Hybrid glass coatings for optical fibers: effect of coating thickness on strength and dynamic fatigue characteristics of silica fibers

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ABSTRACT

Specialty optical fibers operating in harsh aerospace environments are typically exposed to high temperatures and elevated humidity. This calls for better performing protective coatings. Recently developed sol-gel derived inorganic-organic hybrid materials called hybrid glass offered improved protective performance as compared to standard dual polymer coated fibers [1]. In this paper we examine the effectiveness of online UV curing for the protective ability of hybrid glass coatings. For this purpose two types of UV-curable hybrid glass candidates representing two different concentrations of acrylate groups were applied online to silica fibers as single and dual coats. Samples of fibers were collected and subjected to dynamic fatigue testing by two-point bending. The stress corrosion parameter, *n*, as well as the strength of the fibers were determined. Both the strength and *n* were higher for fibers with two layers of coating as compared to single coatings even when the thickness of both one and two layer coatings was the same. This may be caused by the greater degree of cross linking of the inorganic component when the coating is exposed twice to the heat generated in the UV chamber. Coating materials with reduced acrylate group content had higher values of the fatigue parameter *n* but at the same time reduced strength.

Keywords: hybrid glass, protective coatings, optical fibers, reliability, dynamic fatigue.

1. INTRODUCTION

The beneficial properties of hybrids generated by the sol-gel process are well known and broadly described in the literature [2,3]. They combine flexibility and mechanical strength of the organic constituent with the hardness, stiffness, and transparency of the inorganic oxide network – which, in most cases, is a silica network. In our previous studies we recommended sol-gel derived and UV-curable hybrid materials, called hybrid glasses, as protective coatings for optical fibers. The results showed that the hybrid glass materials afforded enhanced corrosion and mechanical protection of the fiber compared to standard polymer coatings used by the fiberoptics industry. These coatings are hard, transparent and impossible to strip since they become intimately bonded to the fiber surface during curing. They are therefore more resistant to abrasion than polymer coatings.

One of the difficulties in application of these hybrid materials is obtaining sufficient cure online since sol-gel derived inorganic oxide components are known to be condensed (solidified) by heat but not by UV irradiation. Our previous results showed that a short UV exposure seems to cure not only the polymeric component but also enhances condensation of the inorganic component as well, presumably because of exposure to the heat of the UV chamber rather than the UV radiation itself [1,4]. Curing, in turn, leads to permanent bonding of the coating material to the fiber surface. It was found that the thickness of the coatings plays some role; thinner coatings tend to exhibit higher strength and *n* value than the thicker ones [1,4].

In this study we examine more closely the effect of the coating thickness and chemistry on the mechanical properties of the silica fibers coated with hybrid glass coatings. For this purpose two types of coating, HG-4 and HG-4MP, were applied to silica fibers as one or two layer coats and UV-cured online. The content of polymerizable acrylate groups in

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coating HG-4MP was 20% lower than in HG-4. It was expected that by reducing the amount of acrylate groups the thermal stability of the coated fiber as well as its mechanical strength would be enhanced.

The drawing and coating experiments produced four fiber samples that were subsequently subjected to dynamic fatigue testing in two point bending. This study highlights the results of fatigue testing and relates them to the coating thickness, application process and coating chemistry.

2. EXPERIMENTAL

2.1 Hybrid Glass Coatings

The coating materials were synthesized by a well-controlled, cost effective one-step sol-gel process, which was described in detail elsewhere [5]. Coating formulations constitute clear, transparent, low viscosity, solvent-free liquids that are stable with a shelf life exceeding four months. The two hybrid glass formulations, HG-4 and HG-4MP, were silica based hybrids with almost identical chemistries that differed only in the amount of polymerizable acrylate groups. HG-4MP had 20% fewer such groups than the HG-4 formulation.

2.2 Fiber Drawing

Fibers were drawn from pure silica Suprasil 300 F rod. The fiber glass diameter was 125 μm for the HG-4 samples and at 200 μm for the HG-4MP samples. Because of differences in viscosity, the actual thickness of the coatings differed for the different formulations – glass and coating thicknesses are listed in parentheses in Table 1. Fibers were drawn at speed 20 m/min in an argon atmosphere. Stainless steel coating applicator cups were employed and a UV chamber equipped with a 100 W/cm^2 mercury lamp was used for curing. For one specimen (fiber 4) an additional heat cure was applied online by a furnace set at 400°C.

2.3 Dynamic Fatigue by Two-Point Bending

The dynamic fatigue of coated silica fibers was measured in the manner described in detail by us earlier [1]. Briefly, the fibers are bent between two faceplates that are brought together until the fiber breaks. An acoustic transducer detects the breaks and the failure stress, σ_f, is calculated from the separation distance between the faceplates at failure. For experiments described in this study all testing was performed in a controlled environment at 25±0.5°C and a humidity of 50±5%. Fibers were preconditioned in this environment for at least 12 hours prior to testing. The two-point bend strengths were measured at four different faceplate velocities (5, 50, 500 and 5000 μm/sec). Approximately 15 fiber specimens were broken at each rate; the log-log dynamic fatigue plots presented here show the (geometric) mean strength and a 95% confidence interval for the mean at each faceplate velocity. Linear regression was used to determine the stress corrosion susceptibility parameter, n, using methods specified in widely used standards documents [*e.g.* 6,7].

Tensile testing, for example of 0.5 m gauge length specimens, is a common method for characterizing optical fiber strength. However, the tensile strength is sensitive to the presence of occasional extrinsic defects that arise during manufacture and handling. However, for the purposes of this work we wish to study the effect of the coating on the fiber strength and are not interested in extrinsic defects which are more a reflection of the quality of manufacture. In contrast to tension, two-point bending has an extremely short effective test length (on the order of tens of micrometers[8]) and so it is unlikely that extrinsic defects will be encountered. A further advantage of two-point bending is that it is much easier to use than tension and can test multiple specimens simultaneously – this makes it an ideal tool for assessing large numbers of different coating formulations. However, since the coatings are manufactured in a research environment, they are not as free of contaminating particles as a commercial coating material and so occasional low strengths are encountered. These low strengths are readily identified since they are significantly weaker than the high strength mode observed on a Weibull probability plot. They have been eliminated from the data analysis used here. This reduces the uncertainty in the measured strengths and fatigue parameters while not significantly biasing the results. Typically only one or two outliers are eliminated from the 60 measurements used to calculate n.

The calculation of failure stress for two-point bend measurements assumes that failure originates at the surface of the glass fiber on the tensile surface on the outside of the bend. The hybrid coatings studied here are hard and brittle compared to polymer coatings but will not be as hard and brittle as the silica fiber. However, there is a possibility that failure originates from the outer surface of the coating, resulting in a crack which propagates through the coating into the fiber. While we have not found fractographic evidence for this (the strengths are too high to retain the original fracture surfaces intact), if this were the case then the calculations of failure strength would underestimate the actual bending or tensile strength of the fiber. In other words, if the failure occurred in the coating rather than the fiber, the results we report here would be too low and so a very conservative estimate of strength.

3. RESULTS AND DISCUSSION

3.1 Strength and fatigue parameter by Two-Point Bending.

Dynamic fatigue plots and Weibull probability plots for the two-point bending strengths of four hybrid glass coated fibers under study are shown on Figures 1-4. Table 1 summarizes the strengths and fatigue parameters for the fibers. The HG-4 fiber with two layers of coating has a significantly higher fatigue parameter and strength measured at 5000 μm/s than the same coating applied in one layer *i.e.* this fiber is both stronger and less prone to stress corrosion than fiber with one layer of the same coat, even if the thickness of the overall coat is the same. This result seems to be a trend since the same was observed in previous on hybrid coating materials [1]. This is presumably the result of higher crosslinking density of the inorganic silica component that was exposed twice to the heat produced by the UV chamber.

Table 1. Description of the fibers used in this study including the diameters of the glass fiber, d_f, and the coating, d_c. The stress corrosion parameter, n, is calculated using linear regression and the range n_{low} to n_{high} represents a 95% confidence interval. The strength of the fibers is represented by the strength measured at a faceplate velocity of 5000 μm/s and includes the 95% confidence interval.

Fiber	Fiber Description	d_f μm	d_c μm	n	n_{low}	n_{high}	$\sigma_{5000\ \mu m/s}$ GPa
1	HG-4, 1 layer	200	250	21.2	19.7	22.9	6.79 ± 0.05
2	HG-4, 2 layers	200	250	26.2	23.9	29.0	7.48 ± 0.14
3	HG-4MP, 1 layer	125	159	30.7	24.5	41.3	5.10 ± 0.23
4	HG-4MP, 1 layer, additional heat cure applied on line	125	156	37.1	28.7	53.1	4.96 ± 0.21

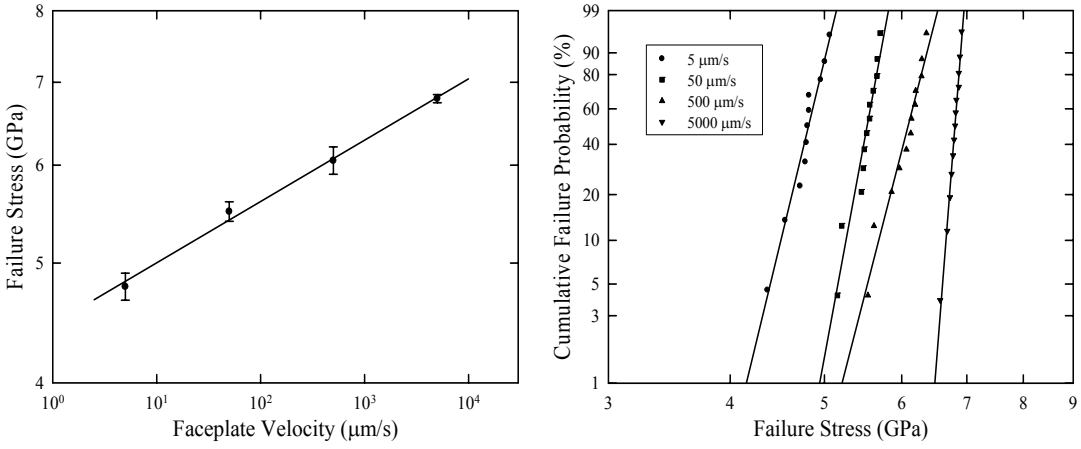

Fig. 1. Dynamic fatigue and Weibull plots of the bending strength for fiber with a single coat of HG-4

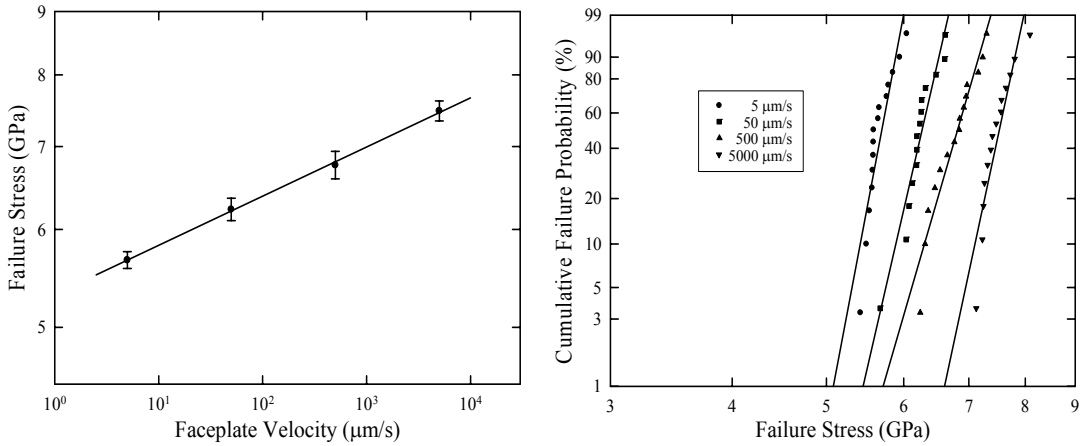

Fig. 2. Dynamic fatigue and Weibull plots of the bending strength of fiber with dual coats of HG-4

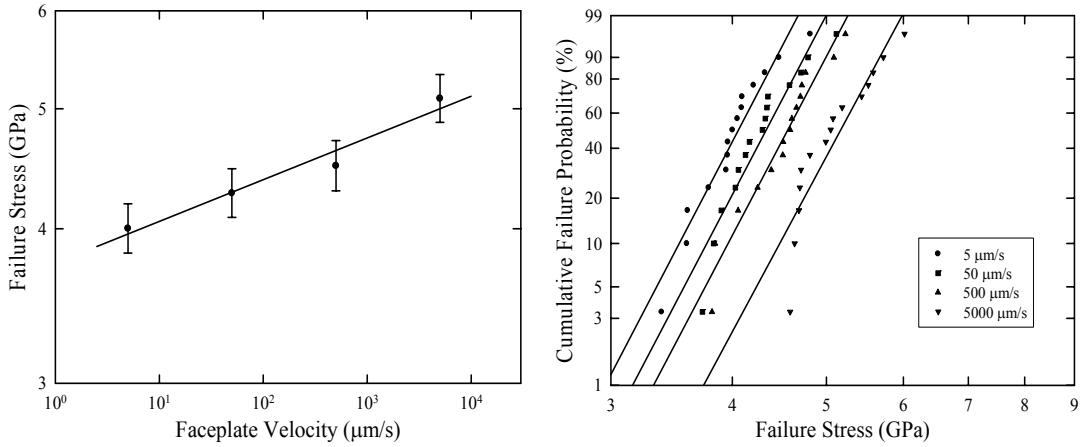

Fig. 3. Dynamic fatigue and Weibull plots of the bending strength of fiber with a single coat of HG-4 MP

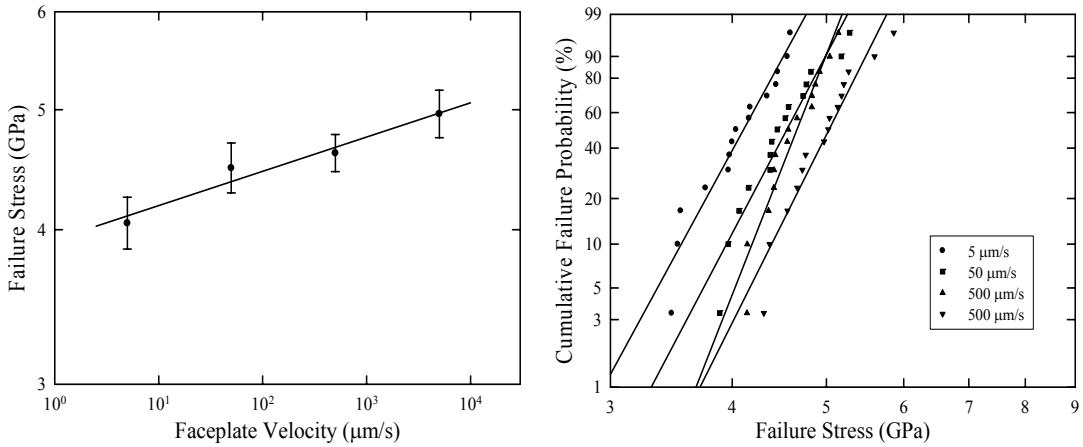

Fig. 4. Dynamic fatigue and Weibull plots of the bending strength for fiber with a single coat of HG-4MP that was heat cured online in addition to the UV cure.

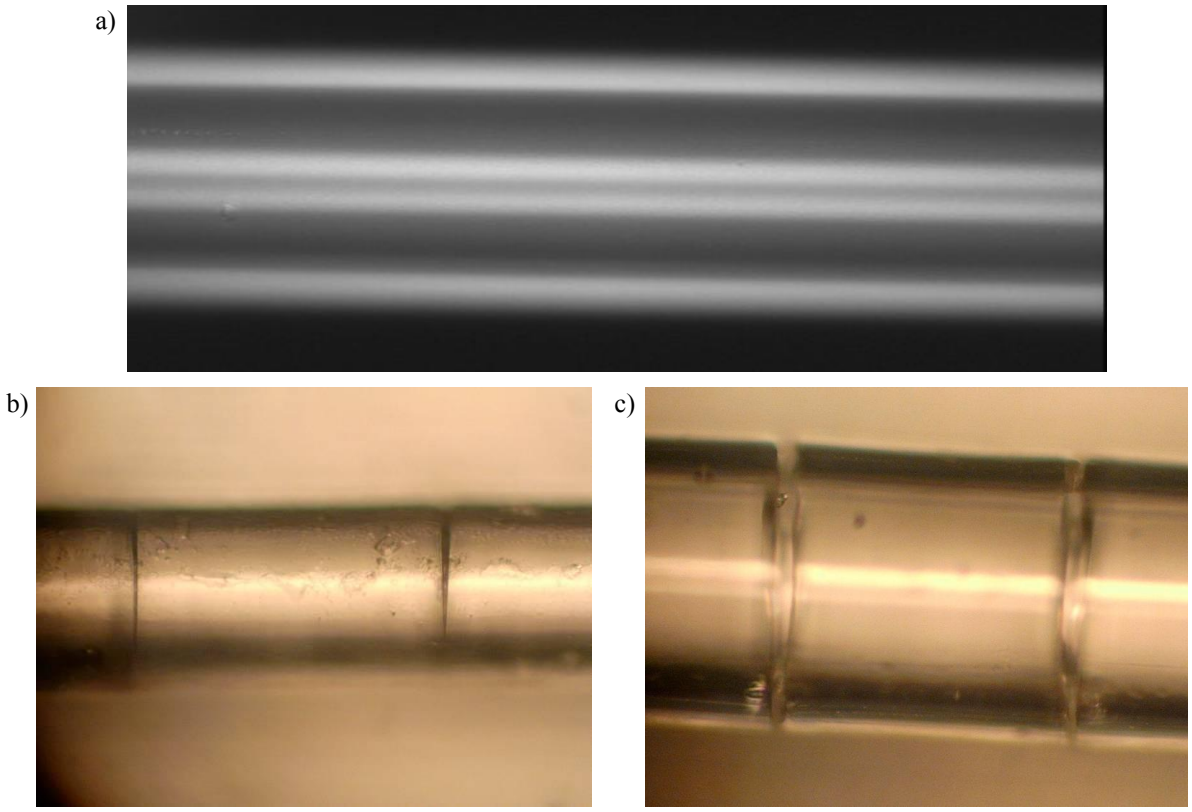

Fig. 5. Optical microscope images of (a) fiber 1 with single coat of HG-4, (b) fiber 3 with a single layer of HG-4MP, and (c) fiber 4 with dual coat of HG-4MP.

For HG-4MP coated fiber there is an increase in *n* value but a significant decrease in strength in comparison with fiber coated either with one or two layers of HG-4 (Table 1). This somewhat lower strength appears to coincide with more scatter in the results as evidenced by the larger confidence bands for both strength and *n* for fibers 3 and 4 compared to fibers 1 and 2. Examination of the HG-4MP fibers in the optical microscope revealed the presence of cracks in the coatings as shown in Figure 5.

The coating HG-4MP has the same chemistry as HG-4 except that it contains 20% fewer polymerizable acrylate groups. It was expected that the lower amount of the crosslinking groups will increase fiber strength and thermal stability. In fact, the lower amount of the acrylate groups induced stress in the solidifying network that caused its rupture when the whole inorganic-organic backbone went through relaxation process after curing. This phenomenon is most probably caused by different ratios of crosslinking of both the inorganic and organic coating components. It is interesting, however, to learn that the reduction of the polymerizable groups produced quite a significant increase in stress corrosion parameter *n*. This increase might be associated with higher crosslinking density and higher than in coating HG-4 amount of the inorganic component, *i.e.* silica. By careful selection of the acrylate to silica component ratio we can expect an increase in the *n* parameter accompanied by reasonably higher fiber strength values.

The results show that adding a second layer of the same coating increases both the fiber strength and stress corrosion parameter. We can say that the measured performance of the coatings offers the promise of improved fiber strength and reliability coupled with a reduced weight and volume.

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