Polymer optical fiber Bragg grating inscription with a single UV laser pulse

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Abstract: We experimentally demonstrate the first polymer optical fiber Bragg grating inscribed with only one krypton fluoride laser pulse. The device has been recorded in a single-mode poly(methyl methacrylate) optical fiber, with a core doped with benzyl dimethyl ketal for photosensitivity enhancement. One laser pulse with a duration of 15 ns, which provide energy density of 974 mJ/cm^2, is adequate to introduce a refractive index change of 0.74×10^{-4} in the fiber core. After the exposure, the reflectivity of the grating increases for a few minutes following a second order exponential saturation. The produced Bragg grating structure rejects 17.9 dB transmitted power, thus providing 98.4% reflectivity, which is well suited for sensing applications. In addition, we report the importance of the fiber thermal treatment before or after the inscription, showing its effects on the lifetime and quality of the grating structures. Optimizing the irradiation conditions and the material chemical composition, a higher refractive index change in the fiber core is feasible. This demonstration significantly improves the potential for commercial exploitation of the technology.

OCIS codes: (060.3738) Fiber Bragg gratings, photosensitivity, (130.5460) Polymer waveguides, (140.3450) Laser-induced chemistry.

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1. Introduction

Polymer optical fiber Bragg gratings (POFBGs) received high attention recently, especially for sensing purposes [1], due to their unique material properties compared with their silica counterparts. Polymers have higher failure strain [2] than silica materials, enabling the POFBGs to be used as strain sensors, and they have lower Young’s modulus [3], which can provide enhanced stress [4] and pressure [5] sensitivity to POFBG sensors. The flexibility and the biocompatibility [6] of polymer optical fiber (POF) can expand the applications of POFBGs in the biomedical sector. Some hydrophilic polymeric materials, such as poly(methyl methacrylate) (PMMA), have affinity for water and therefore humidity monitoring applications are also feasible [7]. Furthermore, the large thermo-optic coefficient of POF generally provides higher temperature sensitivity compared with the silica based Bragg grating sensors [8].

The refractive index modulation forming the Bragg grating structure can be achieved by deliberately exposing the photosensitive POF to a periodic intensity pattern formed by interfering two laser beams [9]. Initially, it was claimed that the main photosensitivity mechanism during the POFBG inscription is the photo-polymerization of unreacted residual monomers [10]. However, it was reported recently that photo-polymerization may compete with a photo-degradation mechanism when POFBGs are produced [11]. When the PMMA photo-degrades, its average molecular weight decreases, which implies random chain scission degradation occurs, especially for shorter UV wavelengths [12]. At longer wavelengths a thermal degradation (unzipping) is also possible. The photo-degradation rate of PMMA is strongly related with the wavelength and the

intensity of the light source [12]. In general, for wavelengths below 250 nm the photo-degradation mechanism usually prevails, but at longer wavelengths mostly a molecular rearrangement takes place, such as photo-crosslinking and photo-polymerization [13].

Considering a relatively low energy density and a material with a low effective absorption coefficient, a number of incubation laser pulses are required before the ablation threshold of the material is exceeded [14]. After the first laser pulse (with energy lower than the ablation threshold), the polymer structure changes, and a refractive index modification is possible. The alteration of the chemical structure after the first laser pulse can increase the effective absorption coefficient of the material and lower its ablation threshold [15]. This phenomenon is called the incubation effect. The depth over which the laser energy is effectively absorbed depends on the polymer absorption coefficient, and the laser wavelength and pulse duration [16]. The ablation threshold can be possibly exceeded with only one laser pulse if it delivers sufficient energy. In general, to change the refractive index and avoid the ablation of PMMA at shorter UV wavelengths, a low laser repetition rate and low energy density should be used.

In 1999, the first POFBGs were inscribed in PMMA optical fibers by using lasers with wavelengths of 248 nm and 325 nm [17]. Using the 325 nm wavelength, the grating structures were inscribed in the fiber core. However, the authors reported a periodic ablation on the fiber surface when the 248 nm wavelength was used, due to the higher absorption of PMMA in shorter UV wavelengths [18]. Since then, lasers with 325 nm wavelength have generally been the preferred option for POFBG inscription in order to avoid any ablation issues [10]. The main reason of the fiber ablation in reference [17] was perhaps the high energy density of the laser pulses that the authors used during the POFBG inscription. In 1993, a refractive index modification in PMMA bulk material was demonstrated that is possible in lower UV wavelengths without introducing the ablation effect [19]. In this case, the authors used a krypton fluoride (KrF) laser operated at 248 nm with energy density of 40 mJ/cm^2 and repetition rate less than 5 Hz. In 2000, a refractive index modification was also demonstrated in PMMA bulk material with a UV laser operating at 193 nm and 248 nm, when energy density of 15-25 mJ/cm^2 and repetition rate of 5 Hz was used [20]. The exposure of PMMA at 308 nm did not introduce any refractive index changes, but the authors in that work used an excimer UV laser with laser pulses up to 20000. Considering that the average pulse duration of an excimer laser is between 10 ns and 15 ns, the total delivered energy perhaps was not adequate to induce any chemical structure changes at this wavelength, considering that the typical POFBG inscription time with the 325 nm laser systems is several tens of minutes [1].

Despite the reported work, the photosensitivity mechanisms are still not fully understood. In 2005, a comprehensive study on the photo-degradation of PMMA under the UV irradiation was reported [20]. The authors demonstrated that a crosslinking between the ester side chains of two polymer molecules is possible at low UV irradiation dose at 248 nm. During crosslinking the material becomes densified and as a result its refractive index increases. At low irradiation dose, the photo-polymerization mechanism can be also present, and residual monomers can be linked together. As the UV irradiation continues, at some point the polymer structure starts to photo-degradate; large molecular sections are separated from the polymer chain, and eventually the whole polymer molecule is gradually defragmented. In this case, the material becomes less dense and its refractive index decreases [20]. The final stage of photo-degradation can be the material ablation, which can be accelerated with higher energy densities [16]. In summary, the chemical structure of the material can be altered either by photo-degradation (molecular bonds are split), or by photo-crosslinking (polymer molecules are linked together), or by photo-polymerisation (the residual monomers are polymerized). All mechanisms can possibly co-exist under UV irradiation, but which one prevails over the other depends on the irradiation conditions (laser wavelength, intensity, pulse duration, and total exposure time) [20].

In 2015, appeared the first report of a POFBG inscribed in the undoped core of a few-mode

PMMA optical fiber using a 248 nm KrF laser [21]. This is the first POFBG produced with an inscription wavelength lower than 325 nm. The authors claimed that an energy density of 33 mJ/cm^2 and repetition rate of 1 Hz were used in order to minimize the ablation of the fiber. However, note that the reported energy density is before the laser pulses passed through a slit and a plano-convex lens. The focusing of the laser pulses can dramatically enhance the energy density applied on the fiber. The total inscription time of the POFBG was less than 30 seconds, which holds the record for the minimum inscription time until now. In this work, we report the first POFBG inscribed with only one UV laser pulse of duration 15 ns, achieving a breakthrough regarding the required photo-inscription time of a Bragg grating in POF. A Bragg grating with more than 98.4% reflectivity has been inscribed in a single-mode PMMA optical fiber with the refractive index change being 0.74×10^{-4}. We believe the major factor of our success relies on the inclusion of benzyl dimethyl ketal (BDK), which is a photo-initiator added in the fiber core to enhance the photosensitivity of the material [22]. The energy delivered from the laser pulse can trigger a photo-polymerization, and possibly a photo-crosslinking process. After the exposure, the change rate of the chemical structure of the material follows a second order exponential decay for a couple of minutes. Different pulse energies and the effects of multiple-pulse exposure have been also investigated. The fiber thermal annealing effect on the lifetime of the devices has also been explored, indicating that a thermal treatment before or after the inscription is perhaps essential.

2. Experimental setup

A single-mode PMMA optical fiber was used for the POFBG inscription, with a core doped with BDK for photosensitivity enhancement. The fiber is 3-ring microstructured with a hole diameter 1.74 μm and average pitch 3.7 μm. Its core size is 8 μm and its external diameter 130 μm. A cross-section image of the POF used in this work is shown in Fig. 1. The quality of fibre surface is relatively poor because the POF was cleaved by hand due to the non commercially available POF cleavers. POFBGs have been previously produced in this custom-made fiber by using a 325 nm helium-cadmium laser for the inscription; for further details see [22]. The authors showed a reduction of the total inscription time from approximately 85 minutes to less than 13 minutes. Their success relies on the addition of BDK, which acts as a photo-initiator and triggers a photo-polymerization process when irradiated with UV light. At this point, we should mention that the absorption coefficient of BDK is much higher at wavelengths shorter than 325 nm as

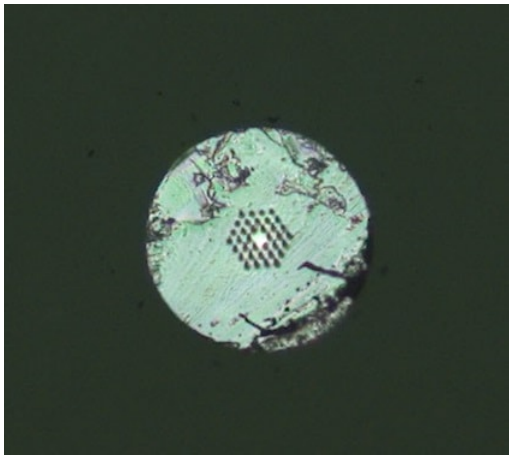

Fig. 1. Cross-section image of the POF used for inscription.

shown in [23], and we believe shorter laser inscription wavelengths could possibly enhance its effects.

Before the inscription, POF pieces of length between 15 cm and 35 cm were glued into demountable FC/PC connectors to facilitate the POFBG interrogation. A KrF excimer laser system (Coherent Bragg Star Industrial-LN), operating at 248 nm wavelength, has been used for the POFBGs inscription. The laser pulse duration is 15 ns and the pulse energy can be pre-set; in this study, energies are selected between 3.1 mJ and 6.3 mJ. The laser beam profile is a rectangular Tophat function with dimensions 6.0×1.5 mm^2 and divergence 2×1 $mrad^2$. The laser beam is focused in the fiber core utilizing a plano-convex cylindrical lens (Newport CSX200AR.10) with effective focal length of 200.0 mm. The effective spot size of the beam on the fiber surface is 20.0 mm in width and 32.4 Îijm in height. In some cases, for practical reasons, a slit (perpendicular to the fiber direction) is used to reduce the width of the beam, which defines the physical length of the grating structure. For the POFBG inscription, we used the typical phase mask technique as depicted in Fig. 2. The phase mask is 10.0 mm in width with a period of Λ_{PM}=567.8 nm and it can produce gratings with Bragg wavelengths approximately at λ_B=844.4 nm, indicating that the PMMA refractive index at this wavelength region is n=1.487 since

$$\lambda_B = n\Lambda_{PM}. \tag{1}$$

To observe the grating spectrum in transmission, the POF is connected between the super luminescent diode (Superlum SLD-371-HP1) and the optical spectrum analyzer (Yokogawa AQ6373B). For monitoring the grating spectrum in reflection, we utilize a 50:50 ratio single-mode silica optical coupler.

3. Experimental results

Initially, the POF has been exposed with multiple laser pulses in order to investigate their effects on the grating's reflected power. The time interval between each pulse was approximately 10-20 seconds. Figure 3 shows the difference between POFBG inscriptions with laser of pulses of 3.1 mJ and 5.5 mJ. The laser pulses initially have energy densities of 35 mJ/cm^2 and 61 mJ/cm^2 respectively, but when they passed through the 3.4 mm slit and plano-convex lens, their energy densities on the POF surface become approximately 482 mJ/cm^2 and 855 mJ/cm^2 respectively. The calculation of the energy density present in the fiber core area (through the 3-ring cladding structure) is practically difficult and out of the scope of this work. However, we assume the energy density in the fiber core will be considerably less, due to the absorption coefficient of the polymer and the presence of the 3-ring cladding structure, which is highly scattering. Figure 3(a) illustrates how the reflected power from the grating structure can change following each pulse with energy density of 482 mJ/cm^2 on the fiber surface. For an unknown reason, the 2nd and 3rd pulse reduced the reflected power, but from the 4th pulse the reflected power was increased. The grating's reflected power reaches a maximum level on the 10th pulse and then it decreases on the following pulses, which perhaps indicate that the grating structure starts to saturate. Figure 3(b) shows the change of the grating's reflected power after each pulse with energy density of 855 J/cm^2 on the fiber surface. In this case, the reflected power increases until the 8th laser pulse before decreasing. Note that in these and following plots, the reflected power shown is measured from the background noise level and provides a qualitative comparison between the different inscription procedures. The plots do not necessarily indicate the exact value of the grating's reflection coefficient, because the fiber connectorisation was custom-made and the insertion losses could be very high and different in each case. These plots illustrate only the effect of each laser pulse on the grating's reflected power. A quantitative measurement of grating strength, obtained by measuring in transmission, is presented later.

Figure 4 shows two POFBG inscriptions utilizing the same pulse energy of 6.3 mJ, and energy density on the fiber surface of 974 J/cm^2. In the first inscription as shown in Fig. 4(a), the reflected

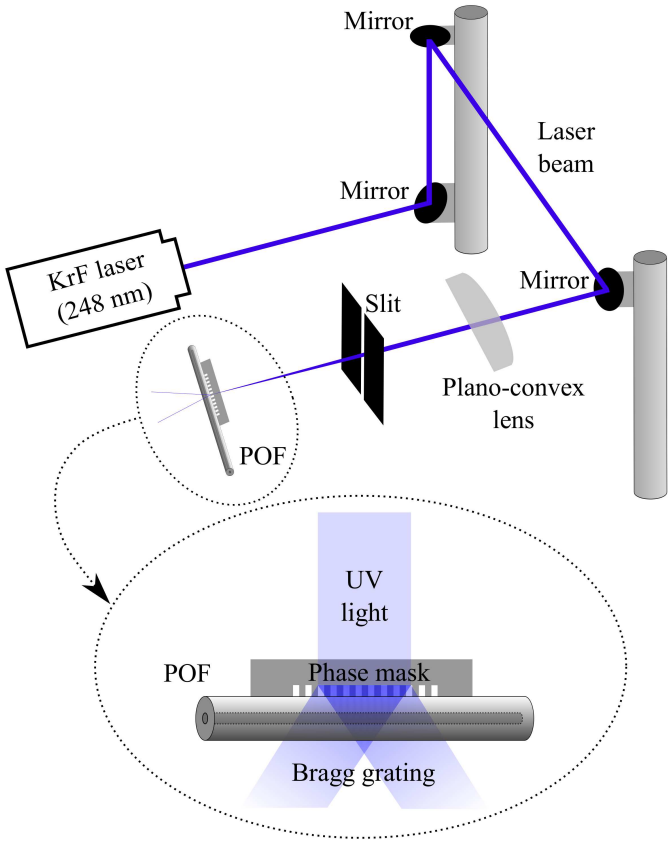

Fig. 2. Apparatus for the gratings inscription.

power of the grating reaches a maximum value on the 5th pulse. The time interval between pulses was between 10-20 seconds. Trying to repeat the same experiment, we noticed that after the first laser pulse, the reflection spectrum was not stable for approximately 4 minutes. During this time, the reflected power changed unexpectedly from 15 dB to more than 22 dB from noise level. Therefore, we decided to increase the time interval between laser pulses to 4 minutes. In this case, the reflected power decreases after the 2nd pulse as shown in Fig. 4(b). In the next step, the same experiment as Fig. 4(b) was repeated, however the reflected power was monitored following illumination with just a single laser pulse. Figure 5 shows the growth of the reflected power, which follows a second order exponential saturation for approximately 4 minutes. Similar growth behavior has been previously reported when Bragg gratings were produced in dye-doped PMMA optical fiber using a neodymium-doped yttrium aluminium garnet (Nd: YAG) laser system with operational wavelength at 325 nm [24]. In that work, the laser pulse energy was 4.5 mJ, the pulse duration was 5 ns, the repetition rate 10 Hz, and the total exposure time was 88 minutes. When the authors stopped the irradiation, the reflectivity of the Bragg grating was close to zero. Then, the authors noticed its growth after the exposure. The reflectivity had a similar growth dynamic as in our case. However, in their case the growth rate was slower, with the saturation level being reached after 8 hours, while in our work the saturation came in 4 minutes.

Additional POFBG inscriptions with single or multiple laser pulses of 6.3 mJ have been made. However, in all cases the reflected power was dramatically reduced the next day after the inscription as shown in Fig. 6. All the above POFBG inscriptions were made in non-annealed

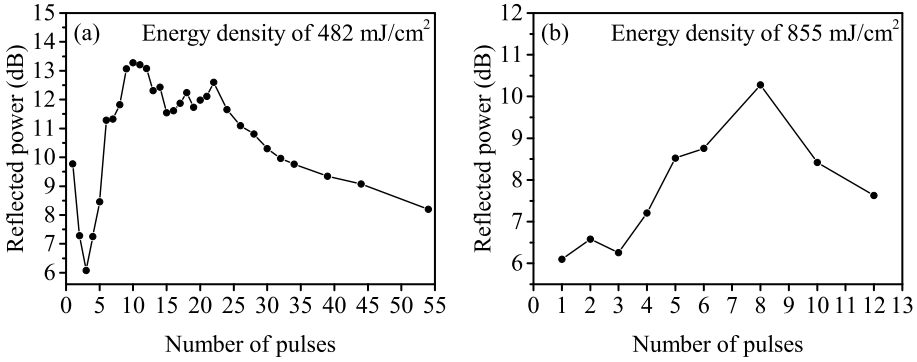

Fig. 3. Reflection of POFBG after a number of laser pulses with energy density of (a) 482 mJ/cm^2 and (b) 855 mJ/cm^2.

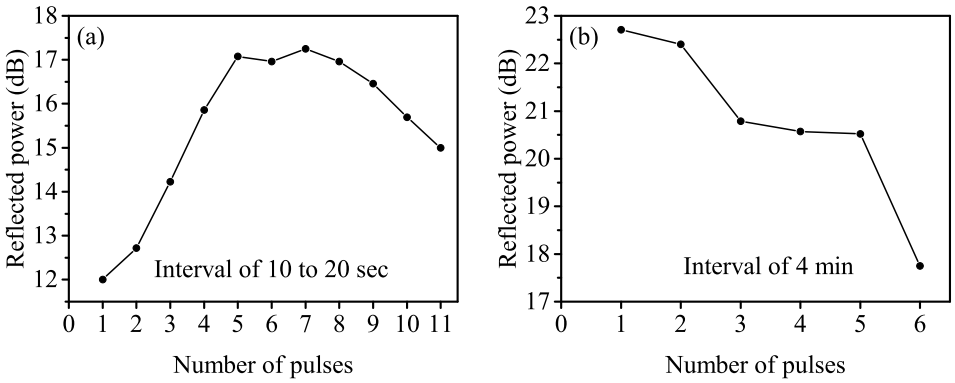

Fig. 4. Reflection of POFBG after a number of laser pulses with energy density of 974 J/cm^2 and time interval of (a) 10 to 20 sec and (b) 4 min.

POFs. A similar phenomenon has been reported recently, which shows the decay of grating reflectivity after the inscription process [25]. The authors demonstrated that the grating spectrum could be recovered with a post-annealing process. To test this with our system, a non-annealed POFBG was annealed the next day to investigate if the reflection spectrum could be recovered. To anneal the fiber, it was placed for 2 minutes in a container filled with water that was heated at 55±1 °C (we recently reported a similar annealing process in [26]). Figure 7 shows the reflected power of the POFBG before and after its thermal treatment, and the results demonstrate how the post-annealing process can recover the reflectivity of the grating. For unknown reason, the POF annealing can conspicuously influence the lifetime of the POFBGs. To further investigate this phenomenon, another POF was pre-annealed for 10 minutes in a container filled with water heated at 55±1 °C, and then a POFBG was inscribed with one laser pulse of 6.3 mJ. Furthermore, another POFBG was inscribed in a non-annealed POF with three laser pulses of 6.3 mJ, and then it was post-annealed at 55±1 °C for 2 minutes. Figure 8 shows the positive effects of both pre- and post-annealing on the reflection of POFBGs over time. Considering the above results, we can conclude that a single laser pulse in a pre-annealed POF is adequate for a POFBG inscription.

A final experiment was conducted to obtain quantitative information as to the grating strength. This time, a 30 cm long POF was pre-annealed by placing it for 30 minutes in a container filled with water heated at 55±1 °C. Then, FC/PC connectors were placed on each side of the fiber, which were polished at the end of the connectorization process to decrease the insertion losses.

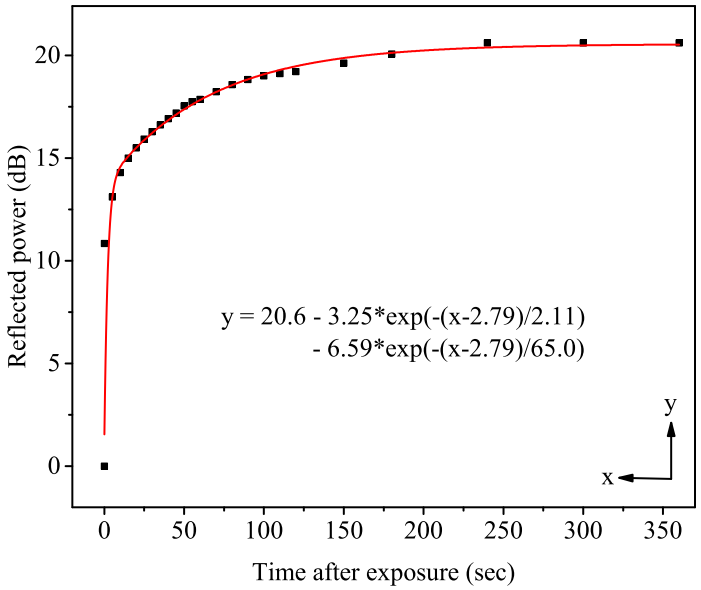

Fig. 5. Reflection growth rate over time after one laser pulse with energy of 6.3 mJ.

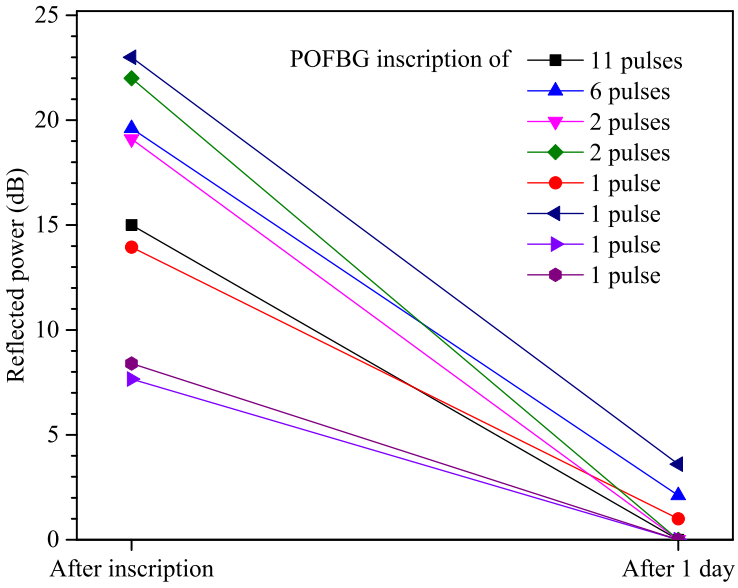

Fig. 6. Reflected power of non-annealed POFBGs just after inscription and after 1 day.

The transmitted spectrum before and after the POF connectorization is shown in Fig. 9. The total loss is approximately 2.3 dB. Considering that the fibre loss at 830 nm wavelength is 2 dB/m [27] and the POF is 30 cm long, the actual connection loss is 1.7 dB. This means the average loss per connector is 0.85 dB, which is considerably much lower compared with the recently reported connection loss of 8.45 dB [28].

In the inscription procedure, the slit (see Fig. 2) was removed from the inscription setup, in order to produce a POFBG with a physical length of 10 mm, which is the maximum limit due to

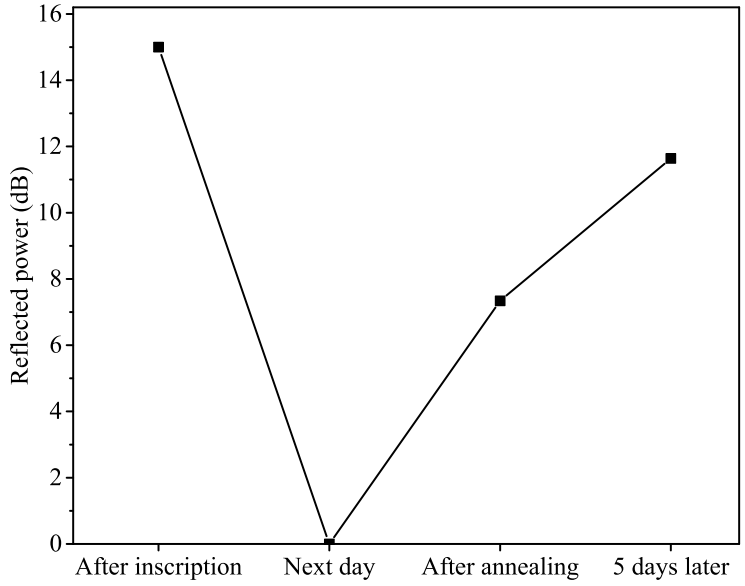

Fig. 7. Reflected power before and after the thermal annealing process.

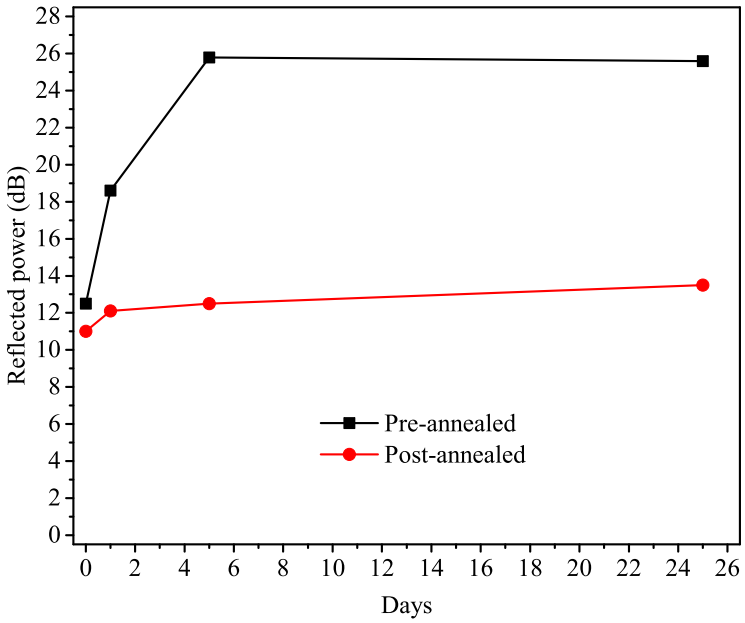

Fig. 8. Thermal annealing effects on pre-annealed and post-annealed POFBGs.

the phase mask width. The pulse energy was 6.3 mJ, and the energy density on the fiber surface was 974 mJ/cm^2. After one laser pulse, the POFBG structure was developed. Figures 10(a) and 10(b) show the reflection and transmission spectra of the POFBG, respectively, which have been taken 7 days after the inscription. The reflection spectrum can be different on each fiber side, which can be strongly influenced from the insertion loss (introduced during the custom-made fiber connectorization). The reflected power at one fiber end was 27.3 dB and at the other end

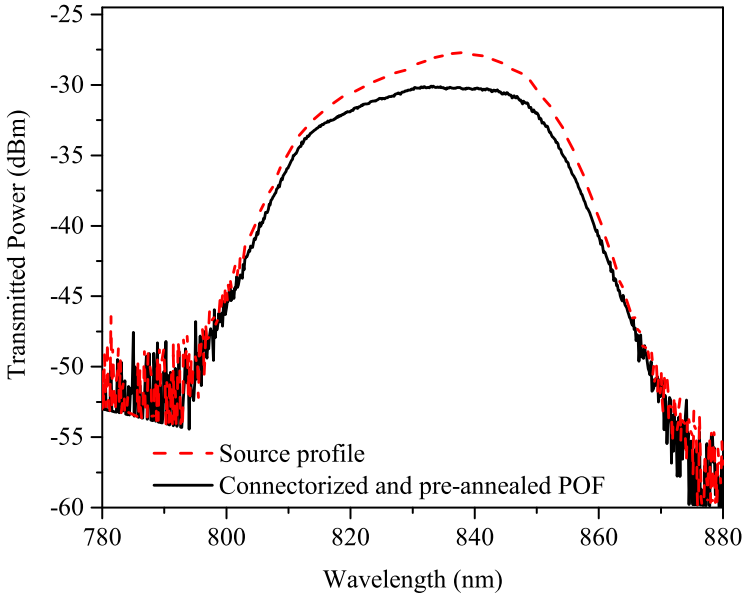

Fig. 9. Transmitted power the connectorized and pre-annealed POF.

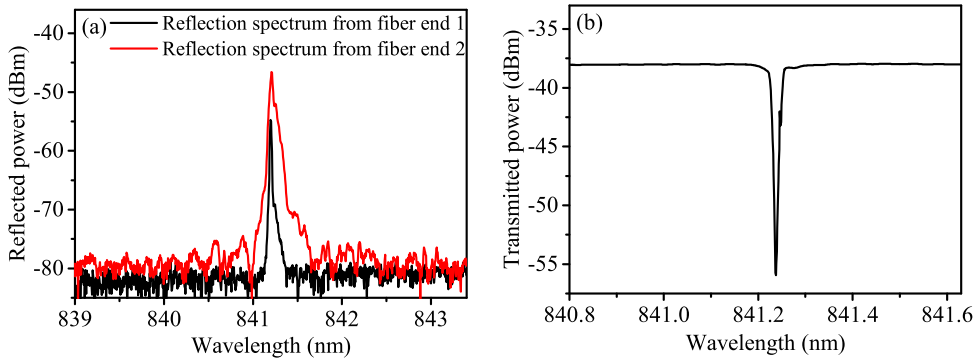

Fig. 10. (a) Reflection and (b) transmission spectra of the POFBG inscribed with one laser pulse.

was 31.5 dB from noise level as shown in Fig. 10(a). Note that the Bragg wavelength has been shifted from 844.4 nm to 841.2 nm, because the fiber was placed with some tension on the inscription setup in order to avoid being bent. Therefore, when the POF was removed from the setup, the POF returned to its original length and the grating period became shorter. Figure 10(b) shows that the POFBG has a 17.9 dB transmission rejection. Therefore, the POFBG obtained R=98.4% reflectivity after the KrF laser pulse. Considering the grating length being L=10 mm, the maximum refractive index change that has been introduced in the material is Δn=0.74×10^{-4} since

$$R = tanh^2\left(\frac{\pi \Delta n L}{\lambda_B}\right). \quad (2)$$

4. Conclusion

We report a POFBG inscription in a single-mode BDK-doped PMMA optical fiber utilizing one KrF laser pulse; its energy density of 974 mJ/cm^2 was adequate to introduce a refractive index change of 0.74×10^{-4} in the fiber core. The POFBG exhibits 98.4% reflectivity, which is satisfactory for sensing purposes. The results presented in this work show that the POFBG reflection power increases for a few minutes after the UV laser pulse, which perhaps indicates a triggered photo-polymerization, and possible photo-crosslinking event. When irradiating the POF with additional laser pulses, the reflected power starts to decrease at some point. In this case, it is likely that the chain scission photo-degradation starts to prevail over the other mechanisms. Even though some variation of pulse energies has been tried, a further investigation is still needed to find the optimum inscription conditions for the best outcome. We believe the inscription conditions may vary depending mostly on the chemical composition of the PMMA and the dopant concentration. The thermal pre- or post-annealing treatment seems to increase dramatically the lifetime of the POFBGs, and possibly enhance their reflectivity.

In conclusion, this report demonstrates the ability to inscribe POFBGs effectively with the potential to significantly reduce their production cost in an industrial environment. Importantly, the inscription process reported in this work could also be used during the POF drawing process, for simultaneous fabrication of the fiber and POFBG devices.

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