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Low loss polycarbonate polymer optical fiber for high temperature FBG humidity sensing

Getinet Woyessa, Andrea Fasano, Christos Markos, Henrik K. Rasmussen, and Ole Bang

***Abstract*— We report the fabrication and characterization of a polycarbonate (PC) microstructured polymer optical fiber (mPOF) Bragg grating (FBG) humidity sensor that can operate beyond 100ºC. The PC preform, from which the fiber was drawn, was produced using an improved casting approach to reduce the attenuation of the fiber. The fiber loss was found reduced by a factor of two compared to the latest reported PC mPOF [20], holding the low loss record in PC based fibers. PC mPOFBG was characterized to humidity and temperature, and a relative humidity (RH) sensitivity of 7.31±0.13 pm/% RH in the range 10-90% RH at 100ºC and a temperature sensitivity of 25.86±0.63 pm/ºC in the range 20-100 ºC at 90% RH were measured.**

***Index Terms*— Annealing, Fiber gratings, Humidity measurement, Optical fiber sensors, Plastic optical fiber, Temperature measurement.**

I. Introduction

THE interest in polymer optical fiber (POF) sensors is steadily increasing because of their low processing temperature, high flexibility in bending, high fracture toughness, ease of handling, and non-brittle nature, which are properties that glass fibers do not have [1]. In addition, POFs have a high elastic strain limit with low Young's modulus and they are biocompatible, which makes them advantageous for a range of strain and bio-sensing applications [2-11]. Some polymers, such as PMMA, are humidity sensitive and strongly absorb water [12-15], while others, such as Topas and Zeonex, have been reported to be insensitive to humidity [16-19]. Therefore, one of the key characteristics of PMMA based POFs is their ability to highly absorb moisture. The moisture absorption leads to a change in the refractive index and size of the fiber, which consequently change the Bragg wavelength [12]. Therefore, PMMA based fiber Bragg gratings (FBGs) are considered as potential candidates for developing humidity sensors [12-13].

The temperature and relative humidity (RH) operational limits and stability of polymer optical fiber Bragg gratings (POFBGs) strongly dependent on two parameters: the glass transition temperature (T_g) and moisture absorbing capability of the fiber material. The temperature operational limit of POFBGs produced from humidity insensitive polymers, such as Topas and Zeonex, relies exclusively upon their T_g and thus they can reliably operate 15-20 ºC below their T_g regardless of the surrounding RH level [16-19]. However, this is not the case for other polymers such as PMMA which have high affinity to water. The temperature operational limit of PMMA POFBGs is strongly dependent on the surrounding RH level and vice versa. At ambient or lower RH level, they can operate 15-20 ºC below their T_g. It has been reported that PMMA POFBGs can operate up to 90 ºC at ambient RH [20]. However, when the surrounding RH is higher than the ambient RH, the temperature operational limit decreases significantly. For instance, when PMMA POFBGs humidity sensors are operated up to 90% RH, the maximum operational temperature is 75 ºC [15]. This is attributed to the fact that glass transition temperature of PMMA decreases with increasing humidity [21]. So far, characterization for temperature measurement of POFBGs has been carried out using a hot plate or a conventional oven with no control on relative humidity [20, 22]. It is known that as the temperature of the hot plate or oven increases the corresponding surrounding relative humidity decreases dramatically. To verify this behavior we performed a systematic investigation using an environmental controlled chamber. The chamber was first programmed to have a fixed RH of 90% and ambient temperature. Releasing the RH of the chamber and by increasing the temperature up to 80 ºC, it can be clearly seen from Fig. 1 how the RH of the chamber significantly and rapidly decreases and reaching an equilibrium at 1% RH in less than 3 hours. Based on this response, we can conclude that similar behavior occurs in a humidity uncontrolled environment such as for example in an open space hot element or oven. At 1% RH, T_g of PMMA is expected to be higher than the one at ambient relative humidity as the water uptake capability will be lower and water acts as a plasticizer for PMMA [21]. Therefore, humidity is a limiting factor in the maximum operating temperature of POFBGs which have strong affinity to water. Similarly, when PMMA POFBGs are used as humidity sensors, the range of operation is highly dependent on the environmental temperature. PMMA mPOFBGs have been operated in the range 10-90% RH at

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maximum limiting temperature of 75 ºC with no hysteresis [15]. At 90% RH and temperature beyond 80ºC, the grating is experiencing a significant degradation and it is unable to operate. Therefore, PMMA based POFBGs can operate beyond 75 ºC provided the corresponding RH level is lower than 90%.

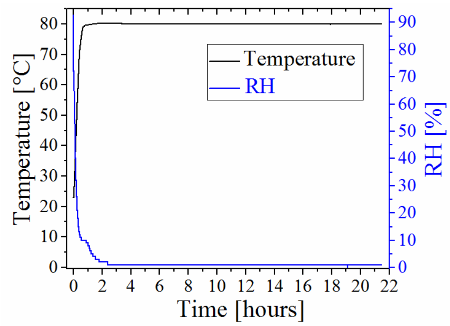

Fig.1 The humidity response of an oven as the temperature is increasing.

Thus, it is extremely important for different applications to develop POFBG humidity sensors which can fulfill and operate at both high temperatures beyond the operational limit of PMMA POFBGs, and also wide range of relative humidity operation. In this work, we demonstrate for the first time a record low loss mPOFBGs humidity sensor that can operate beyond 90% RH and 100 ºC using PC as the fiber material. Recently, it has been demonstrated that PC mPOFBG can operate up to 125 ºC at ambient relative humidity [23]. The glass transition temperature and water absorption (saturation value) at 23ºC for different polymer used for the fabrication of mPOFs is listed in Table I.

made it easier for the water trapped inside the plastic pellets to diffuse out during the drying, thereby improving the overall quality of the casting process. The melting phase was extended to remove any possible micro-sized residuals of crystals left in the final preform. Indeed, incomplete melting due to insufficient melting time might lead to the presence of micro-crystals in the core of the cast preform, which would result in higher scattering loss. This can be seen in the visible region in Fig. 2(b), where the gap between old and new fiber transmission losses increases with decreasing wavelength.

TABLE I
POLYMERS GLASS TRANSITION TEMPERATURE AND WATER ABSORPTION (SATURATION VALUE) AT 23°C

Polymer types	Glass transition temperature	Water absorption (saturation value) at 23ºC
PMMA [24]	106ºC	2.1%
PC [25]	145ºC	0.3%
TOPAS5013S-04 [26]	134ºC	<0.01%
Zeonex 480R [27]	138ºC	<0.01%

The bulk material loss of the PC polymer and the PC mPOFs losses are shown in Fig. 2(a) and 2(b), respectively. The minimum fiber propagation loss was found to be ~ 4.06 dB /m at 819 nm and at this wavelength the material loss is ~ 3.58 dB/m. It should be emphasized that the fiber loss was reduced by a factor of two compared to the first fabricated PC mPOF [23], holding thus the record in PC POFs loss. Both the cane and the fiber have been drawn at 170°C and 10.5 MPa drawing stress. The final core and cladding diameter of the fabricated fiber are 10 μm and 125 μm, respectively. The average size of the holes diameter and the pitch size are 2.5

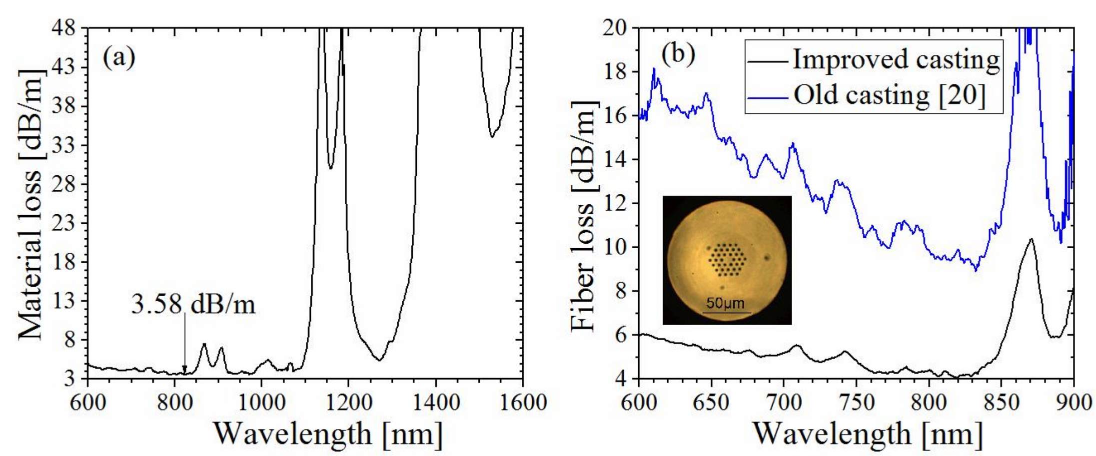

Fig. 2 (a) Bulk material optical loss for the improved PC solid rod. (b) Measured transmission loss of PC mPOF from both old and improved casting methods. Inset: Optical microscope image of the fabricated PC mPOF.

μm and 6.25 μm, respectively. The hole to pitch ratio is 0.4 ensuring that the fiber is endlessly single mode [28]. A microscope image of the PC mPOF end facet, which was cleaved with a custom made cleaver at a temperature of 80°C of both blade and fiber [29], is shown as an inset in Fig. 2(b).

II. EXPERIMENTS AND RESULTS

The microstructured fiber used in this report was fabricated in-house at DTU Fotonik using a drill and draw method described in [23]. However, to reduce the loss of the fiber we used an improved casting approach for the preform production. One of the most crucial factors which define the final fiber loss is perhaps the quality of the cast preform. The casting procedure was optimized with regard to two aspects: drying phase and melting phase. A better water removal was achieved by using an oven with enhanced air circulation. This

A fiber Bragg grating was first inscribed in the fabricated PC mPOF. The phase mask writing technique was used for the FBG inscription while the detailed experimental setup can be found in [30]. The phase mask used for grating inscription has a uniform period of 572.4 nm and the writing laser was a 325 nm HeCd CW UV laser. We used only 5 mW for the inscription of the grating. The grating had a length of 2 mm and the Bragg wavelength was located at 892.24 nm with reflection strength of 30 dB and a full width half maximum of 0.92 nm. The PC mPOFBG was then annealed at 125 ºC for 36 hours in a conventional oven without humidity control. The new Bragg wavelength after annealing was blue shifted to 880.19 nm. To be sure that the PC mPOFBG was properly annealed for humidity sensing operation up to 90% RH and at high temperatures, we further annealed it in the climate chamber at 90 % RH and 100 ºC for 6 hours, as high humidity has been shown to strongly assist the annealing of PMMA POFBGs [15]. For this, the PC mPOFBG was connectorized [31], and placed in a climate chamber (CLIMACELL, MMM Group). A supercontinuum source (SuperK Compact, NKT Photonics) has been used as the broadband light source and a spectrometer (CCS175 - Compact Spectrometer, Thorlabs) has been used to continuously track and record the grating during annealing in the climate chamber. This additional annealing did not lead to any further permanent blue shift, indicating that the POFBG was indeed properly annealed for operation at temperatures and relative humidity levels of 100 ºC and 90% RH, respectively. The normalized Bragg reflection spectrum of the PC mPOFBG at 90 % RH and 100°C is shown as an inset in Fig. 3(b).

After the annealing process, the humidity response of the PC mPOFBG sensor was measured at three different temperatures: 25 ºC, 50 ºC and 100 ºC, in the interval of 10-90 % RH. For each temperature level, the humidity measurement has been done first by increasing the RH from 10% to 90%, with step of 10% and then decreasing it from 90% to 10% with 10% step. For both cases, the chamber was programmed to change the RH in a minute and then to maintain stable the environmental conditions for 45 mins. The response of the PC mPOFBG, for both increasing and decreasing relative humidity at 100ºC, is shown in Fig. 3(a). Fig. 3(b) shows the humidity response at 100ºC where each measurement point was taken at the end of the 45 mins stabilization period. The humidity sensitivity at 100ºC was 7.31±0.13 pm/% RH (R-squared of 0.998), for both increasing and decreasing relative humidity. The corresponding humidity sensitivities at 25 ºC and 50 ºC were measured to be 7.35±0.05 pm/% RH and 7.19±0.11 pm/% RH, respectively. These sensitivity figures confirm that the humidity response of PC mPOFBG is unaffected by temperature, and this is due to the fact that the grating was adequately annealed.

We have also measured the temperature sensitivity at two different RH levels: 50 % and 90 % RH in the range from 20 to 100 ºC. For each RH level, the temperature measurement was first performed from 20 ºC up to 100 ºC, with a 10 ºC step and then decreasing it back to 20 ºC with the same step. For both cases, the chamber was programmed to change the temperature in 10 mins and then to maintain the environmental conditions stable for 45 mins. The response of the PC mPOFBG for both increasing and decreasing temperature at 90% RH is shown in Fig. 4(a). Fig. 4(b) shows the temperature response at 90% RH where each measurement

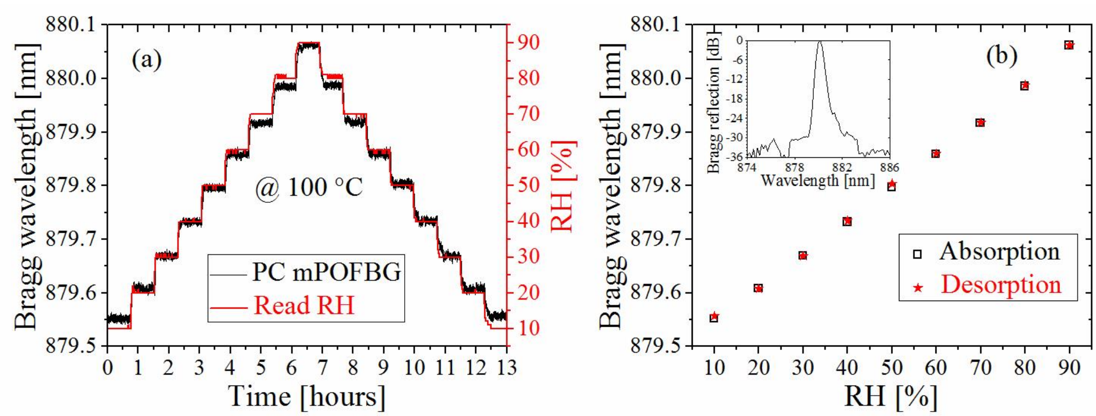

Fig. 3. (a) Measured humidity response at 100 ºC of the PC mPOFBG. (b) Corresponding stabilized humidity response of the PC mPOFBGs at 100 ºC. Inset: Normalized Bragg reflection spectrum of the PC mPOFBG.

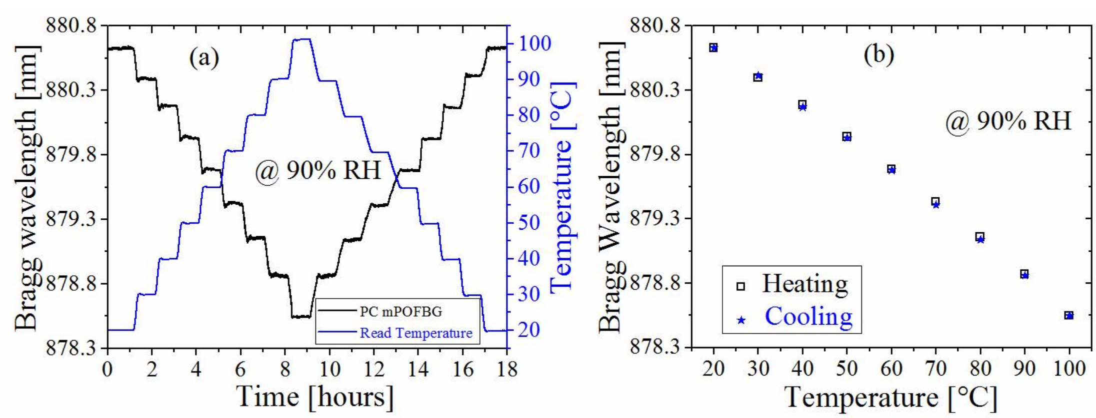

Fig. 4. (a) Measured temperature response at 90 % RH of the PC mPOFBG. (b) Corresponding stabilized temperature response of the PC mPOFBGs at 90 % RH.

point was taken at the end of the 45 mins for the stabilization period. The temperature sensitivity at 90 % RH was 25.86±0.63 pm/ºC. The corresponding sensitivity at 50% RH was 25.62±0.56 pm/ºC. No hysteresis was also observed during the temperature characterization, further confirming the fact that the grating was properly annealed.

III. Conclusion

We have developed and characterized a polycarbonate based mPOFBG humidity sensor that can operate beyond 100 ºC in the relative humidity range 10-90%. The mPOF preform was made by using an improved casting method and the measured loss was found to be two times smaller than the hitherto. The sensor gave a RH sensitivity of 7.31±0.13 pm/% RH in the range 10-90% RH at 100 ºC and a temperature sensitivity of 25.86±0.63 pm/ºC in the range 20-100 ºC at 90 % RH.

The humidity sensitivities of our PMMA mPOFBGs and TOPAS step index POFBGs at 850nm are 45 pm/%RH and 0.45 pm/%RH, respectively. Thus the humidity sensitivity of PC mPOFBGs is 6 times smaller than PMMA mPOFBG and 16 times larger than TOPAS POFBGs which are basically humidity insensitive. However, PC mPOFBG humidity sensors can operate up to 90 % RH at a temperature 25 ºC higher than the maximum operational limit of PMMA mPOFBGs. The temperature sensitivity of PC mPOFBGs is more than a factor of two larger than that of PMMA mPOFBGs. At ambient relative humidity PC mPOFBGs can operate up to 125 ºC while PMMA mPOFBG can only be operated up to 90 ºC. Thus, PC mPOFBGs humidity sensors can be used in several different applications areas where humidity measurement at high temperature is required such as in industry for ceramic driers, in domestic electric appliance for microwave oven and in agriculture for thermo-hygrostatic chamber [32].

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