

# Fatigue behavior of polyimide coated optical fibers at elevated temperatures

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## ABSTRACT

As optical fiber is being used in much harsher environments than traditional telecommunications (e.g. distributed temperature sensing at elevated temperatures) understanding its mechanical properties at high temperatures is urgently needed. As a continuation of our previous work on high temperature strength of silica optical fiber, we report our results in fatigue behavior of polyimide coated silica optical fiber at 300°C in this paper. Fiber fatigue is the degradation in strength caused by a stress dependent chemical reaction between water vapor and the surface of the silica glass. In contrast to the published data on degradation in mechanical properties of silica optical fiber at elevated temperatures, our observations indicate a negligible decrease in strength along with unchanged,  $n$ -value, (fatigue resistant factor) at 300°C. To determine the  $n$  value, we tested tensile strength of the fiber using four different strain rates while the subject under test was at 300°C. The results indicate that the polyimide coating on the silica glass fiber continues to serve as an effective water vapor barrier at 300°C. These results will be compared with data available for room temperature performance of this silica/polyimide combination and possible failure mechanisms will be discussed.

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

## 1. INTRODUCTION

Optical fiber has significantly expanded its role as an enabling technology in many applications from original telecommunications. It can be used as a waveguide for power delivery or a sensing medium in sometimes much harsher environments, such as in medical probes that are sterilized at elevated temperatures or as distributed temperature sensors in oil and gas wells and pipelines applications. Thus, its survivability and reliability in these environments, both mechanically and optically, present a serious concern for its successful adoption.

Optical fibers are typically made by drawing a glass preform into optical fiber and immediately coating the fiber with a polymer, such as acrylate, to provide mechanical protection for the pristine “as-drawn” glass surface. However, since silica optical fiber is expected to degrade in strength under load due to a stress-dependent chemical reaction between water vapor and the surface of the silica glass fiber, known as fatigue, an important role for the coating on the fiber is to reduce fatigue by providing a barrier to water vapor reaching the glass surface. Therefore, for both fiber strength and fatigue resistance, coating is crucially important. The effectiveness of the coating as a barrier for water vapor can be characterized by the fatigue resistant factor, or  $n$  value. Since silica optical fiber shows a dependence of fracture stress on the rate of strain loading, a phenomenon known as dynamic fatigue, the fatigue resistant factor, or  $n$  value, can be experimentally determined.

While the performance of acrylate coated fiber had been well suited for use in the relatively benign telecommunications environment, polyimide coated fibers are often selected for much harsher environments because of polyimide’s superior properties including its high heat resistance (up to 300°C), hardness, and low coefficient of friction. However, the current body of knowledge of mechanical properties and lifetime predictions for silica optical fiber has been mainly established for acrylate coated silica optical fiber based on the data generated for a narrow range of temperatures, around room temperature, representing the benign environments of the fiber in optical telecommunications. In order for the polyimide coated fiber to be successfully used for harsh environments, optical and mechanical performance of fibers at high temperatures need to be understood. This does not merely imply subjecting polyimide coated fibers to high temperatures and subsequently performing optical and mechanical tests at room temperature, rather, it suggests a need to

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perform some of the optical and mechanical tests while the subject fiber is at elevated temperature. More specifically, the tests include the temperature dependence of glass intrinsic strength and thermal stability of the coating as a protective layer and as a barrier for water vapor diffusion. Consequently, effective solutions based on the new knowledge learned can be developed.

In a previous study of fiber strength at elevated temperatures<sup>1</sup>, we observed that the strength of polyimide coated silica optical fiber remained unchanged during short aging time even as the temperature had increased from room temperature to 300°C. The lack of strong dependence of fiber strength on temperature is not predicted by the generally accepted Arrhenius-like behavior model<sup>2</sup>. Interestingly, this observed independence was accompanied by the result of thermogravimetric analysis (TGA) of the polyimide coating which exhibits no significant weight loss for the short heating duration. It seems to suggest that the glass retains its strength as long as the coating is functioning as an effective water vapor barrier. At longer heating durations, formation of surface irregularities on coating was observed and was proven to cause the fiber strength degradation. The coating surface irregularities were shown to be the damage to the coating at high temperature catalyzed by surface contamination. If the fiber surface was cleaned to reduce contamination, fiber strength degradation was significantly alleviated. Therefore the slow degradation in strength at 300 °C for longer heating durations appears to be the direct result of the coating damage that is caused by contaminants, such as dust particles. However, it is conceivable that as the weight loss continues the coating will lose its effectiveness as a water vapor barrier at some point and fiber fracture will occur even in the absence of the surface contamination. In this work, we test the fatigue behavior of the same fiber at 300°C using standard procedure for determining the fatigue resistant factor, the  $n$  value, compare the high temperature results with room temperature results, and provide some insights into the failure mechanism of the fiber at elevated temperatures.

## 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. Four different strain rates (28%/min, 4%/min, 0.57%/min and 0.08%/min) were used to study the fatigue behavior. 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 in order to keep the bending induced stress on the fiber under test sufficiently low. The entire setup was maintained in a controlled ambient environments ( $RH = 50 \pm 5\%$ ,  $T = 23 \pm 2^\circ\text{C}$ ).

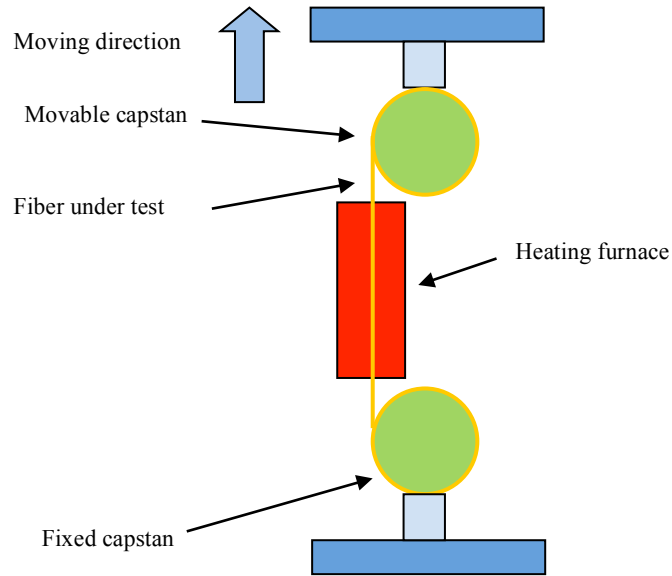


Figure 1. A schematic of a Stationary Capstan Fiber Tester (SCFT) with a heating element controlling the ambient temperature around the fiber under test.

## 2.2 Fiber sample and preparation

All fibers used in the study are polyimide coated silica optical fibers, manufactured by OFS, with a glass clad of 125  $\mu\text{m}$  in diameter and coated fiber diameter of 155  $\mu\text{m}$ , as described in table 1. Prior to each measurement, each fiber sample was carefully cleaned with ethanol to remove dust particles that could lead to the formation of surface irregularities during high temperature treatment.<sup>1</sup>

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

Fiber sample	Silica cladding diameter	Coating diameter	Measurement
Polyimide coated fiber	125 $\mu\text{m}$	155 $\mu\text{m}$	Dynamic mechanical strength

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 takes typically a few minutes to reach 300°C.

## 3. RESULT AND DISCUSSION

### 3.1. Weibull distributions and their strain rate dependence at 300°C

Fig. 2 shows four typical Weibull plots for the polyimide coated fiber tested for tensile strength at four different strain rates with 15 measurements performed at each rate. All the plots for the polyimide coated fiber at 300°C are nearly vertical and with a tight distribution indicated by large Weibull slopes, as seen in Table 2. They are similar to the plots generated for polyimide coated fiber at room temperature indicating similar strength to that of pristine fibers, typically tested at room temperature without high heat exposure as shown in Fig. 3. The vertical lines shown in the Fig.2 indicate excellent fiber strength at 300°C with no appreciable weak points. It clearly displayed a strong dependence of fiber strength on strain rate as expected. The mechanical strengths of fiber samples gradually shifted to the lower values as the strain rate decreases. This behavior is known as dynamic fatigue. Dynamic fatigue data enable us to determine the fatigue resistant factor, the  $n$  value, experimentally.

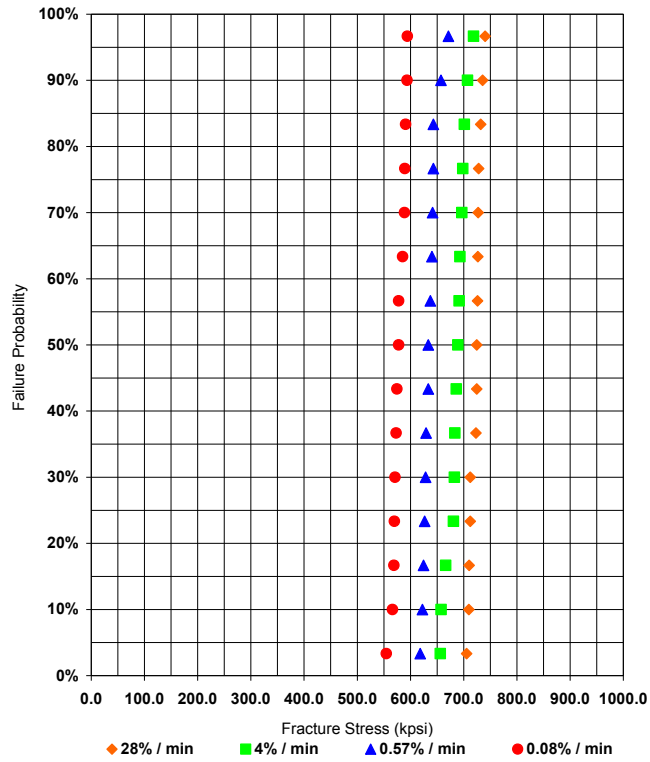


Figure 2. Weibull plots of polyimide coated fiber at four different strain rates at 300°C.

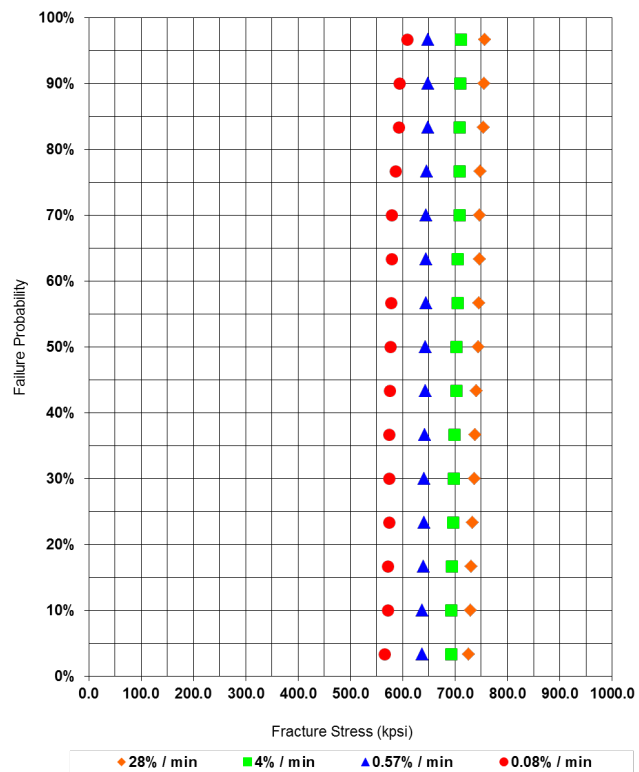


Figure 3. Weibull plots of polyimide coated fiber at four different strain rates at room temperature.

The measurement results are summarized in table 2. The Weibull slope at each rate indicates a tight distribution of mechanical tensile strength, suggesting a single-modal strength regime.

Table 2. Mechanical tensile strength data of polyimide fiber samples at different strain rates at 300°C

Strain rates	Median strength (kpsi/GPa)	Weibull slope
28%/min	724.45	82.19
4%/min	687.19	47.67
0.57%/min	636.65	84.63
0.08%/min	578.37	64.53

The dynamic fatigue data for polyimide coated fiber at 300°C, displayed in Weibull plots in Fig. 2, taken at four different strain rates are also plotted in an ln – ln format in Fig.4 to determine the  $n$  value. The fatigue resistant factor is therefore calculated based the well-established theories for glass fatigue using the following equation as 25.4.<sup>3</sup>

$$\ln \sigma_1 - \ln \sigma_2 = n (\ln \dot{\epsilon}_1 - \ln \dot{\epsilon}_2) - 1, \quad (1)$$

$\sigma_1$  and  $\sigma_2$  are the different strain rates used,  $\dot{\epsilon}_1$  and  $\dot{\epsilon}_2$  represent strains at which the fiber breaks. To compare the  $n$  value for the fiber at room temperature, an identical polyimide coated fiber was tested at room temperature and controlled humidity and in accordance with the Telcordia GR-20 condition (RH = 50 ± 5%, T = 23 ± 2°C).<sup>4</sup> The data are shown in Fig.5. The result, 22.6, turns out to be lower comparing to the value at 300°C. Given the statistical nature of mechanical strength test, our data suggests that the fatigue behavior of the fiber under study is at least not very sensitive to temperature within the considered temperature range (up to 300°C).

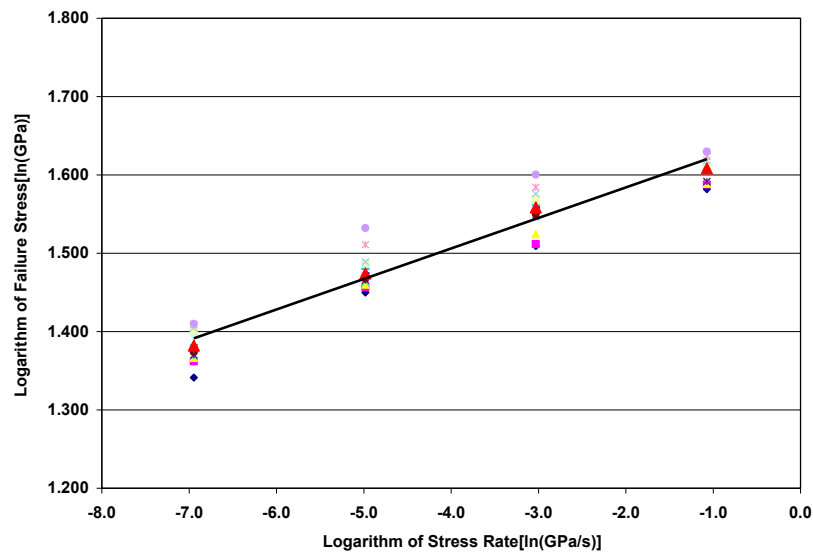


Figure 4. Fatigue of polyimide coated fiber at 300°C.

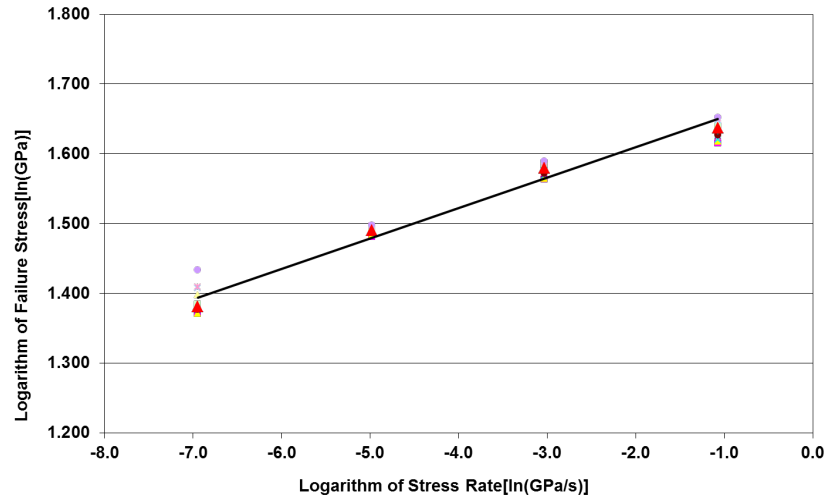


Figure 5. Fatigue of polyimide coated fiber at room temperature.

The polyimide coating previously showed negligible weight loss at 350°C up to 24 hours<sup>1</sup>, indicating the coating would remain intact at 300°C for the same duration, therefore providing sufficient protection to the silica surface of the glass optical fiber. The fatigue behavior observed in the present study further supports that not only the fiber strength remains unchanged as the temperature increases from room temperature to 300°C, but also that the fatigue behavior is retained similarly, as long as the coating remains intact. It is possible that the amount of moisture or other attacking chemicals are reduced significantly at temperatures >300°C, the absence of which may make the optical fiber insensitive to elevated temperatures where strength and fatigue resistance are concerned.

In contrast, hermetically coated fiber was also studied for fatigue behavior in a temperature range from room temperature to 200°C in an earlier study<sup>2</sup>. The results reported in that paper demonstrate a fatigue resistant factor as a linear function of reciprocal temperatures, indicating a strong dependence on temperatures. It is worth mentioning that at all studied temperatures, the fatigue resistant factor remained large enough (>100), likely suggesting that fatigue behavior of silica optical fiber might respond differently to environments when  $n$  value exceeds some limit. An alternative explanation is that the metal coated fiber studied in that literature would behave differently from polymer coated fiber within the considered temperature range. Further study is required to fully understand these characteristics.

Based on our results in high temperature strength and dynamic fatigue, we summarize our observations and analysis as follows:

Polyimide coated silica based optical fiber show a well-defined fatigue behavior at 300°C, that the measured mechanical tensile strength would decrease with the strain rate. The stress corrosion parameter is calculated to be 25.4 at elevated temperatures, comparing to the value measured at room temperature, 22.6. A likely explanation is the reduced amount of moisture or other attacking chemicals that exist during the tests at elevated temperature, causing the fiber to be more resistant to fatigue. The result indicates that the fatigue behavior of polyimide coated fiber remains the same as the temperature increases from room temperature to 300°C as long as the coating remains intact and provides sufficient protection.

#### 4. CONCLUSIONS

We established a test setup and a reliable method to test the fatigue behavior 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. At 300°C, the polyimide coated fiber shows a well-defined fatigue behavior, similar to that of fibers tested in room temperature conditions. The fatigue resistance factor derived at 300°C essentially remained unchanged from the value at room temperature, which probably is due to the reduction of moisture or other attacking chemicals at such condition. This observation suggests that the coating provides sufficient protection at 300°C.

More work will be conducted in order to fully understand the mechanism and to explore the relationships among the variables.

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