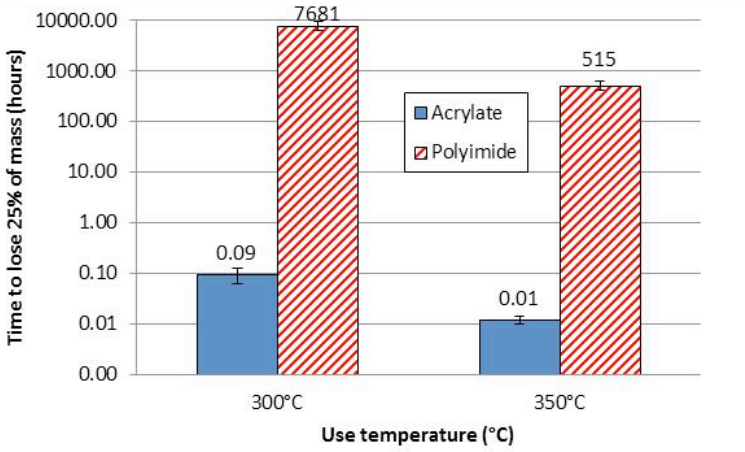

Figure 3. Time to achieve 25% weight loss for polyimide coating (patterned red bars) and dual-acrylate coating (blue bars) at 300 ºC and 350 ºC, respectively.

3.1.3 Weight loss data as a function of heating duration at 350 °C

An isothermal TGA experiment was also carried out to reveal the time dependence of coating decomposition at a constant temperature. The fiber samples were heated to 350°C at 75°C/min and then kept at the temperature for 24 hours. The obtained result is presented in Fig. 4 along with the curve for dual-acrylate coating samples. At 350°C, the polyimide coating lost about 2% of its initial weight within 24 hours. Note that this 2% weight loss is related not only to material degradation, but also to loss of volatiles (water and residual solvent). In comparison, a dual-acrylate coating loses about 76% of its initial weight during same exposure. The isothermal data are in fair agreement with predictions from dynamic TGA analysis.[1]

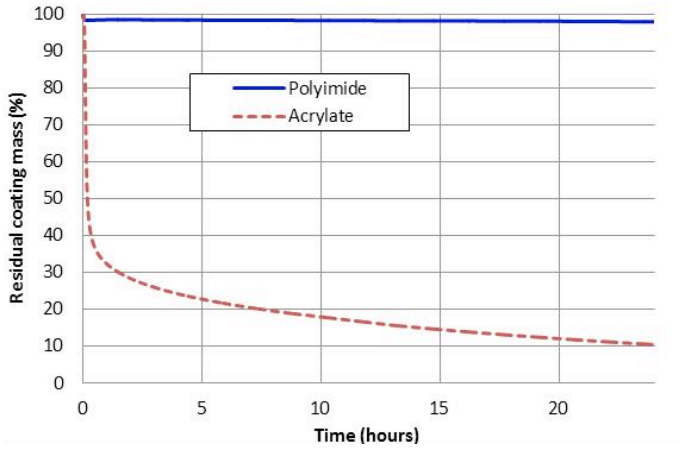

Figure 4. Isothermal TGA curves obtained for polyimide coating (blue solid line) and dual-acrylate coating (red dashed line) in air at 350 ºC.

3.2 Mechanical strength at elevated temperatures

3.2.1 Dynamic tensile strength as a function of temperature

Fiber samples were loaded into the tensile tester at room temperature before heating started and actual tests began as soon as the temperature stabilized at the set point, which took typically a few minutes. We selected 300ºC as a starting point for dynamic tensile strength aging study. Surprisingly, no evidence of significant degradation in strength was observed even after aging for 72 hours. To accelerate the change, we increased the temperature to 350°C. The fiber strength after 60 minutes soaking at 350 ºC decreased and the data variance for strength became greater. The resultant dynamic tensile strength of polyimide coated fiber samples are displayed in Fig. 5.

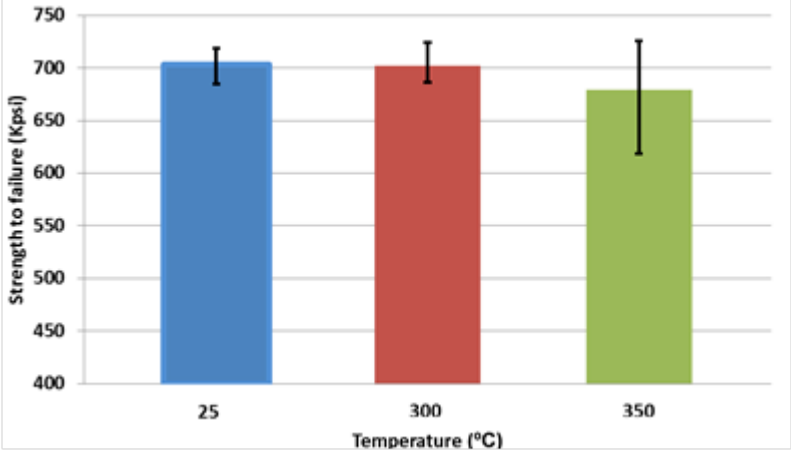

Figure 5. Dynamic tensile strength results of polyimide coated fiber at room condition (blue bar on the left), 300 ºC for roughly 10 mins exposure (red bar, middle) and 350 ºC for roughly 60 mins exposure (green bar, right), respectively. The data points correspond to medians while the error bars show the maximum and minimum observed values.

It is interesting to note that this weak dependence of the strength on the temperature up to 350°C is not predicted by the Arrhenius model proposed based on a strong temperature dependence of silica fiber strength in the temperature from 77 to 473 K.[5] In that paper, both bare glass fiber and hermetically coated fiber were also studied for strength in a temperature range from room temperature to 200°C. The measured strength was observed to decrease as the temperature increases, suggesting a strong temperature dependence.

Our data suggest that the fiber strength remains unchanged as the temperature increases from room temperature to 300°C as long as the coating remains intact and provides sufficient protection. This is supported by the TGA data for polyimide coated fibers illustrated in Figs. 2 and 4.

A possible explanation is that the amount of moisture or other attacking chemicals diminished at temperatures >300°C in the surrounding environment. Alternatively, we speculate that thermal stress created on glass due to the coefficient of thermal expansion mismatch of silica and polyimide is much less than that between glass and metal coating used in the earlier study.[5]

3.2.2 Dynamic tensile strength as a function of time

As mentioned earlier, 300°C was initially selected as a starting point to test fiber strength as a function of time. However, the fibers exhibited no significant degradation of strength even after up to 72 hours. In order to accelerate the experiment, 350°C was selected. The results are displayed in Fig. 6. A significant decrease in strength and increase in data variance were observed. This decrease is inconsistent with the TGA data shown in Fig. 4, which indicates no significant weight loss for polyimide coating at 350°C up to 24 hours. While TGA characterizes material properties of the coating, it may not provide information on localized changes caused by high temperature in the coating. This degradation in fiber strength is suggested to be caused by a different mechanism of the coating.

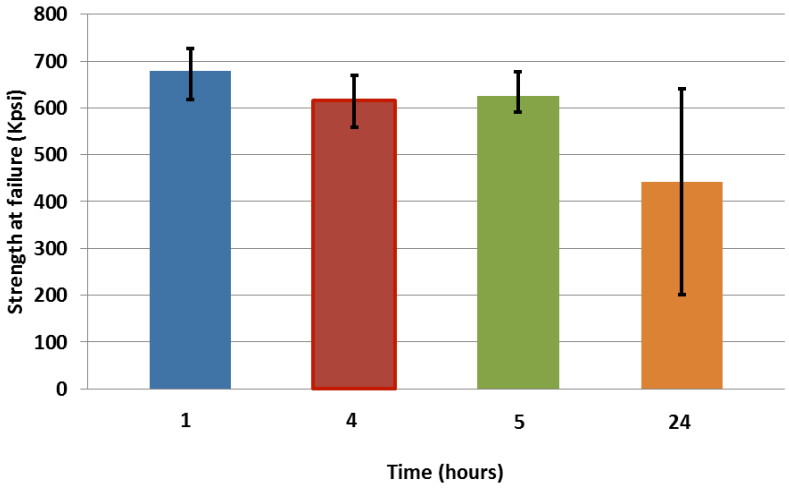

Figure 6. Dynamic tensile strength results of polyimide coated fiber at 350 ºC for dwell time of 1 hr (blue bar), 4 hrs (red bar), 5 hrs (green bar) and 24 hrs (orange bar), respectively. The data points correspond to medians while the error bars show the maximum and minimum observed values.

3.2.3 Observation of changes in surface morphology

To fully understand the high temperature behavior of the fiber, it is necessary to study the effect of high temperature on the coating as the integrity of the coating dictates the strength of the glass fiber. Glass is a brittle material and its strength is determined by the quality of its surface. Visual inspection of the coating surface is a direct method that may provide critical insight. In an earlier study[6], OFS observed the formation of crater-like defects on the coating surface of the polyimide coated fiber samples treated at elevated temperatures. These defects were identified as root cause for mechanical weak performance of the fiber under study. The research concluded that the craters could reach the glass surface leaving it unprotected and subsequently triggering critical crack growth, resulting in fiber fracture. Although tests were performed at room temperature in our previous work, it is reasonable to assume the crater defects could also form during our high-temperature strength tests. This may be a possible explanation of the fiber fracture at elevated temperatures where weight loss of the coating is insignificant. The crater formation mechanism deserves a closer examination.

In a further examination, it was theorized that the craters were initiated at the locations of small-scale debris on the coating surface. To confirm this, the following experiment was performed. Two sets of fiber samples were prepared with one set was carefully cleaned while the other one was intentionally subjected to dust (Fig. 7). The samples were then placed vertically in a thermal oven and simultaneously aged in ambient air at 400 ºC for 24 hours. It can be clearly seen in Fig. 7 (d) that the craters developed on the "dusty" sample surface as consequences of dust particles being "burned-off" and generating very high localized temperatures. Following the aging experiments, two-point bend strength testing was carried out to examine the effect of these craters on strength. Strength data summarized in Table 2 suggest that these craters significantly degraded the mechanical strength of the fiber under test. Note the Weibull slope also changed from a baseline of 116 to 3.3, indicating a much more scattered strength distribution. It is believed that generation of such craters significantly reduces or eliminates the coating locally, exposing the silica glass surface to environmental moisture and significantly reducing the fiber strength and long term reliability.

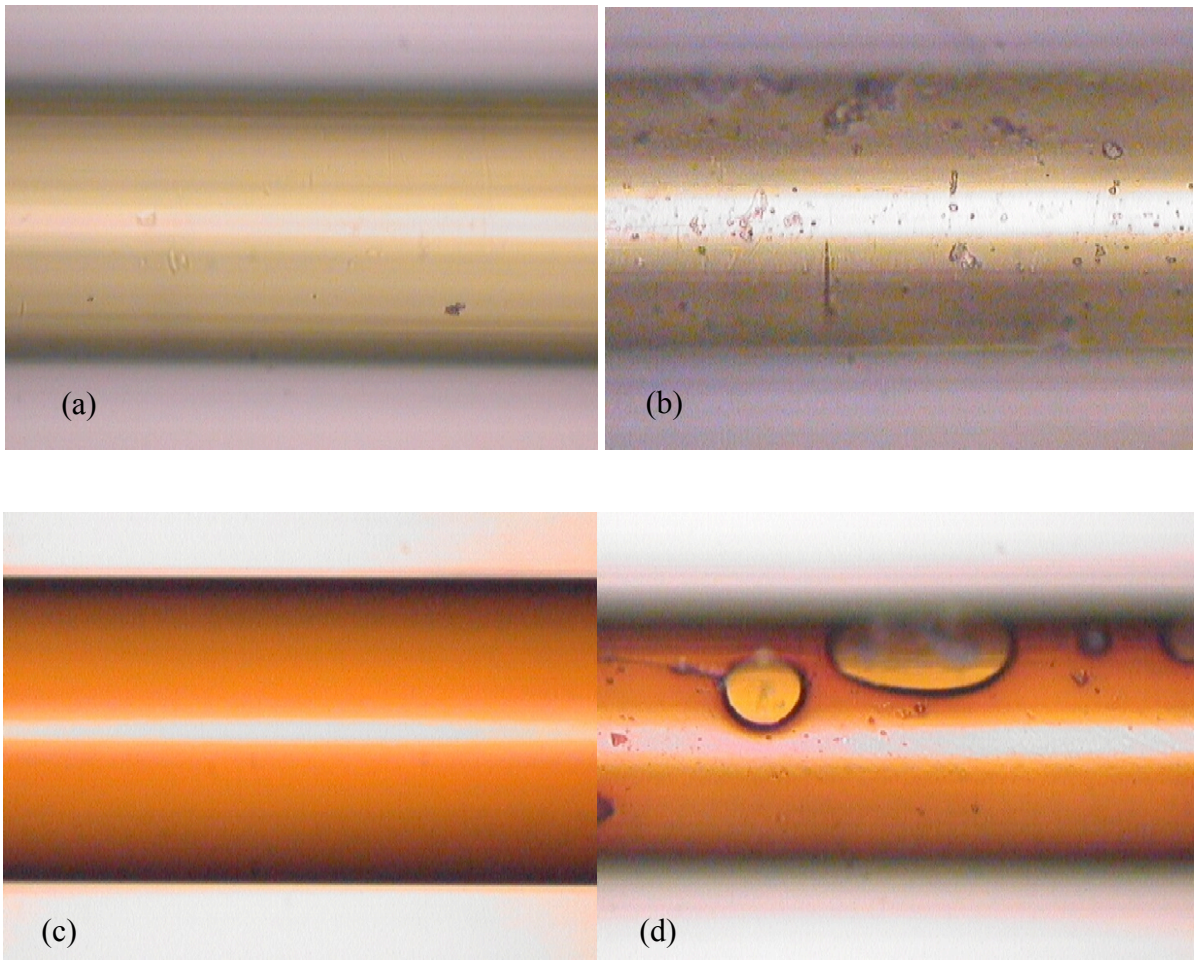

Figure 7. Microscope images of carefully cleaned polyimide coated fiber before (a) and after (c) 24 hour aging at 400°C, and "dusty" fiber sample before (b) and after (d) 24 hour aging at 400°C.

Table 2. Two-point bend strength data of unaged, carefully cleaned and "dusty" polyimide fiber samples

Fiber sample	Median strength (kpsi)	Weibull slope
Unaged	828	116
Aged at 400 ºC for 24 hrs - cleaned	784	42
Aged at 400 ºC for 24 hrs - dusty	526	3.3

This observation provides an explanation for the degradation in strength of polyimide coated optical fibers at a temperature where no significant coating weight loss is observed. The greater scatter of the strength data can also be explained by the random nature of the distribution of dust particles on coating surface.

Based on our results in high temperature strength, TGA, and surface morphology, we may summarize our observations and analysis as follows: Polyimide coated silica based optical fiber does not show a significant degradation in strength as predicted by an Arrhenius-like behavior. A likely cause is the reduced amount of moisture or other attacking chemicals that exist during the test at elevated temperature. Another likely cause may be the mismatch in coefficient of thermal expansion between silica and polyimide that is small so that it does not result in significant surface tension on glass

surface. Large tension on the glass surface is known to weaken the fiber. Polyimide coating retains its mechanical integrity at temperatures up to 400°C as the TGA weight loss data shows essentially no loss below that temperature. Therefore the polyimide coated silica fiber should show no degradation in strength at temperatures, e.g. 300°C. Our high temperature strength data, especially for the short heating durations appear to support the assertion. The long and slow degradation may be explained to be caused by the slow formation of the coating surface defects such as the crater-like features on the surface.

4. CONCLUSIONS

In summary, we have established a test setup and a reliable method to test the strength of optical fibers at elevated temperatures in order to investigate the high temperature behavior of optical fiber and generate fundamental knowledge for applications of optical fibers in harsh environments. Our initial test data on polyimide coated fiber, combined with our TGA data of the coating and surface morphology, revealed some interesting results that have not been reported before. Our high temperature strength data for short heating duration indicate no significant degradation which is not predicted by the generally accepted Arrhenius-like behavior model. TGA data shows that the polyimide coating will maintain its mechanical integrity at temperatures up to 400°C. These two observations seem to indicate that the coating integrity is the weakest link in the strength degradation of the coated fiber. The degradation in strength for longer heating durations suggest some different failure mechanisms other than glass surface crack growth and coating intrinsic degradation due to thermal weight loss. Our work on surface defects formation during high temperature treatment provide a possible explanation for the long and slow strength degradation observed. To fully understand the mechanism and to explore the relationships among the variables, work will continue.

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