Annealing and etching effects on strain and stress sensitivity of polymer optical fibre Bragg grating sensors

A. Pospori*[1], C. A. F. Marques[1], D. Sáez-Rodríguez[2], K. Nielsen[3], O. Bang[3], D. J. Webb[1*]

1 Aston Institute of Photonic Technologies, Aston University, Aston Triangle, Birmingham, B7 4ET, United Kingdom

2 Optical and Quantum Communications Group, ITEAM Research Institute, Universitat Politecnica de Valencia, 46022 Valencia, Spain

3 DTU Fotonik, Department of Photonics Engineering, DK-2800 Kgs. Lyngby, Denmark

*Corresponding author: a.posporis@aston.ac.uk; orcid.org/0000-0003-4866-1361

Abstract: Thermal annealing and chemical etching effects on the strain and stress sensitivity of polymer optical fibre based sensors are investigated. Bragg grating sensors have been photo-inscribed in PMMA optical fibre and their strain and stress sensitivity has been characterised before and after any annealing or etching process. The annealing and etching processes have been tried in different sequence in order to investigate their impact on the sensor's performance. Results show with high confidence that fibre annealing can improve both strain and stress sensitivities. The fibre etching can also provide stress sensitivity enhancement, however the strain sensitivity changes seems to be random.

1. Introduction

Bragg grating sensors recorded in polymer optical fibre (POF) offer some interesting and potentially useful differences compared to gratings in silica fibre [1]. Amongst other things they provide an enhanced sensitivity to force and pressure [2], due to the lower elastic modulus of polymers compared to glass [3], and for certain polymeric fibre materials a sensitivity to the water activity of the environment surrounding the fibre [4]. One of the drawbacks of POF is its viscoelastic nature. The stress and strain in viscoelastic materials are not in phase, as a result hysteresis and creep effects are introduced when cyclic loading is applied to the fibre [5]. POF thermal annealing has been shown to reduce these effects [6]. Thermal annealing of POF was first used for FBG sensor multiplexing purposes, due to the ability of POF to shrink in length when is exposed above the β-transition temperature, which in turn induces a permanent negative Bragg wavelength shift [7]. Then, it was demonstrated that annealing can provide additional benefits, such as strain [8] and humidity [9] sensitivity enhancement and longer operational range in temperature monitoring [10]. Chemically etching POF to reduce the fibre diameter was also demonstrated in order to improve the force and pressure sensitivity [11] as well as the response time to humidity changes [12]. In these cases, benefit is obtained from changing the physical dimensions of the fibre in order to either decrease the cross sectional area where the force is applied or reduce the distance between the external environment and the Bragg grating's location in the fibre core. However, recently it was demonstrated that chemical etching can change not only the physical but also the mechanical properties of the material, such as its Young's modulus [13].

In the work to be reported here, the strain and stress sensitivities of POF Bragg grating sensors are investigated before and after they have been thermally annealed and chemically etched. First, a number of Bragg grating devices have been photo-inscribed in a poly(methyl methacrylate) (PMMA) based optical fibre by using the typical phase-mask technique. After the fabrication, each sensor has been characterised with respect the strain and stress sensitivity. In the next step, some sensors have been thermally annealed while some other have been chemically etched with acetone. The strain and stress sensitivity of each sensor has been characterised again at this point. Then, some annealed sensors were placed for etching and some etched sensors were placed for annealing in order to investigate the possibility for further strain and stress sensitivity enhancement.

2. Experimental details

A continuous-wave He-Cd laser (Kimmon IK3301R-G) emitting light at 325 nm with an output power of 30 mW and a phase-mask technique is used to fabricate the POFBG sensors with Bragg wavelengths located in 850 nm region. The POF that is used in these experiments is PMMA single-mode micro-structured optical fibre with the core doped with benzyl dimethyl ketal for photo-sensitivity enhancement [14]. A super-luminescent diode

(Superlum SLD-371) and an optical spectrum analyser (HP 86142A) were connected with a single-mode silica fibre coupler for POFBG spectra monitoring in reflection. Grating lengths between 1.2 mm and 10 mm were fabricated; this parameter defines only the reflectivity of the grating and it does not play any role in our experimental results. During the photo-inscription process the POF was butt-coupled with a SOF patchcord which was connected with the coupler for interrogation. After the grating fabrication, each POF was placed and glued in a demountable FC/PC connector in order to facilitate easier Bragg wavelength interrogation compared with the butt coupling method.

The strain and stress sensitivities have been calculated before and after the annealing or etching process. The strain sensitivity equals $\Delta\lambda_B/\varepsilon$, where $\Delta\lambda_B$ is the Bragg wavelength shift and ε is the applied strain in the fibre. To strain the fibre, it was attached between a fixed support and a translation stage by using magnetic fibre clamps. The translation stage can stretch the fibre along the fibre axis with accuracy of 1 μm. All POFs were strained 0.5% assuring they did not exceed the elastic limit of the polymer material. In order to stress the fibre, a mass of a known value was added at the end of the fibre which was held by a fibre clamp perpendicular to the ground thereby using gravitational force to stress the fibre. The stress can be expressed as

$$\sigma = \frac{F}{A} = \frac{mg}{\pi\left(\frac{d}{2}\right)^2}, \quad (1)$$

where F is the gravitational force which can be calculated by multiplying the added mass m and the Earth's gravity acceleration g (9.8 m/s), and A is the area where the force is applied which can be calculated by measuring the diameter d of the fibre where the POFBG sensor is located. The diameter of the fibre was measured with a microscope before and after the annealing or etching process. The stress sensitivity can be defined as $\Delta\lambda_B/\sigma$. In order to conduct thermal annealing, each POF was placed in a metallic tank filled with hot water with a constant temperature. The reason of using hot water is to control the equilibrium relative humidity during annealing (100% in this case). Two different temperatures are used for thermal annealing (55 ± 2 °C and 60 ± 2 °C), however the temperature difference does not clearly provide any impact on the annealing results. Chemical etching is performed by placing POFs in a tank filled with pure acetone for 5-7 minutes. The percentage of the fibre diameter reduction after etching is 31% - 41%.

3. Results

Table 1 depicts the strain and stress sensitivities of 6 sensors before and after the fibre annealing. Results show strain and stress sensitivity enhancement in all cases. Our explanation for this observation is as follows. During the fibre drawing, the polymer molecules align with the fibre axis due to drawing force. When these stretched polymer chains are cooled down, they have less mobility compared with the bulk material due to internal stress [15, 16]. It is believed that exposing the material above the β relaxation temperature, which is 50°C for PMMA [17], can reorganise the conformation of the polymer backbone chain and relax the stressed polymer chains [18, 19]. The molecular relaxation can increase the mobility of the molecules and the fibre therefore becomes more sensitive to mechanical stress.

Table 1: Strain and stress sensitivity before and after annealing

Sensor	Strain sensitivity before annealing (pm/με)	Strain sensitivity after annealing (pm/με)	Stress sensitivity before annealing (pm/kPa)	Stress sensitivity after annealing (pm/kPa)
1	0.708 ± 0.007	0.902 ± 0.016	0.180 ± 0.027	0.260 ± 0.030
2	0.541 ± 0.019	0.694 ± 0.012	0.147 ± 0.021	0.201 ± 0.025
3	0.681 ± 0.009	0.739 ± 0.019	0.141 ± 0.021	0.217 ± 0.026
4	0.711 ± 0.007	0.879 ± 0.025	0.163 ± 0.026	0.191 ± 0.018
5	0.726 ± 0.007	0.875 ± 0.025	0.176 ± 0.009	0.208 ± 0.015
6	0.728 ± 0.004	0.889 ± 0.033	0.182 ± 0.006	0.220 ± 0.014

Table 2 shows an additional three sensors that have been annealed and then etched. Strain and stress sensitivities have been also improved after annealing as in Table 1. Then, the same sensors have been etched in order to investigate any changes regarding the strain and stress sensitivity. The results in Table 2 show a slight reduction of strain sensitivity after etching, while the stress sensitivity has been slightly improved. Table 3 depicts additional sensors that were first etched before they were annealed. In this case, there is a strain sensitivity enhancement only for sensors 14 and 15, but due to the high measurement error, no solid conclusion can be made on this matter, even with an indication of strain sensitivity enhancement in literature [13]. On the other hand, the stress sensitivity has been enhanced in the majority of sensors after etching. It is shown that fibre etching can change the internal stress distribution [20] of the fibre and decrease the Young's modulus of PMMA [13]. After annealing, the strain and stress sensitivity of the etched POFBG sensors have been further improved, as was expected from the previous results. The fact that etching can affect the sensitivity of the device to stress and strain suggests that the fibre material is radially inhomogeneous. This may have arisen either during the preform polymerisation or during the fibre drawing process.

Table 2: Strain and stress sensitivity before and after annealing, and after etching

Sensor	Strain sensitivity before annealing (pm/με)	Strain sensitivity after annealing (pm/με)	Strain sensitivity after etching (pm/με)	Stress sensitivity before annealing (pm/kPa)	Stress sensitivity after annealing (pm/kPa)	Stress sensitivity after etching (pm/kPa)
7	0.690 ± 0.011	0.883 ± 0.025	0.854 ± 0.031	0.191 ± 0.016	0.221 ± 0.024	0.259 ± 0.020
8	0.727 ± 0.015	0.849 ± 0.015	0.755 ± 0.012	0.197 ± 0.013	0.258 ± 0.028	0.260 ± 0.011
9	0.771 ± 0.016	0.943 ± 0.016	0.848 ± 0.012	0.173 ± 0.028	0.202 ± 0.030	0.241 ± 0.006

Table 3: Strain and stress sensitivity before and after etching, and after annealing

Sensor	Strain sensitivity before etching (pm/με)	Strain sensitivity after etching (pm/με)	Strain sensitivity after annealing (pm/με)	Stress sensitivity before etching (pm/kPa)	Stress sensitivity after etching (pm/kPa)	Stress sensitivity after annealing (pm/kPa)
10	0.709 ± 0.024	-	0.834 ± 0.031	0.199 ± 0.032	0.191 ± 0.016	0.249 ± 0.019
11	0.649 ± 0.014	-	0.944 ± 0.022	0.169 ± 0.009	0.183 ± 0.015	0.200 ± 0.016
12	0.657 ± 0.014	-	0.962 ± 0.023	0.146 ± 0.020	0.211 ± 0.015	0.223 ± 0.001
13	0.720 ± 0.044	0.667 ± 0.026	-	0.194 ± 0.004	0.238 ± 0.002	-
14	0.726 ± 0.044	0.768 ± 0.034	-	0.185 ± 0.007	0.200 ± 0.010	-
15	0.728 ± 0.044	0.810 ± 0.027	-	0.189 ± 0.003	0.217 ± 0.005	-

4. Conclusion

The effects of annealing and etching on the strain and stress sensitivities of PMMA optical fibre Bragg grating sensors are investigated. The annealing and etching processes have been tried in a different sequence in order to investigate their impact on sensor's performance. Results show with high confidence that thermal annealing can enhance both strain and stress sensitivities. The fibre etching process seems to improve the stress sensitivity in the majority of sensors before or after the annealing process. If the sensors are etched after annealing, the strain sensitivity seems to be slightly reduced, however due to the magnitude of measurement errors a solid conclusion cannot be made for the etching effects on the un-annealed sensors and further investigation on this matter is needed.

5. Acknowledgements

The research leading to these results has received funding from the People Programme (Marie Curie Actions) of the European Union's Seventh Framework Programme FP7/2007-2013/ under REA grant agreement No. 608382. This work was supported by Marie Curie Intra European Fellowships included in the 7th Framework Program of the European Union (projects PIEF-GA-2013-628604 and PIEF-GA-2011-302919).

6. References

[1] D. J. Webb, "Fibre Bragg grating sensors in polymer optical fibres," *Measurement Science and Technology,* vol. 26, p. 092004, 2015.

[2] C. A. F. Marques, A. Pospori, S. D, x00E, R. ez, x00Ed*, et al.*, "Aviation Fuel Gauging Sensor Utilizing Multiple Diaphragm Sensors Incorporating Polymer Optical Fiber Bragg Gratings," *IEEE Sensors Journal,* vol. 16, pp. 6122-6129, 2016.

[3] J. G. A. Griffiths, "Tables of physical and chemical constants. By G. W. C. Kaye and T. H. Laby," *The Analyst,* vol. 73, p. 704, 1948.

[4] W. Zhang, D. J. Webb, and G. D. Peng, "Investigation Into Time Response of Polymer Fiber Bragg Grating Based Humidity Sensors," *Journal of Lightwave Technology,* vol. 30, pp. 1090-1096, 2012.

[5] M. C. J. Large, J. Moran, and L. Ye, "The role of viscoelastic properties in strain testing using microstructured polymer optical fibres (mPOF)," *Measurement Science & Technology,* vol. 20, 2009.

[6] A. Abang and D. J. Webb, "Effects of annealing, pre-tension and mounting on the hysteresis of polymer strain sensors," *Measurement Science and Technology,* vol. 25, 2014.

[7] I. P. Johnson, D. J. Webb, and K. Kalli, "Utilisation of thermal annealing to record multiplexed FBG sensors in multimode microstructured polymer optical fibre," in *21st International Conference on Optical Fiber Sensors (OFS21)*, Ottawa, Canada, 2011, pp. 7327-7753.

[8] W. Yuan, A. Stefani, M. Bache, T. Jacobsen, B. Rose, N. Herholdt-Rasmussen*, et al.*, "Improved thermal and strain performance of annealed polymer optical fiber Bragg gratings," *Optics Communications,* vol. 284, pp. 176-182, 2011.

[9] G. Woyessa, K. Nielsen, A. Stefani, C. Markos, and O. Bang, "Temperature insensitive hysteresis free highly sensitive polymer optical fiber Bragg grating humidity sensor," *Optics Express,* vol. 24, pp. 1206-1213, 2016/01/25 2016.

[10] K. Carroll, A. Argyros, K. Kalli, M. Large, D. J. Webb, and C. Zhang, "Extending the working temperature range of Bragg gratings in microstructured polymer optical fibre by annealing," in *International Conference on Plastic Optical Fibres*, Turin, Italy, 2007.

[11] G. Rajan, B. Liu, Y. Luo, E. Ambikairajah, and G.-D. Peng, "High Sensitivity Force and Pressure Measurements Using Etched Singlemode Polymer Fiber Bragg Gratings," *IEEE SENSORS JOURNAL,* vol. 13, pp. 1794-1800, 2013.

[12] G. Rajan, Y. M. Noor, B. Liu, E. Ambikairaja, D. J. Webb, and G.-D. Peng, "A fast response intrinsic humidity sensor based on an etched singlemode polymer fiber Bragg grating," *Sensors and Actuators a-Physical,* vol. 203, pp. 107-111, 2013.

[13] K. Bhowmik, P. Gang-Ding, E. Ambikairajah, V. Lovric, W. R. Walsh, B. G. Prusty*, et al.*, "Intrinsic High-Sensitivity Sensors Based on Etched Single-Mode Polymer Optical Fibers," *Photonics Technology Letters, IEEE,* vol. 27, pp. 604-607, 2015.

[14] D. Saez-Rodriguez, K. Nielsen, H. K. Rasmussen, O. Bang, and D. J. Webb, "Highly photosensitive polymethyl methacrylate microstructured polymer optical fiber with doped core," *Optics Letters,* vol. 38, pp. 3769-3772, 2013.

[15] S. Fakirov, *Oriented polymer materials*: Wiley Online Library, 1996.

[16] S. Kukureka, G. Craggs, and I. Ward, "Analysis and modelling of the die drawing of polymers," *Journal of materials science,* vol. 27, pp. 3379-3388, 1992.

[17] K. Fukao, S. Uno, Y. Miyamoto, A. Hoshino, and H. Miyaji, "Dynamics of α and β processes in thin polymer films: Poly(vinyl acetate) and poly(methyl methacrylate)," *Physical Review E,* vol. 64, p. 051807, 10/24/ 2001.

[18] E. Muzeau, J. Perez, and G. P. Johari, "Mechanical spectrometry of the .beta.-relaxation in poly(methyl methacrylate)," *Macromolecules,* vol. 24, pp. 4713-4723, 1991/08/01 1991.

[19] K. Schmidt-Rohr, A. S. Kulik, H. W. Beckham, A. Ohlemacher, U. Pawelzik, C. Boeffel*, et al.*, "Molecular Nature of the .beta. Relaxation in Poly(methyl methacrylate) Investigated by Multidimensional NMR," *Macromolecules,* vol. 27, pp. 4733-4745, 1994/08/01 1994.

[20] K.-S. Lim, H.-Z. Yang, W.-Y. Chong, Y.-K. Cheong, C.-H. Lim, N. M. Ali*, et al.*, "Axial contraction in etched optical fiber due to internal stress reduction," *Optics Express,* vol. 21, pp. 2551-2562, 2013/02/11 2013.