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# Fast and stable gratings inscription in POFs made of different materials with pulsed 248 nm KrF laser

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**Abstract:** This paper presents fiber Bragg grating (FBG) inscription with a pulsed 248 nm UV KrF laser in polymer optical fibers (POFs) made of different polymers, namely polymethyl methacrylate (PMMA), cyclic-olefin polymer and co-polymer, and Polycarbonate. The inscribed gratings and the corresponding inscription parameters are compared with grating inscribed in POFs made of the aforementioned materials but with the hitherto most used laser for inscription, which is a continuous wave 325 nm UV HeCd laser. Results show a reduction of the inscription time of at least 16 times. The maximum time reduction is more than 130 times. In addition, a reflectivity and a bandwidth close to or higher than the ones with the 325 nm laser were obtained. The polymer optical fiber Bragg gratings (POFBGs) inscribed with the 248 nm laser setup present high stability with small variations in their central wavelength, bandwidth, and reflectivity after 40 days.

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## 1. Introduction

Optical fiber sensors are compact, lightweight, allow multiplexing systems, present electromagnetic immunity and electrical isolation [1]. Although optical fiber sensors are commonly made of silica fibers, advances in polymer processing [2], characterization [3] and fiber Bragg grating (FBG) inscription [4] enable the application of polymer optical fibers (POFs) in sensing applications. POFs present additional advantages when compared with their silica counterparts, which include flexibility in bending, higher fracture toughness, and lower Young Modulus that provides higher sensitivity in strain sensing applications [5]. In addition, the biocompatibility and non-brittle nature of POFs means that they are clinically acceptable and can be employed in in-vivo applications [6]. Such advantages of POF sensors enable their application to measure parameters like temperature [7], strain [8], humidity [9], curvature [10], acceleration [11], and liquid level [12]. Furthermore, POF sensors are employed in biomedical applications such as antibody [13,14] and glucose sensors [15].

Of all POF materials, polymethyl methacrylate (PMMA) is yet the most employed material in POF production [5]. However, PMMA has a low glass transition temperature ( $T_g$ ) compared to some of the other polymer materials for fiber fabrication, such as polycarbonate (PC), which can limit its application at higher temperatures [8]. Furthermore, the higher moisture absorption capability of PMMA can harm its application in temperature or strain sensing where a humidity cross-sensitivity is undesirable [16]. In order to mitigate the issue of humidity cross-sensitivity, POFs made of cyclic olefin copolymer such as Topas grade 8007 [16,17] and 5013 [18,19] and cyclic olefin homopolymer such as Zeonex 480R [20] can be employed. Gratings inscribed in fiber made of these materials demonstrated a humidity sensitivity at least 30 times lower than that of PMMA POFBGs. However, the glass transition temperature can vary significantly among different grades of Topas. For instance, Topas 8007 presents  $T_g$  of only 78°C, which is even lower than the PMMA glass transition temperature [17] and which implies that fiber cleaving parameters are quite different for POFs made of PMMA and Topas [21]. The low glass transition temperature of Topas 8007 fibers severely limits the range of temperature sensing. In order to combat this, POFs made of another grade of Topas with a  $T_g$  of 134 °C was demonstrated [18,19]. Another polymer material employed for high temperature and strain sensing is Zeonex 480R, which has a  $T_g$  of 138°C. Zeonex 480R material presents further advantages compared to Topas 5013 such as superior drawability that allow a more robust fabrication of microstructures in the fiber [20]. Both Topas and Zeonex have low loss at THz frequencies and have been applied extensively in this field also [22–24]. In addition to the aforementioned POF materials, polycarbonate (PC) polymer can also be used for fiber fabrication [25]. PC has a  $T_g$  of 145°C, which is higher than that of Topas 5013 and Zeonex 480R [8]. Furthermore, PC POFs can withstand higher stresses when compared with PMMA and Topas POFs [8].

The inscription of polymer optical fiber Bragg gratings (POFBGs) has been made with different setups throughout the years. An appropriate wavelength regime is around 650 nm, where PMMA has low material loss, but the more common wavelength for POFBGs is around 850 nm, which we will also focus on here [26,27], since the polymer presents high losses at the 1550 nm region [27]. Conventionally, the inscription of POFBGs is made with the phase mask technique using continuous wave (CW) 325 nm HeCd UV lasers [25]. With this laser, the writing time in POFs was ranging from few to hundreds of minutes depending on the polymer material the fibers were made from, the inscription power, the structure of the fibers, and so on [8, 19, 21, 22]. These inscription times can be significantly reduced by doping the fiber, which can be made with trans-4-stilbenemethanol (TS) that can also increase the grating reflectivity [28,29]. Benzyl dimethyl ketal (BDK) PMMA POFs also demonstrated inscription times as short as 4 minutes with a HeCd CW laser [30] and to 40 seconds with a femtosecond laser [31] or even to 1 pulse using a KrF pulsed laser [4].

However, such doped POFs [32] are more expensive and difficult to fabricate. In addition, they are unsuitable for in-vivo applications and the dopants employed generally lead to higher transmission losses in the final sensor [33]. Since most polymers are intrinsically photosensitive, the doping is not necessary for gratings inscription [34] and the only issue of undoped POFs is the longer inscription time. Such long inscription times lead to some challenges related to the necessity of higher stability of the setup during inscription [16]. Furthermore, shorter inscription times are important for the grating stability after inscription [34]. Nevertheless, the time taken for a POFBG inscription can be reduced by several orders of magnitude with the application of a 248 nm laser with low fluence and repetition rate through the phase mask technique [35]. It was also demonstrated that the inscription time can be lowered by performing a thermal treatment on the fiber preforms [33].

To establish general POFBG technology it is extremely important to have a general FBG writing set-up that is suitable for all different polymer materials with different sensing properties. Direct femtosecond writing is one such technology [36,37], but it is very expensive. In order to use UV writing to obtain low inscription times for undoped POFs made of different materials, this paper presents FBG inscription in several different POFs using both a pulsed KrF @ 248 nm laser and a CW HeCd @ 325 nm laser with all fiber pieces properly pre-annealed. The comparison between the POFBGs obtained with both setups is made with respect to the inscription time, bandwidth and strength. In addition, the reflection and transmission spectra of the gratings obtained with the pulsed laser system are presented. Also, the POFBG stability is assessed by analyzing the grating variation over several days.

This paper investigated the inscription of gratings in POFs made of different materials using pulsed UV KrF @248 nm laser. The results obtained are compared with those gratings inscribed in the same fibers but with a continuous UV HeCd @325 nm laser system. The comparison between the POFBGs obtained with both setups is made with respect to the inscription time, bandwidth and strength. In addition, the reflection and transmission spectra of the gratings obtained with the pulsed laser system are presented. Also, the POFBG stability is assessed by analyzing the grating variation over several days.

## 2. Experimental Setup

The POFs used for the grating fabrications were PMMA, Topas 8007, Topas 5013, Zeonex 480R and Polycarbonate microstructured polymer optical fibers (mPOFs), and Topas-Zeonex step index POF [19]. All the POFs employed were fabricated at Technical University of Denmark (DTU) as presented in [8,16,18–20,34]. Table 1 presents the dimensions of each fiber, such as the cladding structure, hole diameter and pitch for mPOFs and the core and cladding diameters for step-index POFs. Furthermore, the  $T_g$  of each fiber is also presented.

**Table 1. Dimensions and  $T_g$  of each POFs employed for the grating fabrication**

POFs	Type	Cladding Structure	Hole diameter/pitch ( $\mu\text{m}$ )	Core/Cladding Diameter ( $\mu\text{m}$ )	$T_g$ ( $^{\circ}\text{C}$ )
PMMA [34]	Microstructure	3 rings hexagonal	1/3.75	8/125	110
Topas 8007 [16]	Microstructure	2 rings hexagonal	2/6	~9/240	78
Topas 5013 [18]	Microstructure	3 rings hexagonal	2.2/6	~10/130	134
Topas 5013 core – Zeonex 480R cladding step index POF [19]	Step index	-	$V = 2.38$ at 850 nm	4.8/150	134
Zeonex 480R [20]	Microstructure	3 rings hexagonal	2.2/5.5	8.8/150	138

Polycarbonate [8]	Microstructure	3 rings hexagonal	1.75/4.375	7/150	145
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Annealing is a heat treatment during which the fiber on a temperature close to the glass transition temperature for some hours [38]. Such treatment provides a reduction of the internal stress in the fiber created during fabrication process. For this reason, it reduces the POFBGs sensors hysteresis, and can also decrease the inscription time and increase the grating stability [33]. The annealing can be made before the grating inscription (pre-annealing) or after the inscription (post-annealing). If it is made after the grating inscription, the annealing can lead to a blue-shift of the Bragg wavelength [9]. All fibers except of PC were pre-annealed. PC has a high refractive index of about 1.58 and the Bragg wavelength was therefore outside the analyzer wavelength range (830-870 nm). Post-annealing was therefore made on this fiber in order to blue-shift the Bragg wavelength and to be seen on the analyzer [9]. Although the post-annealing at high humidity conditions and an optimization of the annealing time can provide larger blue-shifts, the goal here is a blue-shift sufficient only to obtain a Bragg wavelength within the analyzer range, which was achieved with the annealing time employed. Table 2 presents the annealing parameters employed for each POF used for the gratings inscription. Since it cannot be higher than the material  $T_g$ , the annealing temperature needs to be different for each POF material. For this reason, the annealing temperature of the Topas fiber with low  $T_g$  is only 60°C and the one employed for the PMMA mPOFs is 80°C, whereas, the annealing temperatures of the materials with higher  $T_g$  are higher than 100°C.

**Table 2. The annealing parameters applied for the POFs used for grating inscription**

POFs	Annealing Type	Temperature (°C)	Annealing time (h)
PMMA mPOFs	Pre-annealing	80	24
Topas 8007 mPOFs	Pre-annealing	60	24
Topas 5013 mPOFs	Pre-annealing	115	24
Topas 5013 core –Zeonex 480R cladding step index POF	Pre-annealing	115	24
Zeonex 480R mPOF	Pre-annealing	120	24
Polycarbonate mPOFs	Post-annealing	130	30

Figure 1 presents the inscription setup, which is based on a pulsed 248 nm KrF Bragg Star™ Industrial-LN excimer laser, emitting 15 ns pulses at a repetition rate of 1 Hz. The setup presents mirrors, focal lens and a slit with 4.5 mm width to position the UV beam on the phase mask employed. The laser beam profile is a rectangular Tophat function with dimensions  $6.0 \times 1.5 \text{ mm}^2$  and divergence  $2 \times 1 \text{ mrad}^2$ . The laser beam is focused onto the fiber core using a plano-convex cylindrical lens (Newport CSX200AR.10) with effective focal length of 200 mm. The effective spot size of the beam on the fiber surface is 20.0 mm in width and 32.4  $\mu\text{m}$  in height. The setup presented in Fig. 1 employs the phase mask technique, where a phase mask for 248 nm operation with a 10 mm length is used and its period is 567.8 nm. The FBG is inscribed in the fiber that is placed close to the phase mask with two 3D translational stages to guarantee the correct positioning of the fiber and avoid undesired bending of the POF.

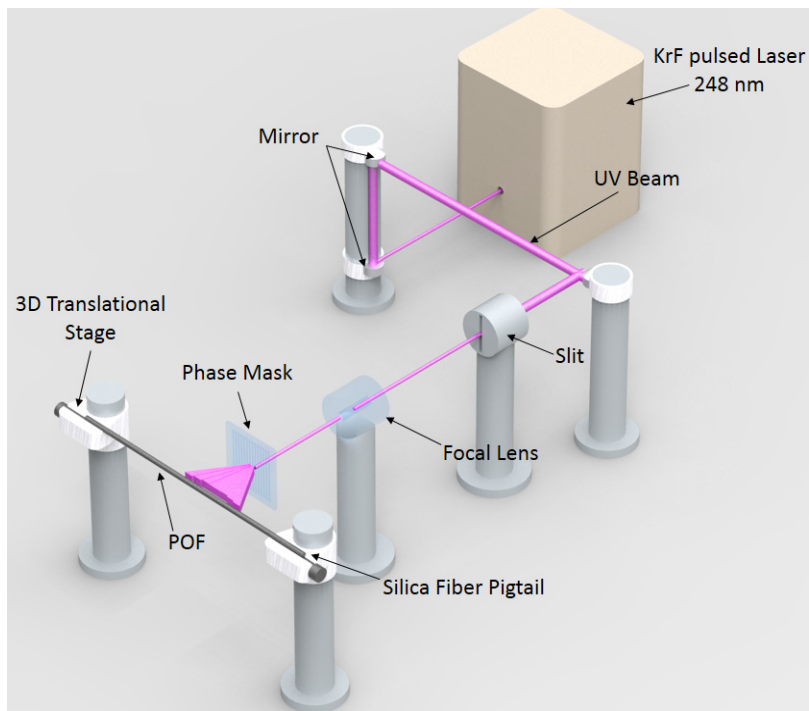


Fig. 1. Setup employed for the POFBGs inscription.

The POFs were cleaved with a hot blade on a plate using the suitable temperature reported for each fiber [8,16,18,20,21,34,38], which were butt-coupled with a silica pigtail. In order to carry out the inscription experiments, the POFs sections were UV-glued (LOCTITE AA 3936) to two  $8^\circ$  angled silica fiber pigtails on both sides of the POFs. The index matching UV-glue used between fibers could avoid Fabry–Perot cavity effects and the angled silica fiber could reduce the Fresnel reflections generated in the interface between silica fiber and UV-glue because of large refractive index difference between cured UV-glue (refractive index  $\sim 1.49$ ) and silica fiber. Provided by the manufacturer, the tensile strength of the UV-glue is 2780 psi after curing. Since the splice is the weakest part in POFBG applications, we have further increased the strength of the splicing part. An epoxy resin was dip-coated on the UV-glue splice, in order to increase its resistance. The FBGs were inscribed at the same distance from the two glue points to compare the reflected FBGs strength. All gratings were inscribed with a total length of 4.5 mm. To interrogate the reflection and transmission spectra of the gratings we used a super luminescent diode and an optical spectrum analyzer (OSA). A 50/50 optical coupler was used for the reflection spectrum.

### 3. Results and Discussion

The gratings inscribed in the aforementioned fibers with the setup presented in Fig. 1 are compared with those gratings inscribed with the CW 325 nm HeCd laser for the same fibers. The grating inscription results obtained in the literature with the 325 nm laser are presented in Table 3 with respect to the inscription time, full width at half maximum (FWHM) and grating reflectivity.

Table 3. POFBGs inscription with the CW UV HeCd laser at 325 nm

POFs	Inscription time (min.)	FWHM (nm)	Reflectivity (dB)	Grating Length (mm)
PMMA mPOF [34]	7	0.40	26	3

Topas 8007 mPOF [16]	58	0.24	11	10
Topas 5013 mPOF [18]	Not reported	0.40	20	12
Topas-Zeonex step index POF [19]	4	0.29	30	2
Zeonex 480R mPOF [20]	< 5	0.52	30	2
Polycarbonate mPOF [8]	6	0.46	25	2

For the Topas 5013 mPOF the inscription time was not reported, whereas, the inscription time reported in [16] for the Topas 8007 mPOF ranges from 58 to 338 minutes and the result with the shorter inscription time (58 minutes) is presented in Table 3. The results obtained with the 248 nm laser are presented in Table 4. Besides the inscription time, FWHM and grating reflectivity, the optimal energy per pulse in mJ is also given. Several FBGs were inscribed in each POF. If the energy per pulse is too high for the POF material, an overheating will occur, which causes an ablative process on the fiber [35]. Since the grating inscriptions were made in different POFs, a detailed study was performed to achieve the optimal energy per pulse to avoid overheating by the cumulative heating on each fiber material.

In terms of minimizing fabrication times, this laser type was recently employed to fabricate FBGs in BDk doped fiber with a single UV laser pulse with a pulse energy of 6.3 mJ, providing an energy density per inscription of 974 mJ/cm<sup>2</sup> [4]. The pulse duration is 15 ns and a repetition rate of 1 Hz was applied. Since the laser pulse duration is very short when compared with the repetition rate, it is considered that each pulse has a total time of 1 second.

**Table 4. POFBGs inscription with the pulsed UV KrF laser at 248 nm**

POFs	Inscription time (seconds)	FWHM (nm)	Reflection band (dB)	Optimal energy (mJ)
PMMA mPOF	25	0.4	32	6.0
Topas 8007 mPOF	25	0.6	31	5.5
Topas 5013 mPOF	20	0.6	23	6.0
Topas 5013 step index POF	11	0.8	31	5.0
Zeonex 480R mPOF	15	0.7	28	3.5
Polycarbonate mPOF	14	0.6	23	3.0

The results presented in Table 4 show a reduction of the inscription time of at least 16 times compared with the inscription times listed in Table 3. The highest reduction of the inscription time was obtained for the mPOF made of Topas 8007, for which it was 130 times shorter. Furthermore, the FWHM was equal to or higher than the FWHM obtained with the CW laser system. Regarding the grating strength, the reflectivity of the gratings inscribed were close to or higher than the ones obtained with the CW laser. The improvement of the inscription time and grating strength is mainly related to the pulsed laser @248 nm employed. However, such improvement can also be related to the pre-annealing made on the samples, since the results presented in [35] shows a writing time of 30 seconds with the same pulsed laser system at 248 nm presented in this work. This result was obtained for the 6-rings microstructured PMMA mPOF with a large core diameter of 18 μm, where it was not reported an annealing on the fiber prior to the grating inscription. In addition, the grating reflectivity was 20 dB and its 3-dB bandwidth was 0.16 nm. However, it was inscribed in the 1550 nm spectral region.

The reflection spectrum of each POFBG is presented in Fig. 2, where Fig. 2(a) presents the reflection for the PMMA POFBG, Fig. 2(b) shows the spectrum of the Topas 8007 POFBG, Fig. 2(c) presents the reflection spectrum obtained for the Topas 5013 POFBG. The response of Topas 5013 step index POF is presented in Fig. 2(d), whereas the reflection spectra of Zeonex 480R and PC POFBGs are presented in Figs. 2(e) and 2(f), respectively. The reflection spectra presented in Fig. 2 show a peak wavelength well above the noise floor



for each material tested. Furthermore, the central wavelength in each case is in the 850 nm region, with the longest central wavelength of the PC and Topas 5013. However, the central wavelength of the PC POFBG was even higher and was reduced to about 866 nm after the blue-shift due to the post-annealing process. Similarly, the insets of Fig. 2 present the transmission spectra of the POFBGs. To the authors' best knowledge, this is the first time that the transmission spectra of Zeonex, Topas and PC POFBGs are compared one-to-one. The differences of the initial power of each POF employed is due to the different losses of each fiber material, which was characterized in [8,18–20,34]. In addition, the POF connectorisation to the silica pigtail can also induce different losses at POF transmission spectrum.

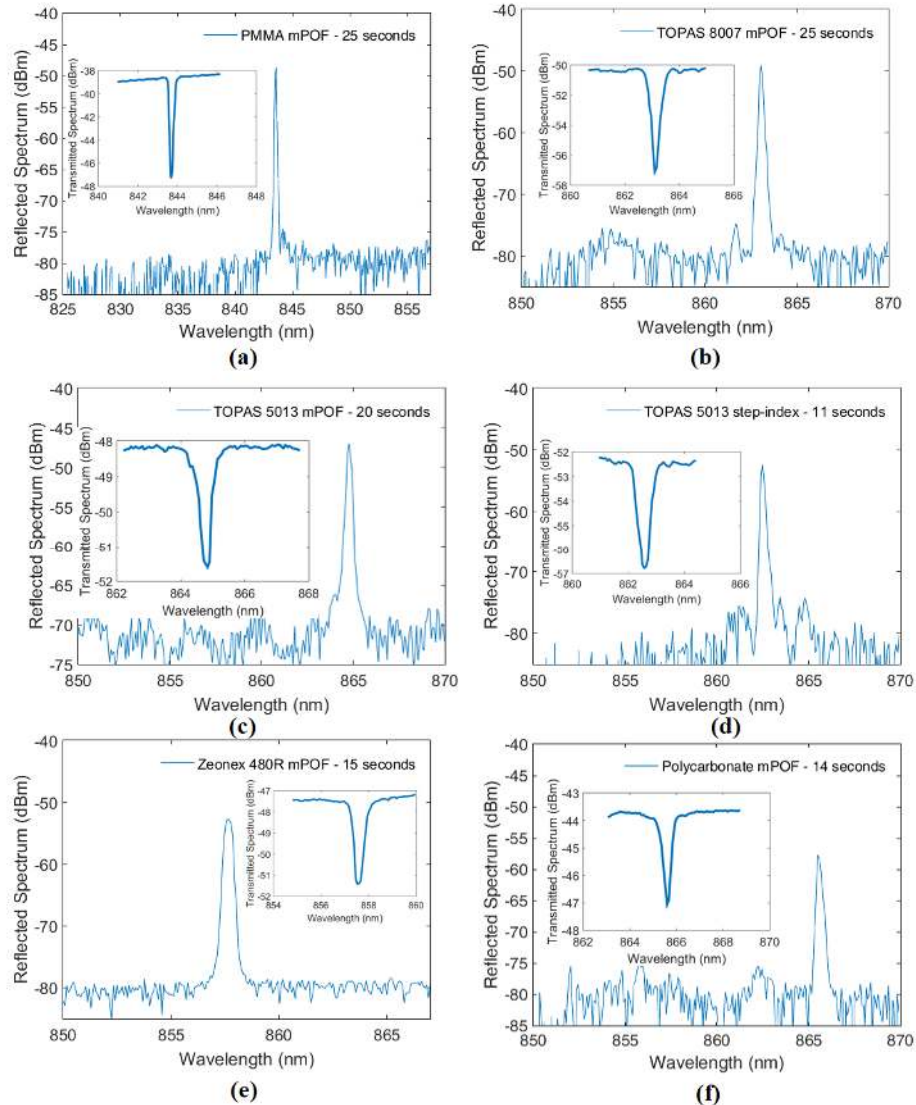


Fig. 2. Reflection spectra of the POFBGs inscribed with the pulsed UV KrF @248 nm laser. The inscription times are also presented for (a) PMMA POFBG, (b) Topas 8007POFBG, (c) Topas 5013 POFBG, (d) Topas 5013 step index POFBG, (e) Zeonex 480R POFBG and (f) Polycarbonate POFBG. The insets show the transmission spectrum of each grating.

Besides the necessity of keeping the laser and inscription setup stable for several minutes, the reduction of the gratings inscription time can also improve the FBG stability [34]. In

addition, the heat treatment made on the fibers can influence the grating stability. For this reason, the POFBGs reflection spectra were monitored for several days to verify any instability on the grating strength, bandwidth and central wavelength. In particular, all samples were kept undisturbed for 40 days at room temperature (controlled environments conditions). Figure 3 presents the results obtained for each POFBG in terms of grating reflectivity, bandwidth and central wavelength, which were monitored over 40 days by measuring them every 5 days. Figures 3(a) and 3(b) show the gratings reflectivity and bandwidth, respectively, whereas Fig. 3(c) presents the central wavelength of each POFBG. The results show a reduction in the reflectivity of all gratings by an amount lower than 3 dB that can be even smaller if the pre-annealing is made under high humidity conditions [39]. The only grating that presented a larger reduction in grating strength is the Topas 8007 POFBG. In addition, the bandwidth of each POFBG remains almost constant in all cases analyzed. The larger variation was obtained in the PC POFBGs for the measurement made after 15 days, which might be due to some torsion in the fiber, since the value returned to the initial ones in the following measurements. However, all the POFBGs presented a stable central wavelength. Since the sensor applications for FBGs are related to the central wavelength monitoring, such stable wavelength enables the use of the presented setup for fast inscription of POFBGs in sensing applications of different parameters.

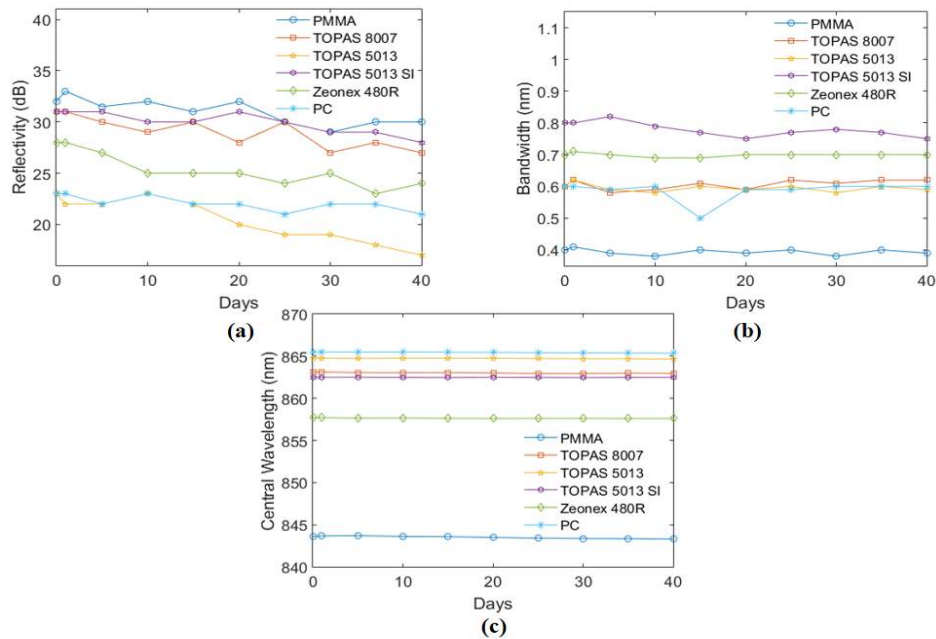


Fig. 3. Stability of each POFBG after 40 days. (a) Gratings reflectivity, (b) bandwidth of the POFBGs and (c) central wavelength.

#### 4. Conclusions

This paper presented the POFBG inscription with different POF materials with a pulsed UV KrF laser at 248 nm. The fibers employed in the grating inscription were PMMA, Topas 8007 and 5013, Zeonex and PC mPOFs and Topas 5013 step-index POFs. A pre-annealing was made of all fibers with a temperature below their glass transition temperatures and annealing times in the range of 24 hours. The comparison of the POFBGs inscribed with the technique presented in this work and with the conventional continuous 325 nm laser reported in the literature show a reduction in the inscription time of at least 16 times, where the maximum reduction was obtained for Topas 8007 mPOF (more than 130 times). Furthermore, a reflectivity of the spectra higher than the one obtained with the 325 nm laser was obtained in

almost all cases. In addition, the created POFBGs with the pulsed laser at 248 nm show stability throughout 40 days, where slight modifications of their bandwidth, reflectivity and central wavelength were observed.

The results presented in this work impact on the methods conventionally employed for the FBG inscription, where the use of a different laser setup, combined with a suitable heat treatment of the fiber, provides a substantial decrease of the inscription time without harming the grating strength, bandwidth and stability. In addition, the presented setup is similar to the one employed for FBG inscription in silica fibers, which means that, with slight modifications of the setup, it is possible to inscribed FBGs in different POF materials. The setup and the obtained results suggest that a much simpler fabrication of POFBGs is possible, which makes this technology more promising and easy to scale up, paving the way for its commercial applications.

It is important to mention that with the pulsed 248nm laser FBGs could be inscribed in 6-ring mPOFs, whereas this is not possible with the CW laser, for which typically only 3-ring mPOFs can be used. Since the loss of the mPOF decreases with the number of rings this makes the pulsed 248 nm laser FBG writing technology potentially even more advantageous.

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