# Endcapping of high-power 3 µm fiber lasers

YIGIT OZAN AYDIN, 1,2 D FRÉDÉRIC MAES, 1,2,\* VINCENT FORTIN, 1 Souleymane T. Bah, 1 Réal Vallée, 1 and Martin Bernier 1

**Abstract:** Fiber tip photodegradation through OH diffusion currently limits the long term operation of high-power fiber lasers and amplifiers operating near 3 µm. To address this issue, we investigate the resistance to OH diffusion of fluoride and oxide endcaps manufactured out of ZrF<sub>4</sub>, AlF<sub>3</sub>, GeO<sub>2</sub>, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> fibers. To this extent, the endcaps are spliced at the output of a 20 W continuous-wave fiber laser operating at 2.8  $\mu m$  and their degradation over a 100 h time period is monitored. While the fluoride-based endcaps underwent failure during the first 10 h, their oxide counterparts survived the experiment, although showcasing degradation which was reflected as an increase of the endface temperature over time. To overcome this issue, we propose a novel method to completely suppress OH diffusion which consists in sputtering a nanoscopic diffusion barrier film made of silicon nitride  $(Si_3N_4)$  on the output face of the endcap. The effectiveness of the approach is validated on Al<sub>2</sub>O<sub>3</sub>, ZrF<sub>4</sub> and AlF<sub>3</sub> endcaps which show no sign of degradation after being used for more than a 100 h at the output of a 3 µm high-power fiber laser.

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#### 1. Introduction

Fiber laser technologies are playing an instrumental role in the development of various applications [1]. However, as the output power of fiber laser systems increases, the likelihood of end-face damage increases accordingly. In the case of the well-known Yb<sup>3+</sup>:silica fiber lasers operating at 1 μm, such failure is related to the fact that their output intensity exceeds the air-glass surface damage threshold, and damage occurs either due to overheating in continuous-wave (CW) regime or laser-induced breakdown due to intense pulses [2]. To mitigate this issue, fiber-based endcaps, spliced at the output of the fiber laser systems, have been developed to allow the beam to expand in a controlled manner and therefore lower its intensity below the glass' damage threshold. Such endcaps have enabled the demonstration of fiber laser systems delivering over 100 kW of output power in CW operation [3].

Fluoride-based fiber lasers provide the means to achieve powerful laser emission between 2.8 and 4 µm [4–7], although their current output power is significantly less than their silica counterparts. Yet, we recently reported an erbium-doped zirconium fluoride fiber laser delivering 42 W of CW output power at 2.83 µm which highlighted the potential of 3 µm fiber lasers for further power-scaling up to the 100 W level [4]. Such all-fiber laser sources are coveted in the development of biological tissue ablation and remote-sensing applications given their excellent overlap with the OH bond's strong vibrational absorption band, their unrivalled beam-quality as well as their compact yet rugged design [8]. Nonetheless, the widespread deployment of high-power 3 µm-class all-fiber lasers is currently hindered by the short lifetime of such laser sources due to fiber tip degradation. Contrarily to silica fiber laser systems, this issue is the direct consequence of operating within the OH absorption band at 3 µm and the hygroscopic nature of fluoride-based glasses. Through analytical modeling, it was shown that the time elapsed before catastrophic failure of the fluoride fiber tip was inversely proportional to the square of the 3 µm output power [9]. As a result, for a 20 W power level, the all-fiber cavity reported in [4] lasted

 $<sup>^{</sup>l}$ Centre d'Optique, Photonique et Laser (COPL), Universite Laval, Québec, Québec G1V 0A6, Canada

<sup>&</sup>lt;sup>2</sup>These authors contributed equally to this work.

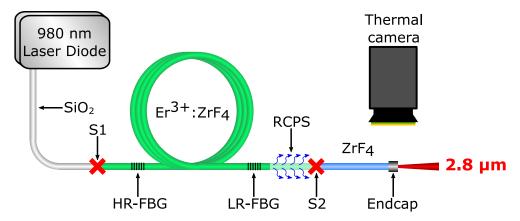
<sup>\*</sup>frederic.maes.1@ulaval.ca

less than 10 h before the fiber laser's fluoride-based endcap underwent catastrophic failure due to OH diffusion.

To address this issue, this work investigates the efficiency of different endcap materials to mitigate fiber tip degradation of 3  $\mu$ m-class high-power fluoride fiber lasers. To this extent, we monitored over a 100 h time period the degradation of endcaps made out of fluoride-based glass fibers (zirconium and aluminum fluoride), oxide-based glass fibers (fluorogermanate and silica) as well as single crystal sapphire fibers when spliced at the output of a high-power fiber laser operating near 3  $\mu$ m. Upon experimentation, fluoride-based endcaps underwent catastrophic failure after less than 10 h during the test, suggesting they should only be used in low-power (Watt - level) 3  $\mu$ m fiber laser systems. Although the oxide and crystal-based endcaps survived the 100 h long experiment, they showcased a significant rise of their temperature over time, hence indicating they should only be used in medium-power (20 W - level) fiber laser systems to ensure long term operation. Finally, this work proposes a definitive method to suppress OH diffusion within any type of endcap material by using an efficient OH diffusion barrier. This method, which consists in sputtering a thin-film of silicon nitride (Si<sub>3</sub>N<sub>4</sub>) on the output face of the endcap, was validated on ZrF<sub>4</sub>, AlF<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> endcaps which have shown no sign of degradation under high-power 3  $\mu$ m radiation over more than 100 h of experimentation.

# 2. Experimental setup

The home-made high-power 3  $\mu$ m-class fiber laser used to investigate the degradation of the different endcap materials is depicted in Fig. 1 and is similar to the system reported in [4]. Briefly, it is made out of a 6.5 m double-clad 7 mol % erbium-doped fluorozirconate (Er³+:ZrF4) fiber manufactured by *Le Verre Fluoré*. The 15  $\mu$ m diameter core of the fiber has a numerical aperture of 0.12 enabling singlemode operation above 2.4  $\mu$ m. The fiber laser cavity is bounded by two intracore fiber Bragg gratings (FBGs) written through the polymer of the fiber using femtosecond pulses with the scanning phase-mask technique [10,11]. The entrance high-refectivity (HR) FBG has a reflectivity > 99 % at 2.825  $\mu$ m while the output low-reflectivity (LR) FBG has a reflectivity of 8 %. The active fiber and the HR and LR-FBGs were spooled on a 32 cm diameter grooved and fan cooled aluminum spool, and secured with UV-cured polymer.



**Fig. 1.** Experimental setup used to monitor the degradation over time of the different endcaps when subjected to  $20\,W$  of output power at  $3\,\mu m$ .

In contrast to the system reported in [4], the system was solely pumped from the forward end by a 135 W commercial InGaAs 980 nm multimode laser diode whose silica delivery fiber was fusion-spliced (S1) to the Er<sup>3+</sup>:ZrF<sub>4</sub> fiber. At the output of the fiber laser cavity, a residual cladding pump stripper (RCPS) was fabricated by applying high-index UV-cured polymer on

the bare  $Er^{3+}$ : $ZrF_4$  fiber. This pumping scheme enabled an efficiency of 23 % with respect to the launched pump and a pump power limited maximum output power of around 29 W at 2.825  $\mu$ m. A singlemode fusion-splice (S2) was made between the output  $Er^{3+}$ : $ZrF_4$  fiber and a mode-matched passive  $ZrF_4$  relay fiber to carry out multiple endcap degradation experiments. The relay fiber has a 15  $\mu$ m core diameter, a numerical aperture of 0.12 and a 250  $\mu$ m cladding diameter. The high-power all-fiber laser cavity was operated at an output power of around 20 W for all degradation experiments.

The degradation over time of the various endcaps was monitored by measuring the temperature of the endcaps' output face with a thermal camera (Jenoptik, Variocam) equipped with a close-up lens. Simultaneously, the output power of the laser system was recorded with a thermopile detector (Gentec E-O, UP25N-250F-H12-D0) to ensure it operated at a 20 W output power level throughout the experiment. It should be noted that the laser cavity was operated at this power level with the same nominal performances for over 800 h during these experiments with a stability similar to the one showcased in [4], i.e. RMS fluctuations less than 0.1%.

### 3. Endcap splicing and manufacturing

In this investigation, we studied the degradation of 6 different endcap fiber materials: fluorozirconate ( $ZrF_4$  -  $BaF_2$  -  $LaF_2$  -  $AlF_3$  - NaF -  $SrF_2$  -  $HfF_4$ ), fluoroaluminate ( $AlF_3$  -  $AlCl_3$  - NaF -  $ZrF_4$  -  $YF_3$  -  $SrF_2$  -  $BaF_2$  -  $LaF_2$ ), fluorogermanate ( $GeO_2$  - ZnO - PbO -  $K_2O$  -  $PbF_2$ ), silica ( $SiO_2$ ) and sapphire single crystal ( $Al_2O_3$ ). All endcap materials were provided in fiber (or single crystal fiber) form and their specifications are presented in Table 1. The silica fiber was home-drawn using a *Heraus* preform composed of a F-300 pure silica core and a F-320 fluorine-doped silica cladding [15]. Manufacturing an endcap out of a 50 %-doped  $Er^{3+}$ : YAG single crystal ( $Y_3Al5O12$ ) fiber was also studied given the unavailability of an undoped YAG fiber. Moreover, in the remainder of this work, the endcap materials will be referred to by their main constituent, for example  $ZrF_4$  for fluorozirconate.

 $\alpha^{c} [\times 10^{-6} \text{K}^{-1}]$  $T_g^d$  [°C]  $\emptyset_c^f$  [µm]  $L^g$  [µm] Endcap Manufacturer ZrF<sub>4</sub> Le Verre Fluoré 1.49 17.2 265 200 480  $AlF_3$ Fiberlabs 18.6 390 200 450 1.46  $GeO_2$ Infrared Fiber Systems 1.83 10.9 420 230 380  $GeO_2$ Le Verre Fluoré 10.9 420 230 410 1.83  $SiO_2$ Heraeus F-300 1.42 0.55 1175 242 190 Er3+:YAG 1.79  $T_f = 1940^{e}$ 220 320 Shasta Crystals 6.14  $Al_2O_3$ Shasta Crystals 1.72 5 - 5.6  $T_f = 2030^{e}$ 240 N.A.

Table 1. Endcap Specifications $^a$ .

The endcaps were fusion-spliced to the passive ZrF<sub>4</sub> relay fiber using a Vytran GPX system equipped with an iridium filament (Vytran, FRAV4). For the ZrF<sub>4</sub> endcap, the filament was positioned at the splice point between the relay fiber and the endcap fiber. All other fibers were spliced to the ZrF<sub>4</sub> relay fiber by offsetting the longitudinal position of the filament in direction of the endcap fiber material as detailed in [16]. Once the fusion splice was achieved, the endcap fiber material was cleaved at a given length with a Vytran LDC cleaver. Images of the final

<sup>&</sup>lt;sup>a</sup> Optomechanical properties taken from [12–15].

<sup>&</sup>lt;sup>b</sup> Refractive index around 3 μm.

<sup>&</sup>lt;sup>c</sup> Thermal expansion coefficient.

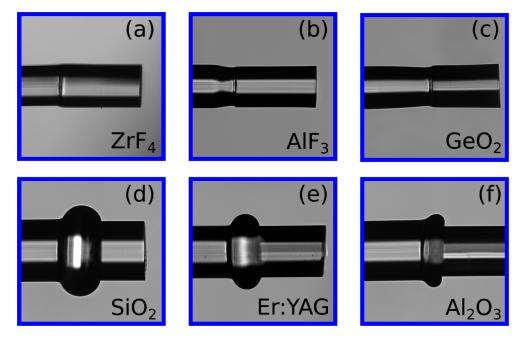
d Transition temperature.

<sup>&</sup>lt;sup>e</sup> Melting temperature.

f Core diameter.

g Length.

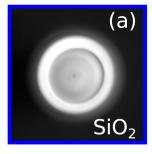
endcaps resulting from this manufacturing process are presented in Figs. 2(a)-2(c). Typical output power losses at  $2.83 \,\mu m$  after splicing the fluoride and  $GeO_2$  endcaps were around 4% and 8%, respectively, including fiber transmission losses and Fresnel reflections at the output endcap face and splice interface. Prior to the degradation tests, the assembly was secured using a low-index UV-cured polymer in a copper V-groove to ensure good heat conduction from the endface to the heat sink. Special care was taken to limit the length of the endcap protruding out of the copper V-groove. In contrast to natural convection, which was used in the past to study the degradation of  $ZrF_4$  fiber tips [9], thermal conduction was chosen in the current experiment given this cooling method is more efficient, thus limiting at maximum the endface temperature.

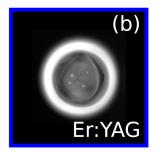


**Fig. 2.** Photographs of the different endcaps taken with the imaging system of the Vytran GPX.

In contrast to fluoride and GeO<sub>2</sub> fibers, the SiO<sub>2</sub>, Er<sup>3+</sup>:YAG and Al<sub>2</sub>O<sub>3</sub> fibers form no thermal bonds when spliced to the ZrF4 glass fiber. To overcome this limitation, we relied on the fact that the thermal expansion of ZrF4 glass is significantly larger than that of all the oxide-based fiber materials tested, as seen in Table 1. Hence, by pushing in a controlled manner the oxide-based fiber material into the ZrF<sub>4</sub> fiber, after the former has been heated sufficiently, a permanent and robust joint is created as seen in Figs. 2(d)–2(f). The joint's strength is provided by the ZrF<sub>4</sub> glass which tightly wraps around the oxide-based fiber material once the splice point cools down. In fact, all fusion-splices resulting from this splice procedure were proof-tested at a tension of 200 g (roughly 4.4 MPa) prior to being used in the tests. It should be noted that this splicing method was also used to achieve the splice (S1) between the silica delivery fiber of the InGaAs multimode laser pump diode and the active Er<sup>3+</sup>:ZrF<sub>4</sub> fiber shown in Fig. 1. A similar splicing method, relying on the same principle but using a CO2 fusion-splicer, was recently shown to enable robust single-mode splices between silica and ZrF<sub>4</sub> fibers [17]. This principle was most-likely also employed in a number of other reports where silica and ZrF<sub>4</sub> fibers, or silica and chalcogenide fibers, were fusion-spliced [18–22]. The typical output power losses at 2.83 µm after splicing the SiO<sub>2</sub>, Er<sup>3+</sup>:YAG and Al<sub>2</sub>O<sub>3</sub> endcaps were 8%, 10% and 16%, respectively.

In the case of the SiO<sub>2</sub> and Er<sup>3+</sup>:YAG endcaps, it was possible to cleave the fiber material after the splice process, as seen in Figs. 2(d)–2(e), and cool the assembly in the same manner as the fluoride and GeO<sub>2</sub>-based endcaps. It should be noted that the length of the SiO<sub>2</sub> endcap was shortened as much as possible due to the fiber's high absorption losses of  $\sim 25 \, \mathrm{dB}$  /m near  $2.825 \,\mu m$ . For the  $Er^{3+}$ :YAG endcap, cleaving was greatly simplified by the fact that that the crystalline planes of the Er<sup>3+</sup>:YAG fiber are perpendicular to its optical axis, as shown in [23]. A photograph of the splice interface between the ZrF<sub>4</sub> fiber and the SiO<sub>2</sub> and Er<sup>3+</sup>:YAG endcap is shown in Figs. 3(a)-3(b). From these images, it is clear that the interface between the  $SiO_2$ endcap and the ZrF<sub>4</sub> fiber is smooth and that it does not deteriorate the quality of the laser beam. For the Er<sup>3+</sup>:YAG endcap, one can see some bubbles at the interface which might degrade the beam-quality if they are located in the beam path. However, we believe optimization of the splice recipe can prevent the formation of such bubbles and enable flawless splice interfaces similar to that of the SiO<sub>2</sub> endcap. For the sapphire single crystal fiber, it was not possible to cleave or polish the fiber material without breaking the splice point. This stems from the fact that the crystalline planes are at 45° with respect to the optical axis of the fiber and also from it's high mechanical strength. Therefore, the whole length of the sapphire fiber (50 cm) was kept for the degradation test. Given the sapphire fiber is coreless, any attempts at cooling the fiber extremity using the copper assembly described above resulted in leakage of the 3 µm signal fom the side and the eventual failure of the assembly. Therefore, the sapphire fiber tip was tested under natural heat convection instead of heat conduction as for the other endcaps.





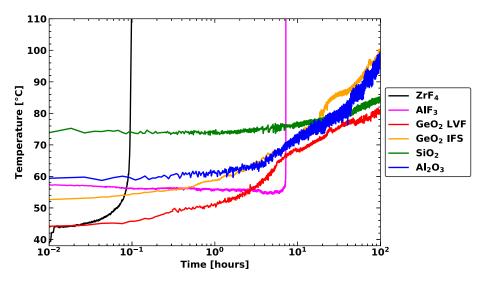
**Fig. 3.** Photographs of the interface between the  $ZrF_4$  relay fiber and (a) the  $SiO_2$  and (b) the  $Er^{3+}$ :YAG endcaps.

#### 4. Results and discussion

#### 4.1. Endcap degradation

Figure 4 showcases the degradation of the various endcaps under the action of 20 W CW output power at  $2.83 \,\mu m$  over a  $100 \,h$  time period. The initial temperature of the various endcaps varies between  $40 - 75 \,^{\circ}C$ , a variation accounted for by the difference of initial OH compound concentration, absorption coefficient at  $2.83 \,\mu m$ , thermal conductivity [9] as well as their refractive index which determines the intensity of the Fresnel reflection at the endcap interface. As reported in [4], the fluoride fiber-based endcaps did not survive the experiment more than  $10 \,h$ . While the initial temperature of the multimode  $ZrF_4$  fiber endcap was the lowest of all the endcaps that were tested ( $40 \,^{\circ}C$ ), it underwent catastrophic failure after only  $10 \,^{\circ}C$  minutes. The degradation curve of the  $ZrF_4$  endcap, as well as the time elapsed before failure, are in agreement with the results found in [9]. It should be noted that in the latter, the fiber tip experienced natural heat convection which increased the thermal resistance between the fiber and its surroundings, and resulted in an accelerated degradation rate. As for the  $AlF_3$  endcap, it survived for about  $10 \,h$  under similar conditions given its glass matrix is more than ten times more stable in water than that of  $ZrF_4$  [24].

It is therefore clear that AlF<sub>3</sub>, and even more so  $ZrF_4$ ,-based endcaps are not suitable long term solutions when dealing with output powers above a few watts around 3  $\mu$ m. Based on the different reports having used AlF<sub>3</sub> endcaps to protect fiber laser systems from photo-degradation [25,26], it can be stated that AlF<sub>3</sub> endcaps should be restricted to 3  $\mu$ m fiber laser systems delivering output powers of a few watts in order to guarantee their long term operation. On the other hand, multimode  $ZrF_4$  fiber endcaps should only be used in low-power systems where the output power is kept below the watt-level range. However, due to their perfectly matched refractive index,  $ZrF_4$  endcaps produce the lowest splice interface reflection among all the tested endcaps; a significant advantage for mode-locked and in-amplifier supercontinuum  $Er^{3+}$ : $ZrF_4$  fiber laser systems. In both cases, the performance of these systems is greatly limited by parasitic lasing at 2.8  $\mu$ m and it is therefore paramount to use an endcap producing the lowest possible feedback [27,28].



**Fig. 4.** Measured temperature of the endcaps' output face as a function time for a constant  $3 \mu m$  output power of 20 W.

From Fig. 4 it can be seen that all the oxide-based and crystalline endcaps that were tested survived the 100 h long degradation experiment. However, the experiment also revealed that their temperature increased over time, hence indicating the existence of some degradation phenomenon. Table 2 summarizes the degradation performance of the SiO<sub>2</sub> and GeO<sub>2</sub>-based endcaps. The initial temperature of the SiO<sub>2</sub> endcap (74 °C) is significantly higher than that of the GeO<sub>2</sub> endcaps ( $\approx 50$  °C), a direct result of the strong absorption of SiO<sub>2</sub> around 3  $\mu$ m. Given the constant ambient temperature during the experiment of 20 °C, the initial temperature rise of the SiO<sub>2</sub> endcap per watt of output power at 3 µm is found to be 2.7 °C/W while that of GeO<sub>2</sub> endcaps is roughly twice less, i.e. 1.4 °C/W. Hence, in the perspective of power-scaling the output power of 3 µm-class all-fiber lasers to 100 W, we can expect a SiO<sub>2</sub> endcap to reach an initial temperature of 290 °C while the temperature of a GeO<sub>2</sub> endcap would be between 140 - 218 °C. This allows us to conclude that GeO<sub>2</sub> endcaps are better candidates for high-power 3 µm systems since the splice between the SiO<sub>2</sub> endcaps and the ZrF<sub>4</sub> fiber cannot sustain temperatures in excess of the transition temperature of ZrF<sub>4</sub> (i.e. 270 °C [12]). Nonetheless, for medium-power systems (≈20 W), SiO<sub>2</sub> fibers could be considered a better alternative than GeO<sub>2</sub> fibers, given their degradation rate is more than three times slower than that of GeO<sub>2</sub>. This fact has enabled the  $SiO_2$  endcap to reach after 100 h a similar final temperature as the  $GeO_2$  (LVF) endcap although their initial temperature difference was 33 °C. Moreover, SiO<sub>2</sub> fibers, comparatively to GeO<sub>2</sub> fibers, are less expensive and significantly easier to handle and process. Additionally, the

refractive index of  $SiO_2$  around 3  $\mu$ m (1.42) is closer to the refractive index of  $ZrF_4$  glass (1.49) than that of  $GeO_2$  (1.83). This characteristic also favors  $SiO_2$  endcaps in the design of powerful MIR mode-lock or in-amplifier fiber lasers [27,28], as discussed earlier. It should be noted that both  $GeO_2$  endcaps that were tested behaved similarly during the experimentation, although the endcap manufactured out of the fiber provided by *Le Verre Fluoré* had a lower initial temperature. However, it is difficult to assess if this is the result of a less OH diffusion prone glass composition or a more efficient cooling.

Table 2. SiO<sub>2</sub> and GeO<sub>2</sub> Endcap Performances.

Endcap	$T_i \overset{a \circ}{\sim} C$	$\Delta T_i / \Delta P^{b \circ} C / W$	$T_{i,100W}$ $^{c\circ}$ C	$\Delta T/\Delta t^{d\circ}$ C
GeO <sub>2</sub> LVF	44	1.20	140	0.37
GeO <sub>2</sub> IFS	53	1.65	218	0.47
$SiO_2$	74	2.70	290	0.10

<sup>&</sup>lt;sup>a</sup> Initial temperature.

As stated earlier, the  $Al_2O_3$  fiber tip surprisingly showed signs of degradation over time under the influence of high power 3 µm laser light. The initial temperature of the  $Al_2O_3$  fiber tip was 60 °C and its final temperature is 97 °C, which results in a 0.37 °C/h degradation rate. While the initial temperature and degradation rate of the  $Al_2O_3$  fiber tip is comparable to those of  $GeO_2$  endcaps, it should be noted that the former experienced natural convection instead of heat conduction, a condition which accelerates degradation by easily a tenfold [9]. Therefore, we believe  $Al_2O_3$  endcaps are potentially an interesting solution for high-power 3 µm fiber laser systems, contingent upon the ability to manufacture endcaps out of single crystal  $Al_2O_3$  fibers. An alternative to manufacturing such endcaps would be to inscribe depressed cladding single-mode waveguides with femtosecond pulses in the  $Al_2O_3$  rod fiber, as shown recently in [29]. This method would preserve the beam-quality of the 3 µm fiber laser although long lengths of  $Al_2O_3$  fiber are used for beam delivery purposes.

Similarly to fluoride-based endcaps, we believe the degradation witnessed in the  $SiO_2$  and  $GeO_2$  endcaps is related to ambient water vapor diffusion within the glass matrix. In both cases, the OH vapor could be incorporated into the glass matrix in the form of GeOH or SiOH groups, as discussed in [30–33]. It is unlikely that water vapor diffused within the matrix of the single-crystal  $Al_2O_3$  fiber given the high degree of order of its crystalline matrix. Instead, we believe the temperature increase observed during the experiment is related to water vapor adsorption at the polished surface of the sapphire fiber [34]. The occurrence of water adsorption is caused by the fact that at the surface of the single-crystal sapphire fiber, the chemistry of a pure crystal does not hold given Al-O-Al compounds are deprived of neighboring compounds. This gives rise to various chemical mechanisms through which OH can bind itself to the surface and alter the latter's properties with increasing pressure, humidity, temperature and time. However, further investigations need to be conducted in order to validate how ambient OH vapor interacts with the different oxide-based and crystalline endcaps.

As for the  $Er^{3+}$ :YAG endcap, it could not be tested given its temperature at a 3 µm power level of 2.4 W was already around 120 °C. This temperature was measured at the splice point between the relay fiber and the  $Er^{3+}$ :YAG endcap. By analyzing with a near-infrared optical spectrum analyzer the output of the 2.83 µm all-fiber laser cavity, a clear, yet weak, peak at 980 nm was observed. This indicated that some residual cladding pump remained at the output of the laser cavity which was absorbed by the heavily-doped (50 %)  $Er^{3+}$ :YAG endcap and caused its excessive heating. In spite of this, we believe undoped YAG single crystal fibers are highly

 $<sup>^</sup>b$   $T_i$  variation with 3 µm output power.

<sup>&</sup>lt;sup>c</sup> Extrapolated T<sub>i</sub> at 100 W of output power.

<sup>&</sup>lt;sup>d</sup> Temperature variation over time.

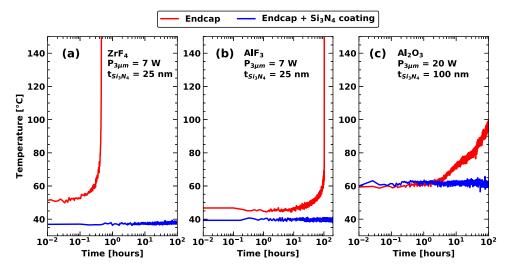
interesting endcap materials given they possess thermal and mechanical properties similar to  $Al_2O_3$  fibers while simultaneously offering the possibility to be easily processed into endcaps at the tip of  $ZrF_4$  fibers. It should also be noted that coreless calcium fluoride ( $CaF_2$ ) crystals have been recently used to endcap 3  $\mu$ m fiber laser systems operating at 16 W near 2.8  $\mu$ m [35]. However, given the unavailability of such material to the authors, it was not possible to evaluate it's long term degradation behaviour in the current experiment.

# 4.2. $Si_3N_4$ coatings for high-power 3 $\mu$ m fiber lasers

In order to inhibit in a definite manner OH diffusion within endcap materials, we propose to coat the output face of the endcap with a nanoscopic thin-film of silicon nitride  $(Si_3N_4)$ . Such materials are already extensively used in electronics as a diffusion barrier for  $SiO_2$  dielectric layers or passivation layers in flexible electroluminescent devices [36].

In this experiment, the  $Si_3N_4$  thin-films were deposited on the facet of the endcaps using reactive ion beam-assisted double magnetron sputtering as described in [37] under a  $1.46\times10^{-3}$  Torr environment. The target material was a 6" diameter 99.99 % pure silicon disk. The temperature of the substrate was kept at 115 °C and deposition of the thin-film was done at a 0.24 nm/s. The argon gas flow was maintained at 32 sccm during the sputtering process and the reactive gas nitrogen gaz (22 sccm) was introduced into chamber by the ion source. The uniformity of the deposited thin-film was enhanced by a rotating the substrate holder at 80 rpm.

Figure 5 compares the degradation of  $Si_3N_4$ -coated and uncoated  $ZrF_4$ ,  $AlF_3$  and  $Al_2O_3$  endcaps under the impact of 3 µm light over a 100 h long time period. For the  $ZrF_4$  and  $AlF_3$  endcaps, the  $Si_3N_4$  coating had a 25 nm thickness and the output power of the 3 µm fiber laser was 7 W, while in the case of the  $Al_2O_3$  endcaps they were 100 nm and 20 W, respectively. For both  $ZrF_4$  and  $AlF_3$ , the coating thickness was limited to less than 1% of the wavelength to limit the Fresnel reflection given the high refractive index ( $n \sim 1.95$ ) of  $Si_3N_4$ . Moreover, all endcaps experienced natural convection to accelerate the photodegradation process. From Fig. 5 it is clear that the  $Si_3N_4$  coating inhibits OH diffusion for all the tested endcaps given no increase of temperature over time is recorded. It also demonstrates that  $Si_3N_4$  coatings can be applied on a variety of fiber materials.



**Fig. 5.** Measured temperature of the (a)  $ZrF_4$ , (b)  $AlF_3$  and (c)  $Al_2O_3$  endcaps compared to their  $Si_3N_4$  coated counterparts as a function of time. The 3 µm output power and  $Si_3N_4$  coating thickness used for the  $ZrF_4$  and  $AlF_3$  endcaps were 7 W and 25 nm, respectively, while those for the  $Al_2O_3$  endcap was 20 W and 100 nm, respectively.

However, some  $Si_3N_4$  coatings showed signs of cracking a few months after their deposition upon the fibers as result of high surface stresses. Such stresses can nonetheless be relieved through the deposition of thinner  $Si_3N_4$  thin-films. Hence, future investigation will attempt to find a thickness range where  $Si_3N_4$  coatings do not crack over time while simultaneously preserving their OH impermeability. These investigations will also consider the introduction of oxygen within the  $Si_3N_4$  matrix which has been show to improve its flexibility [38]. Finally, the  $Si_3N_4$  coating optimization will also be facilitated by the number of potential endcap substrates upon which they can be deposited, i.e.  $ZrF_4$ ,  $AlF_3$ ,  $GeO_2$ ,  $SiO_2$ , YAG and  $Al_2O_3$ . We believe that an optimized endcap properly coated with  $Si_3N_4$  will enable the long term operation (> 10,000 h) of 100 W - level 3 µm-class fiber laser systems in the near future. It should be noted that silicon nitride or silicon oxynitride could also be used to coat the outer glass cladding of optical fibers. Such coatings could constitute a transparent alternative to thin carbon, metallic or ORMOCER coatings which have been shown to provide an excellent protection against OH diffusion within  $SiO_2$ -based fibers [39–41].

#### 5. Conclusion

In this work, the OH degradation of various endcaps spliced at the output of a 20 W all-fiber laser at 3  $\mu$ m was monitored over a 100 h time period. This investigation showed that fluoride, i.e. ZrF<sub>4</sub> and AlF<sub>3</sub>,-based endcaps lasted for less than 10 h before undergoing catastrophic failure, hence indicating they should only be used for low-power applications. On the other hand, the oxide-based endcaps (GeO<sub>2</sub> and SiO<sub>2</sub>) as well as the tip of a Al<sub>2</sub>O<sub>3</sub> fiber survived the experiment, which makes them interesting endcap solutions for medium-power systems ( $\sim$  20 W). To the best of our knowledge, this is the first report in which oxide-based materials are spliced and processed into endcaps at the output of a fluoride fiber laser. Nonetheless, all oxide materials showcased a clear temperature increase over time, an observation which is believed to stem from OH diffusion in the GeO<sub>2</sub> and SiO<sub>2</sub> glass matrix or presumably OH adsorption in the case of Al<sub>2</sub>O<sub>3</sub>.

In order to inhibit OH interaction with endcap materials under the irradiation of intense 3  $\mu$ m light, we also propose in this work to coat the output face of endcaps with a silicon nitride (Si<sub>3</sub>N<sub>4</sub>) thin-film. The effectiveness of the proposed method is demonstrated on Si<sub>3</sub>N<sub>4</sub> coated ZrF<sub>4</sub> and AlF<sub>3</sub> endcaps, as well as on a Al<sub>2</sub>O<sub>3</sub> fiber tip. Upon illumination with 3  $\mu$ m light for a 100 h, the coated endcaps and fiber tip showed no sign of degradation, whereas their uncoated counterparts either underwent catastrophic failure (ZrF<sub>4</sub> and AlF<sub>3</sub>) or showed a significant temperature rise (Al<sub>2</sub>O<sub>3</sub>). We believe optimized Si<sub>3</sub>N<sub>4</sub>-coated endcaps will allow the long-term operation of 100 W-level 3  $\mu$ m class all-fiber lasers in the near future, and spark the development of cutting-edge mid-infrared applications.

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