Relative Humidity Sensing by Polymer CYTOP/XYLEX FBGs: Gamma Radiation Tuning of Sensing Properties

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Abstract: We demonstrate RH sensitivity tuning for polymer CYTOP/XYLEX FBGs by gamma irradiation. We present the dependency of RH sensitivity versus the irradiation dose, and confirm RH sensing with no temperature cross-sensitivity. © 2023 The Author(s)

1. Introduction

Polymer optical fibers (POFs) are known to absorb humidity, and therefore, they are potentially suitable to use for relative humidity (RH) sensing [1]. Indeed, various works were dedicated to the investigation of the RH sensitivity of fiber Bragg gratings (FBGs) inscribed in POF of various types [2-5]. The main problem of RH sensors based on polymer FBGs is temperature cross-sensitivity. Recently, FBGs inscribed in POF with CYTOP[®] core/cladding structure and XYLEX[®] overclad demonstrated the ability for temperature and RH sensitivity tuning (in particular, decreasing temperature sensitivity down to zero) by applying a pre-strain or by gamma radiation pre-treatment [6,7].

In our recent research, we demonstrated that CYTOP/XYLEX FBGs changed their response to climatic conditions as a result of their exposure to gamma irradiation [7,8]. We noted that the sensitivity to RH increased with received gamma irradiation dose, whereas the temperature sensitivity decreased to zero, with subsequent change of sign from positive to negative. Therefore, applying a particular, optimal gamma radiation dose (\approx 200 kGy), resulted in the temperature sensitivity to be minimized (1.77 pm/°C for the case of 200 kGy dose) [8]. It is apparent that this particular case is advantageous for RH sensing due to minimized temperature cross-sensitivity.

In this work, we consider the CYTOP/XYLEX FBGs for RH sensing. We refine the dependency of RH sensitivity on the received dose applying a wide set of gamma radiation doses (80, 120, 160, 200, 220, 240, 280 and 520 kGy). Then, for the case of 200 kGy, we calibrated the FBG for the measurement of RH and performed temperature cycles at low and high RH values confirming the ability to measure RH with negligible temperature cross-sensitivity.

2. Experimental setup

The few-moded POF used in this work contained a CYTOP[®] core/cladding structure and an overclad made of XYLEX[®] material (blend of polycarbonate and an amorphous polyester). The core had a diameter of 20 μ m, a gradient refractive index profile and an effective refractive index of 1.34. The diameter of the protective overclad was 250 μ m. The FBGs were inscribed using a femtosecond laser (HighQ laser femtoREGEN source, λ =517 nm, 220 femtoseconds pulse duration, and 1 kHz repetition rate) using the plane-by-plane method [9]. The fiber samples with FBGs were UV-glue connected with standard silica SMF pigtails. Fig. 1 presents a typical microscope image of the FBG (Zeiss Axio Imager M2 microscope) and the FBG's reflection spectrum.

We used a calibrated γ -radiation ⁶⁰Co sources for FBGs irradiation. The sources were located at a depth of seven meters in a water pool and provided a dose rate of 5.3 kGy/h (Brigitte facility, SCK-CEN). Irradiation was conducted at a temperature of 41-44 °C. The temperature was stabilized using an oven located within a hermetic container and controlled by a Eurotherm 2408 temperature controller.

Experiments were conducted using a climatic chamber Weiss SB22/300. Temperature and RH were measured by the internal sensors of the chamber and by the Thorlabs TSP01 sensor for additional control. Prior to the main experiments, we performed a temperature annealing for three hours at 65°C and 40 %RH to enhance the stability of the results and reduce possible hysteresis. To monitor the FBG reflection spectra and trace the Bragg wavelength, we used a standard commercial interrogator FiberSensing FS2200 operating in the 1500-1600 nm wavelength range.



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Fig. 1 (a) Microscope image of the FBG and (b) the typical FBG reflection spectrum [8].

3. Experimental Results

To obtain the dependency of RH sensitivity on the received dose, we irradiated FBG samples to a set of doses: 80, 120, 160, 200, 220, 240, 280 and 520 kGy. Then, we performed several RH cycles at a constant temperature of 30°C in the climatic chamber. We programmed the RH to increase stepwise from 40% to almost 100% with a step of 20% and then to decrease stepwise to the initial value of 40% [7,8]. The duration of each step was four hours. Using a linear approximation of the Bragg wavelength (BW) shift dependency on the RH, we calculated the sensitivity to RH for increasing and decreasing parts and then averaged the two values. The dependency of the RH sensitivity versus the irradiation dose and the table of corresponding values are presented in Fig. 2. It is seen that the dependency can be considered linear up to the dose of 280 kGy. The deviations of the values can be caused by uncertainties of the obtained doses (the irradiation setup was calibrated by the Harwell Red4034 dosimeters with 5% uncertainty) and by the non-ideal performance of the climatic chamber (temperature or RH could deviate from the target values; the RH was slightly modulated at some values). A dose of 520 kGy demonstrates the saturation of the RH sensitivity. It should be mentioned that with this dose we observed some instabilities in the BW evolution during the RH experiments: at some moments, the BW quickly increased by several hundreds of picometers. The reason for this effect can be the strong RH sensitivity of the 520-kGy FBG that resulted in a total BW change of more than three nanometers. This could cause tension and subsequent sliding of the core/cladding structure inside the overclad. Thus, having a higher number of irradiation doses, the graph in Fig. 2 demonstrates a wider range of linear dependency compared to the results in our previous work [7]. Nevertheless, further increase of the dose leads to the saturation of the dependency between 280 and 520 kGy.

60 1	×	Dose (kGy)	Sensitivity
₩ 50	×,/		(pm/%RH)
ud) ∕0	×,×	0 (pristine)	13.3
.≩ ⁴⁰		80	21.2
sitiv	, ix	120	26.2
ens	, x	160	32.9
ω Τ 20		200	41.3
፼ 20		220	40.7
10		240	47
10	0 100 200 300 400 500	280	52.3
	Dose (kGy)	520	56.8

Fig. 2 Dependency of the RH sensitivity of the FBGs on the received gamma radiation dose and the table with the corresponding values.

We demonstrated in our previous work that the dose of 200 kGy is close to the ideal for RH sensing due to its close-to-zero temperature sensitivity (1.77 pm/°C) and respectively high RH sensitivity (41.3 pm/%RH). To confirm the RH sensing ability of the FBG that received the 200-kGy dose, we calibrated the FBG to measure the RH level and we performed temperature characterization at two RH values. Fig. 3 shows the RH measured by the FBG during one full temperature cycle at 40% RH and a half temperature cycle at 85% RH. It is seen that during the first temperature cycle, the RH measured by a calibrated FBG accurately reproduces the RH measured by the reference RH sensor (Thorlabs TSP01), in the full range of applied temperatures (25-50°C). This confirms the absence of temperature cross-sensitivity of the FBG. It is also seen that the RH is fluctuated more in higher temperatures, which is an operational characteristic of the climatic chamber. The FBG, however, does not respond to this fluctuation

because of a reduced time response to RH changes. In particular, we observed that when the RH was high (the second temperature cycle in Fig. 3): it took more than 10 hours for the FBGs to indicate the target RH value.



Fig. 3 The first calibration test of the temperature insensitive FBG (200 kGy irradiation dose): the full temperature cycle at 40% RH and a half of the temperature cycle at 85% RH (the second cycle was stopped because of the slow stabilization of the FBG).

To investigate the FBG at high RH values and considering the necessity of a long stabilization time from the previous test, we performed the experiment presented in Fig. 4. The RH stabilized at 90%, quickly dropped towards 40% and was kept there for 7 hours. Then, the RH was quickly increased back to 90% and kept at this value for 20 hours during the FBG stabilization. Finally, the temperature cycle was performed starting from 25°C, increasing to 50°C, and decreased to 15°C to test a wider temperature range.



Fig. 4 The second calibration test of the temperature insensitive FBG (200 kGy irradiation dose): temperature cycle at high RH after RH rise and 20-hours FBG stabilization.

We observe that the RH measured by the calibrated FBG accurately matches the values measured by the reference sensor, except for the stabilization time resulting from the RH increase (40% to 90%). It should be mentioned that the FBG stabilized significantly faster after the RH drop at the beginning of the experiment: the stabilization took approximately 3.5 hours whereas the stabilization after the RH rise took the entire period of stabilization (20 hours). We note that we observed the stabilization time of less than one hour after the 20% RH changes in our earlier work [7]. Thus, we surmise that high RH values (close to 100%) require longer stabilization time to saturate FBGs, as expected. During the temperature cycle, the RH measured by the calibrated FBG was well matched with the RH measured by the reference sensor; the maximum difference was < 2% RH. The error can be related to the very small but not zero temperature sensitivity of the FBG and the uncertainties of the experiment. Thus, the experiment presented in Fig. 4 confirms the absence of the temperature cross-sensitivity of the irradiated FBG at high RH values and the ability to measure RH in various fluctuating temperatures.

4. Conclusion

In this work, we considered polymer CYTOP/XYLEX FBGs for RH sensing. We presented the refined dependency of RH sensitivity on the received gamma radiation dose. For the FBG that received the 200-kGy dose, which provides close-to-zero temperature sensitivity, we performed temperature cycles at two different RH values. The graphs of the Bragg wavelength calibrated for the RH measurement confirmed the capability of RH sensing independently of temperature variations. We also observed that high RH values required longer stabilization times for the FBG, as expected.

5. Acknowledgements

Fonds De La Recherche Scientifique – FNRS (T.0163.19 "RADPOF"). The research leading to these results has received funding from the Horizon 2020 programme of the European Union (Marie Skłodowska-Curie Actions - Individual Fellowships) under REA grant agreement No. 844618 (project POSPORI). This work is also funded by the research project EXCELLENCE/0918/0324 (T-Sense Project) by the Republic of Cyprus through the Research and Innovation Foundation and European Development Fund and the Cyprus University of Technology.

6. References

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