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Impact of high UV fluences on the mechanical and sensing properties of polymer optical fibers for high strain measurements

JULIEN BONEFACINO,* XIN CHENG, CHI-FUNG JEFF PUN, STEVEN T. BOLES, AND HWA-YAW TAM

Photonics Research Center, Department of Electrical Engineering, The Hong Kong Polytechnic University, Kowloon, Hong Kong S.A.R, China

*julien.bonefacino@gmail.com

Abstract: PMMA-based fibers are widely studied for strain measurements and show repeatable results for Fiber Bragg Gratings (FBGs) inscribed using 325 nm laser and 248 nm laser. However, there is no available material mechanical behavior characterization of the UV source impact on the fiber properties. In this manuscript, fibers are irradiated with high fluence of 325 nm and 248 nm lasers and the fibers properties are investigated using dynamic mechanical analysis and tensile strain for potential use of these fibers past the yield point. It is demonstrated that the UV sources shifted the ultimate tensile strength and changed the strain hardening behavior. Tensile strain measurements show excellent repeatability for gratings inscribed with these two sources with similar sensitivity of 1.305 nm/mε for FBG inscribe with 325 nm laser, and 1.345 nm/mε for grating written with 248 nm laser in the range 0 to 1.5 % elongation. Furthermore, tests far beyond the yield point (up to 2.8 % elongation) show that grating inscribed with lower UV wavelength exhibit hysteresis. Finally, we demonstrate that 248 nm laser fluence shall be chosen carefully whereas even high 325 nm laser fluence do not critically impact the sensor properties.

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1. Introduction

Polymer optical fibers (POFs) are gaining interest from a variety of industries due to their intrinsic properties. Indeed, they exhibit numerous advantages compared to silica fibers such as lower Young modulus [1,2], higher ultimate tensile strength and fracture strength, improved bend radius, and are biocompatible [3]. Among POFs, PMMA-based fibers are the most commonly used as they can be easily fabricated, and large range of dopants can be added in the core to tailor the fibers properties such as photosensitivity or light generation [4–6].

Over the years, the investigations of dopants in PMMA fibers to reduce the attenuation and decrease grating inscription times have led researchers to explore doping the fiber core with a multitude of dopants. Two classical and still widely use core composition are diphenyl sulfide (DPS) to increase the core refractive index and Trans-4-stilbenemethanol (TS) to improve UV photosensitivity [4,7,8], and UV photosensitive benzyl dimethyl ketal (BDK) [9,10]. Historically, a 325 nm laser has been the premium choice to write FBG in POFs as it is well known that PMMA exhibit main chain scission upon irradiation below 300 nm [11,12]. Inscription times using the aforementioned dopants were still very long, in the order of minutes, until diphenyl disulfide (DPDS) was investigated, permitting a decrease of the fiber attenuation and rapid FBGs inscription time reduced to 7 ms, limited by the shutter speed [2,13]. Over the past two years, the use of 248 nm excimer laser to write FBGs in POF [14,15] have demonstrated significant reduction of the FBG writing time to as short as 15 ns [14]. Recently, [16] reported the writing of FBG in POF using only one 8-ns pulse of the fourth harmonic (266 nm) of a Nd:YAG system. However, there is no comparison of the impact of 325 nm and 248 nm UV sources on the intrinsic

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mechanical properties of the fibers, although significant dynamic mechanical analysis (DMA) studies were performed on PMMA based fibers [17–19].

Among all the sensing properties of PMMA fibers that have been investigated, one which is of particular interest is tensile strain measurements due to the material properties listed earlier. The largest elongation reported is 6.55 % [20] but only one loading cycle was used. Similarly, groups reported maximum values of 6.5 % [21] and 3.61 % [22]. Interestingly, most studies show repeatable results in the range 0 to 1.5 % elongation [23,24]. It is believed that this specific range comes from empirical results for which researchers recorded repeatable results. It is well known that the range of interest for sensing is the linear-elastic region response and there is no available characterization permitting to delimit the different mechanical behavior regions of core doped PMMA fibers upon strain tests, which could give clear indication of ranges where the sensor could be used.

In this manuscript, we solely focus on PMMA-based fiber doped with 2 %mol of diphenyl disulfide (DPDS). Firstly, the impact of high UV fluence of 248 nm excimer laser and CW 325 nm laser on the core refractive index modulation of the fibers are investigated, and the FBG wavelength and reflected peak power fluctuations post-UV exposure are recorded until stabilization. To address comments that have arisen concerning our previous reported results using fiber core doped with 4 %mol of DPDS, the authors would like to demonstrate that stabilization time post-UV exposure can be significantly reduced by post-annealing process. A comprehensive and exhaustive analysis of fiber mechanical properties is presented where DMA measurements allowed for the recording of the impact of the UV sources on the Young modulus and other mechanical properties derived from tensile testing. Specifically, one key region of interest is between the yield strength and the ultimate tensile strength in an attempt to gather information about the fiber behavior and potentially improve the sensing range. For that purpose, tensile strain measurements were performed slightly above the yield point (three cycles from 0 to 1.5 % elongation) and significantly beyond the yield point (three cycles from 0 to 2.8 % elongation).

2. Fibers investigated and FBG fabrication scheme

The preforms were fabricated in a glove box using the pull-through method and draw using our custom made POF drawing tower into 120 µm fiber [25], with core of theoretical value of 5.5 µm. The fiber attenuation was measured using the cut-back method and value of 9 dB/cm at 1550 nm was recorded. The fibers were pre-annealed at 80°C for two days prior to FBGs inscription. Two UV light source were investigated to write FBG in our polymer optical fibers, namely CW 325 nm laser (KIMMON IK3501R-G) and 248 nm excimer laser (COHERENT BraggStar M).

A single shot of 20-ns pulse at 35 mJ was used to inscribe a 6 mm grating in the fiber using 248 nm laser, corresponding to a UV fluence of 1944 mJ/cm^2, whereas a 1 min irradiation was used to write a 1-cm grating using 325 nm laser corresponding to a UV fluence of 16 520 mJ/cm^2. The UV fluence were chosen following two considerations: good quality FBGs shall be obtained, and much larger fluence shall be used to witness potential changes in the mechanical behavior of the fibers. The rationale behind is that if fibers that have been overexposed to UV demonstrate good response over large strain range measurements, it is very likely that fibers irradiated with low UV fluence would demonstrate long term reliability. Note that the set up used for 248 nm laser inscription is similar to the one used in [26], whereas the set up for 325 nm was the same referenced in our previous publications [2,13,25].

Figure 1(a) below shows the reflected peak power fluctuations of sets of FBGs inscribed using 248 nm laser and 325 nm laser within one-week post-UV irradiation. It is demonstrated that the gratings stabilized within two days in laboratory conditions, with a decay observed the first day after irradiation followed by regrowth and stabilization for FBGs inscribed using 325 nm laser. For gratings inscribed using 248 nm laser, growth of 11dB is observed within the first two days

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post-UV irradiation followed by stabilization. Furthermore, as discussion have arisen among the POF community concerning the extremely long stabilization time for FBGs inscribed within less than 10 s in 4 %mol DPDS core doped POF (reported previously [2,13]), the authors note that this process can be sped up by annealing the fibers at 60°C post-UV irradiation, similarly to previous reported method [27]. Those results are shown in Fig. 1(b), where sets of 200 ms FBGs inscribed in 4 %mol DPDS core doped fibers stabilized within 250 days at room temperature (blue) and within 2 hours when heated at 60°C (orange).

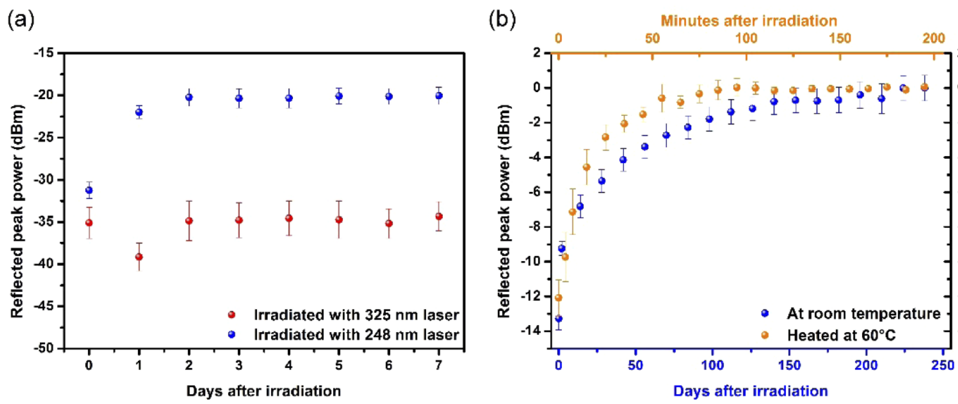

Fig. 1. (a) Reflected peak power growth recorded within one-week post UV irradiation for FBGs inscribed using 325 nm laser (red) and 248 nm laser (blue). (b) Impact of heat on the FBG stabilization time for 200 ms FBG inscribed in 4 %mol DPDS core doped POF using 325 nm laser.

The impact of UV sources on the refractive index modulations of the fibers was investigated prior to characterization. Fibers irradiated using 4 shots of 20 ns each of 248 nm laser and 4 min of 325 nm laser to mimic FBGs inscribed with 1 shot of 248 nm laser and 1 min of 325 nm laser respectively, were sent to Interfiber Analysis, LLC (Sharon, Massachusetts, USA) for refractive index measurements. Figure 2 shows the refractive index measurements performed on the two FBGs. The core and cladding refractive index of the fiber are respectively 1.497512 and 1.493379. Besides, using FWHM, it is deducted that the core diameter is 6.4 µm, slightly larger than our theoretical calculations. Figure 2(a) demonstrates that the refractive index modulation provoked by 325 nm laser is positive (1.2*10-4), which is consistent with our previous findings

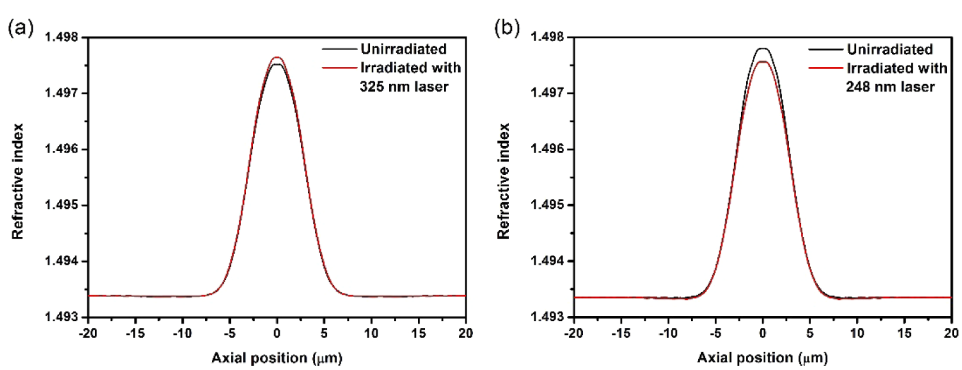

Fig. 2. Refractive index modulation recorded for (a) 4 min of 325 nm laser irradiation, and (b) four shots of 248 nm laser.

[2], where the authors showed that cleavage of the OCH_3 group of PMMA side-chain and binding of the sulphenyl radical (PhS: UV-cleaved DPDS molecule) were the dominant effects. On the other hand, the refractive index modulation induced by the excimer laser is negative (Fig. 2(b)), with value of −2.4*10-4, being indicative of main chain scission occurring in our polymer fibers, and thus permanent damage [28,29].

3. Analysis of the mechanical properties of the fiber

3.1. Dynamic mechanical analysis

To characterize our polymer fibers and the impact of the different UV source on the mechanical properties of the polymer, dynamic mechanical analysis (DMA) measurements were performed prior to tensile strain measurements. The aim of this study was to gather information about the different material response region to study the possibility of using the fibers past the yield point. The investigation was carried out using METTLER TOLEDO DMA1 on 120 µm fibers. The first characterization was the record of the impact of the UV source on the Young modulus of our fibers (Fig. 3(a)). For these tests, the 527-1:2012 standard was used for Young Modulus evaluation on polymers as previously employed by Leal-Junior *et al.* [1]. Note that in Figs. 3(a) and 3(b), "UIF" represents a batch of unirradiated fibers, "325IF" a batch of fibers irradiated with 325 nm laser, and "248IF" is a batch of fibers irradiated with 248 nm laser.

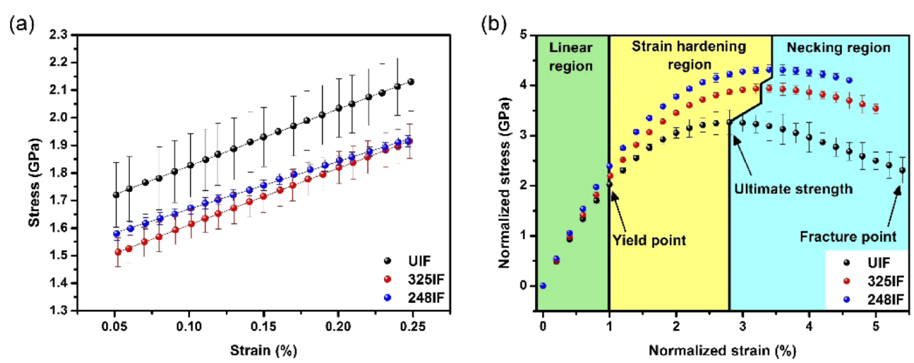

Fig. 3. Results of DMA measurements performed on unirradiated fibers (UIF), fibers irradiated with 325 nm laser (325IF), and fibers irradiated with 248 nm laser (248IF). (a) Young' modulus measurements in the range 0.05 % to 0.25 % and (b) Stress-Strain measurements.

A very interesting point in Fig. 3(a) is that the Young modulus of our homemade PMMA fibers was recorded at 2.0664 GPa, which fall into the lower range of PMMA's Young modulus as typical value record is about 4 GPa [30], and thus the fibers are expected to perform very well for strain-based measurements. The Young modulus recorded after irradiating the fibers with 325 nm and 248 nm lasers are 2.0598 GPa and 1.7260 GPa, respectively. Those values are a first hint that irradiation with 325 nm laser may not significantly changes the mechanical properties of the fibers whereas 248 nm excimer laser may induce significant changes in the fiber chemical structure leading to decrease of its Young modulus.

Further tests were performed using Stress-Strain measurements, where force of 0.1 N/min was applied to the fibers until they fracture, as shown in Fig. 3(b), where the different points of interest are marked for the unirradiated fiber (UIF). The tests were performed in controlled environment with temperature set at 25 °C and humidity recorded at 40 %RH. The measurements show in Fig. 3(b) permit collection of valuable parameters such as the linear region, the yield point, the strain hardening region, the ultimate strength, the necking region, and the fracture point [31], also given below in Table 1. First of all, Fig. 3(b) shows that all the fibers exhibit very similar yield points recorded for elongation of about 1 %, which indicate that the UV source is of no incidence for sensing in the elastic regime, which is obviously the range one shall use to obtain good and repeatable FBG response. Moreover, it can be seen on Fig. 3(b) and perhaps more accurately on Table 1, that the elongation at ultimate tensile strength is pushed further away using 248 nm laser with value of 3.44 %, to be compared with 3.33 % and 2.81 % for fiber irradiated with 325 nm laser and unirradiated, respectively. On the other hand, the elongation at fracture is following an inverted trend with value of 5.53 %, 5.04 % and 4.78 % for UIF, 325IF, and 248IF, respectively. Consequently, UV irradiation enlarge the strain hardening region compared with unirradiated fibers (1.83 %), with typical range of 2.46 % for 248IF and 2.34 % for 325IF. To the best of our knowledge, the present study is the first of its kind defining the different regions and demonstrating the impact of UV sources on them.

Table 1. Resume of the impact of UV sources on the Young modulus, failure point and yield point of DPDS core doped PMMA fibers

Fibers	Elongation at yield point (%)	Elongation at ultimate strength (%)	Elongation at fracture point (%)	Elongation range in the strain hardening region (%)	Elongation range in the necking region (%)
UIF	0.98	2.81	5.53	1.83	2.72
325IF	0.99	3.33	5.04	2.34	1.71
248IF	0.99	3.44	4.78	2.46	1.34

3.2. Tensile strain measurements

The previous subchapter as allowed us to define the optimum workable region for tensile strain measurements, namely between 0 to 1 % elongation. In an attempt to investigate the impact of UV fluence from different sources on the fiber behavior in the strain hardening region, tensile strain measurements were subsequently performed.

Firstly, a classical incremental loading/unloading of the fiber from 0 up to 1.5 % elongation was performed by step of 0.1 % elongation. The tests were performed using two 3-axis stages, one being static while the other one was used to pull the fiber, following method commonly used in the field. The range was chosen as it includes part of the strain hardening region and it was believed that minor fluctuations in the sensors responses would be observed. The strain values were maintained for 1 min and the signal was recorded using Micron Optics SM125 interrogator. For each test condition, three fibers were used to obtain good averages and verify repeatability. The data shows very good repeatability over three cycles of loading and unloading as seen on Figs. 4(a) and 4(b) for FBGs inscribed using 325 nm laser and 248 nm laser, respectively. The sensitivity for grating written using 325 nm laser was 13.05 nm/ %elongation (1.305 nm/mε) and was 13.45 nm/ %elongation (1.345 nm/mε) for FBG inscribed using 248 nm laser. Minor hysteresis can be observed for both sensors at the 0 value, since part of the tested range belongs to the strain hardening region. Interestingly, no damage in the reflected peak spectra were observed within this range for both sensors.

To further extend our study, we decided to use the same fibers tested above and extend the range from 0 to 2.8 %, by step of 0.4 %. The maximum range value was chosen to be extremely close to the ultimate strength point. It is well known for a person skill in the state of the art, that the spectra of POF FBGs are seriously deteriorated past range of about 2 % [22]. Besides, to the best of our knowledge, there is no reported study of multiple measurements above 1.5 % tensile strain. Investigation of the fibers behavior upon cycling close to the ultimate tensile strength point would be indicative of potential range of applicability for single use sensor and would give valuable information about the mechanical properties of the fibers in the strain hardening region. Therefore, cycling was performed three times and as damage of the spectra (both shape

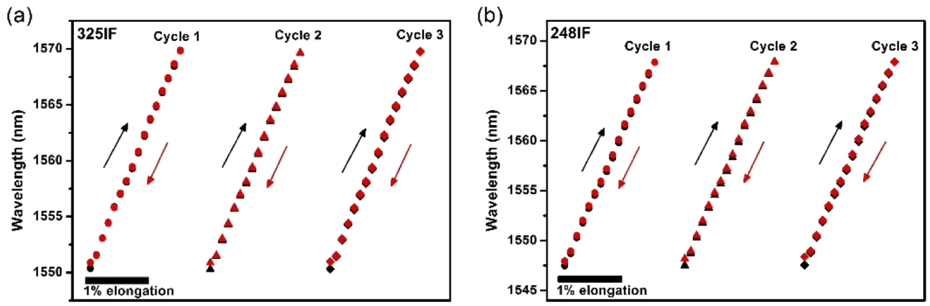

Fig. 4. Results for three cycles of loading and unloading between 0 to 1.5 % elongation for (a) FBG inscribed using 325 nm laser, and (b) grating inscribed using 248 nm laser.

and reflected peak power) occurred, the grating was left to rest in laboratory condition after each cycles and changes in the spectra were recorded until stabilization. The results of these three cycles are presented in Figs. 5(a) and 5(b), respectively for FBGs inscribed with 325 nm laser and 248 nm laser. Note that for both cases, the spectra could perfectly be recorded up to 2.8 % elongation when the strain was increased for three cycles, which is the first demonstration reported of multiple large strain range loading. However, we were only able to record the full range while decreasing the load over three cycles for FBGs inscribed using 325 nm laser has the spectra was not critically damaged. For gratings inscribed using 248 nm laser, only the first unloading data cover the full range while missing data range are respectively 1.6 % to 2.4 %, and 1.2 % to 2.4 % for the second and third unloading due to severe spectra deterioration.

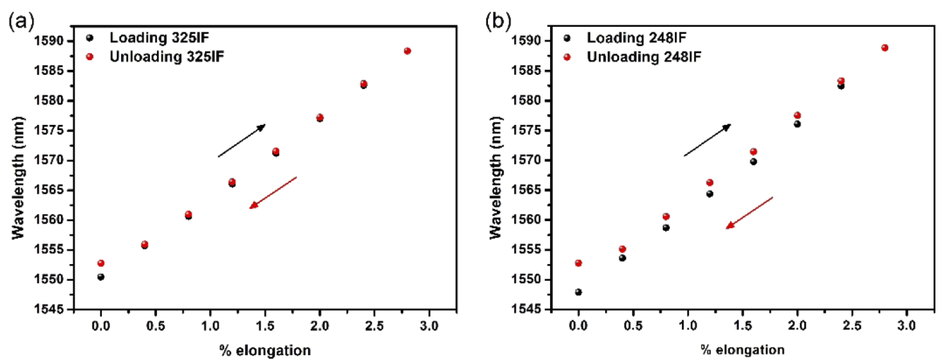

Fig. 5. Sensitivity over three cycling tests with elongation ranging from 0 % up to 2.8 % for (a) FBG inscribed using CW 325 nm laser, and (b) grating inscribed using excimer 248 nm laser.

Those findings are interesting as they demonstrate that high fluence of 248 nm irradiation has negatively changed the fiber properties in the hardening strain region. For both UV sources used, the spectra while unloading the fibers deteriorate after each cycle and the noise increased, although those deterioration were extremely severe for grating written using 248 nm laser. The spectra recorded during the third loading/unloading cycle are shown in Figs. 6(a)–6(d). Figures 6(a) and 6(c) shows that at the start of the third cycle, the fiber with enough resting time post-tensile test have recovered no matter which irradiation wavelength was employed to inscribe the gratings. Furthermore, Fig. 6(b) shows that the spectra were not critically distorted as the peak could be track during unloading for 325IF. In the other hand, the third unloading cycles performed for 248IF demonstrate critical changes in the reflected spectra with classical peak splitting and broadening. Besides, large hysteresis is recorded for 248 nm laser, and it is seen that for grating written with 325 nm laser, there is a 2.27 nm hysteresis at 0 % elongation, whereas this value is of 4.8 nm for FBG inscribe with excimer laser (Figs. 5(a), 5(b), and 6(a)–6d)). The sensitivity for FBG inscribe using 325 nm laser was of 13.60 nm/ %elongation (1.36 nm/mε), whereas it was of 14.51 nm/ %elongation (1.451 nm/mε) for grating inscribe using excimer laser. In both case, the COD was over 99 %. However, allowing the fibers to rest for five hours after each loading/unloading test demonstrated that the fibers came back to their original behavior as seen on the data for the loading tests, where no hysteresis is seen (Figs. 5(a) and 5(b)).

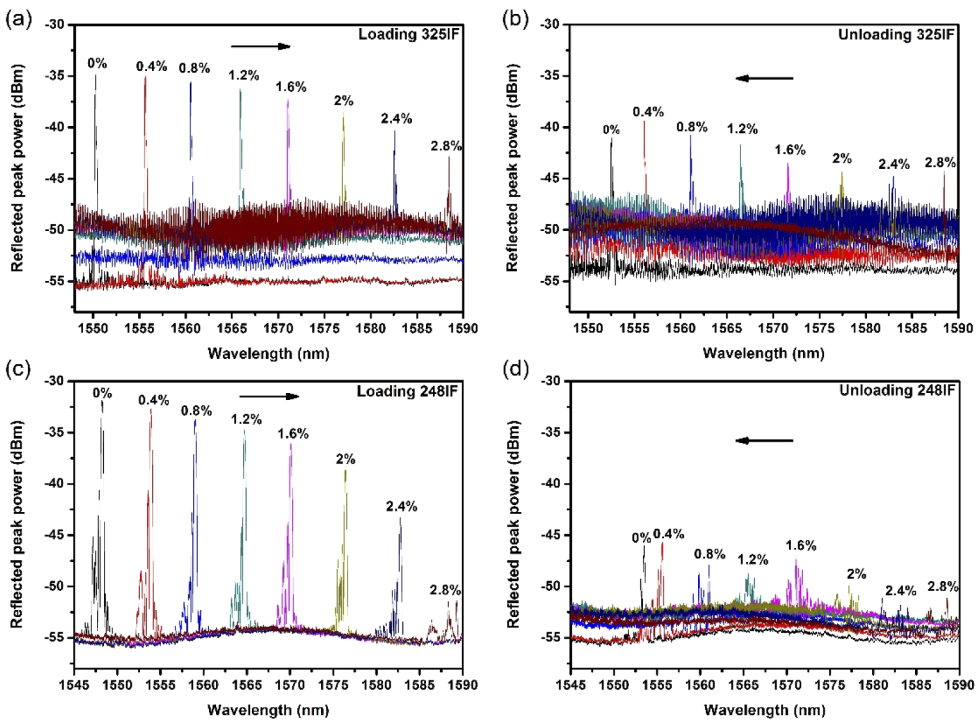

Fig. 6. Spectra recorded during the third cycling up to 2.8 % for (a) 325IF during loading, (b) 325IF during unloading, (c) 248IF during loading, and (d) 248IF during unloading.

Lastly, as we previously mentioned, after each cycle, the fibers were left for 300 minutes to stabilized and the wavelength shift and reflected peak power fluctuations were recorded. This protocol was proven successful as the spectra perfectly recovered in term of shape and reflected peak power. The wavelength and reflected peak power fluctuations post tests are shown in Figs. 7(a) and 7(b), respectively for FBGs inscribed with 325 nm laser and 248 nm laser.

The wavelength exhibits blue shift and stabilize within 200 minutes and 220 minutes for FBG inscribed respectively with 325 nm and 248 nm laser. Meanwhile it is interesting to note that the reflected peak power for FBG inscribed using 325 nm laser stabilized much faster than those written using 248 nm laser, with times of 70 minutes and 220 minutes, respectively. Obviously, those waiting time between tests do not qualify the fibers for sensing application, however they do demonstrate that the working range of the sensor can be further extended, and that 248 nm laser is relatively more harmful for the fiber than 325 nm laser. Moreover, all the loading tests showed repeatability which may be useful for applications were large strain would be sporadically applied to the fibers. Finally, it is believed that these resting time between tests could be reduced by heating the fibers or pre-straining the fiber prior to testing, which will be the subject of future investigations.

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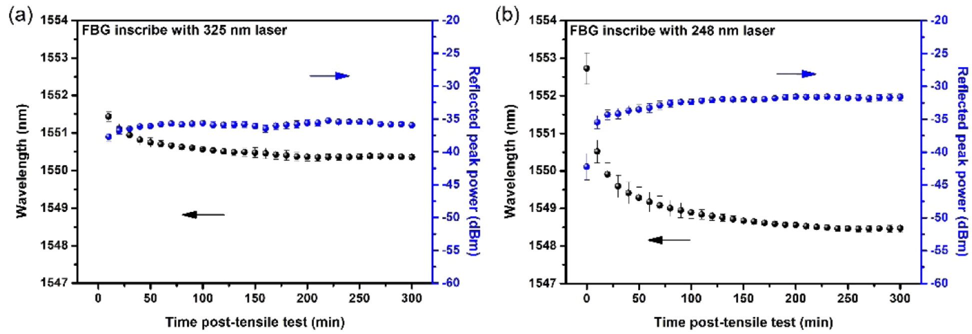

Fig. 7. Wavelength and reflected peak power fluctuation post-tensile strain measurements (a) FBG inscribed using CW 325 nm laser, and (b) grating inscribed using excimer 248 nm laser.

4. Conclusions

In this manuscript, we have demonstrated the impact of UV wavelength on the refractive index modulation in DPDS core doped PMMA fibers. It was shown that 325 nm laser induced positive refractive index modulation whereas 248 nm laser induced negative modulation, being evidence of different chemical reorganization happening in the fiber core. In our investigation of the different material response region characterization, DMA measurements permitted to demonstrate that both UV laser source have different impact on the material mechanical properties. We recorded that irradiated fibers show that the ultimate strength point shifted to higher elongation percentage and that the fracture point occurred sooner, thus enlarging the hardening strain region and reducing the necking region compared to unirradiated fibers, with 248 nm laser inducing more pronounced effect than 325 nm laser. Furthermore, the tensile strain measurements performed in the range 0 to 1.5 % elongation demonstrated excellent repeatability with average sensitivity of 1.305 nm/mε and 1.345 nm/mε for FBGs inscribed with 325 nm laser and 248 nm laser, respectively. However, small hysteresis was seen for gratings written with 248 nm and tests performed deeper in the strain hardening region (within the range 0 to 2.8 %) confirmed that point. Interestingly in that large range, repeatability can be achieved over 0.4 % to 2.8 % for FBGs inscribed using 325 nm laser but at the cost of a very long relaxation time post-tests. Those results indicate that the fibers could potentially be used past the yield point considering two conditions: the laser used to write FBG shall have low UV fluence, and the relaxing time shall be speed up.

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J.B. inscribed the gratings, conceptualized the tests protocol, performed all the DMA, gratings measurements and analysis, and wrote the manuscript. X.C. fabricated the preform and drew the fibers. All the authors discussed the manuscript.

Disclosures

The authors declare no conflicts of interest.

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