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## Etching process related changes and effects on solid-core single-mode polymer optical fiber grating

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# Etching process related changes and effects on solid-core single-mode polymer optical fiber grating

## Abstract

Etching process related material and mechanical changes on solid-core single-mode polymer optical fiber (POF) and their influence on the characteristic properties of polymer fiber Bragg gratings (PFBGs) are studied. A poly(methyl methacrylate)-based POF is etched to different diameters, and it is experimentally demonstrated that etching can lead to a change in the Young's modulus of the fiber. It is found that etching process induced material changes of the polymer fiber can enhance the reflectivity and inherent sensitivity of PFBGs. It is demonstrated that gratings based on etched POF exhibit higher reflectivity with a shorter exposure time compared with unetched ones. Highest reflectivity of 98.54% is observed within 7 seconds of exposure for a diameter of 85  $\mu\text{m}$ . The stability of fabricated Bragg gratings is also studied. By tailoring the etching diameter of POF, Bragg gratings with high sensitivity and high reflectivity can be fabricated.

## Keywords

fiber, grating, related, changes, effects, solid, core, etching, single, process, mode, polymer, optical

## Disciplines

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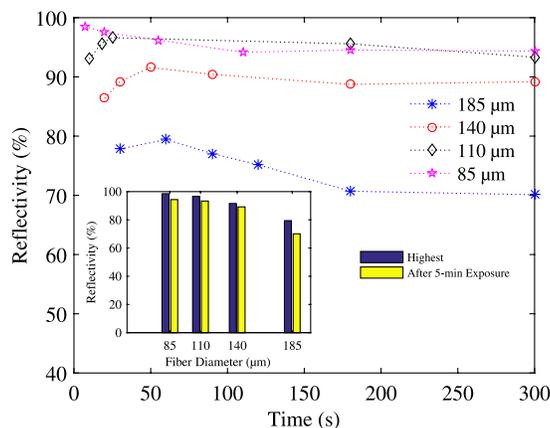
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# Etching Process Related Changes and Effects on Solid-Core Single-Mode Polymer Optical Fiber Grating

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**Abstract:** Etching process related material and mechanical changes on solid-core single-mode polymer optical fiber (POF) and their influence on the characteristic properties of polymer fiber Bragg gratings (PFBGs) are studied. A poly(methyl methacrylate)-based POF is etched to different diameters, and it is experimentally demonstrated that etching can lead to a change in the Young's modulus of the fiber. It is found that etching process induced material changes of the polymer fiber can enhance the reflectivity and inherent sensitivity of PFBGs. It is demonstrated that gratings based on etched POF exhibit higher reflectivity with a shorter exposure time compared with unetched ones. Highest reflectivity of 98.54% is observed within 7 seconds of exposure for a diameter of 85  $\mu\text{m}$ . The stability of fabricated Bragg gratings is also studied. By tailoring the etching diameter of POF, Bragg gratings with high sensitivity and high reflectivity can be fabricated.

**Index Terms:** Polymer optical fiber (POF), fiber Bragg grating (FBG), etching, Young's modulus, reflectivity.

## 1. Introduction

Single mode polymer optical fiber (POF) was first demonstrated in early 1990s by Kuzyk *et al.* [1]. During the later 1990s and early 2000s, research carried out by Chu and Peng *et al.* [2], [3] lead to the fabrication of several photosensitive PMMA based POFs made from undoped, dye-doped, and oxidated preforms, which lead to the research and development of Bragg gratings

based on POF. Single-mode photosensitive POF with different diameters those ranging from 100  $\mu\text{m}$ –250  $\mu\text{m}$  for the cladding and 6  $\mu\text{m}$ –12  $\mu\text{m}$  for the core are reported. The first demonstration of polymer optical fiber Bragg grating (POFBG) was in 1999 [3], and it has generated interest among scientists due to its unique properties such as low Young's modulus, large strain measurement range, high temperature sensitivity, and biocompatibility [4], [5]. Grating with 28-dB transmission rejection level [6] and the distinctive Type I and Type II POF Bragg gratings [7] and their growth dynamics have also been reported. More recently the interest in POF has increased and these unique properties are exploited and different applications of POFBG sensors such as strain, temperature, pressure, humidity, etc. are reported [8]–[10]. Some other applications of POFBG, such as high sensitivity accelerometer [11], highly sensitive liquid level monitoring [12], and polarization effects in transverse force sensing [13] are also reported. Since then, through the use of different fiber types and inscription processes, considerable progress has been realized towards the realization of high quality gratings in polymer fibers.

Previously, it has been reported that the inscription time for a grating fabrication in polymer fiber was approximately 1 hour [7] and more recently, together with the use of high photosensitivity POF and a 50 mW He-Cd laser with 325 nm emission, the inscription time was reduced to approximately 5–10 minutes [14], [15], but still, this performance is not sufficient for commercial bulk production for application research and not competitive with gratings based on silica fiber. Recently, the grating fabrication time for a microstructured polymer fibers reduced to second level that are reported in ref [16] and [17]. Therefore, it is important to optimize the grating fabrication procedure for a solid core single-mode polymer fiber so that POFBGs can be used in its full potential in a range of application areas. Although some significant research has been achieved in polymer grating including some applications, but still, a lot of questions regarding the formation mechanism and the growth behavior remain unclear.

For certain applications such as in biomedical/biomechanical sensors, high sensitivity with high reflectivity is required. Due to material advantages of polymer fiber compared to silica fiber, gratings based on polymer fiber could be a suitable alternative for these requirements. By modifying the material properties of the polymer fiber through solvent etching, we proved that the sensitivity can be improved intrinsically, but still high reflectivity issue is unanswered that need to be addressed. Therefore, any single technique that can provide high sensitivity and high reflectivity with less inscription time will be valued higher. In this paper, we report the potential of etched single-mode polymer fibers to develop high sensitivity and high reflectivity Bragg gratings utilizing the material property changes exhibit in the polymer fiber due to etching.

## 2. Etched Polymer Optical Fiber

The photosensitive polymer fiber used in this experiment was an in-house produced step-index single-mode fiber, one of the highest photosensitive fiber reported so far [18]. The single-mode POF sample used had an outer diameter of  $\sim 185 \mu\text{m}$  and a core diameter of 12  $\mu\text{m}$  and the fibers were single-mode at the wavelength of operation (1520–1570 nm). The difference in the refractive index between core and cladding was 0.0086.

### 2.1. Etching

To reduce the cladding thickness, the POF was etched using solvent etching technique as described in ref [14] and [19]. A laboratory grade acetone with 99.5% concentration was mixed with methanol by 1:1 ratio and then one end of the fiber was inserted into this mixture solution and then removed and cleaned. The etching time depends on the required cladding thickness and in our experiment to obtain the lowest diameter of 85  $\mu\text{m}$ , the fiber was immersed in the solution for approximately 7 minutes and the etching rate was approximately 14  $\mu\text{m}/\text{minute}$ . Microscopic images of a fiber before (185  $\mu\text{m}$ ) and after etching (85  $\mu\text{m}$ ) are shown in Fig. 1.

### 2.2. Etching Effects on the Material Properties of POF

Recently Hu *et al.* [20] reported that a cladding diameter reduction of 12% is an ideal trade-off to produce highly reflective gratings in Trans-4-stilbenemethanol-doped photosensitive polymer

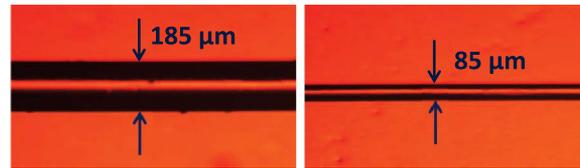


Fig. 1. Microscopic images of the polymer fiber before etching (185  $\mu\text{m}$ ) and after etching (85  $\mu\text{m}$ ).

TABLE 1

Measured Young's Modulus of the Polymer Fiber

Sample-1		Sample-2	
Fiber Diameter ( $\mu\text{m}$ )	Young's Modulus (GPa)	Fiber Diameter ( $\mu\text{m}$ )	Young's Modulus (GPa)
180 (un-etched)	2.56	450 (un-etched)	1.04
100 (etched)	2.07	324 (un-etched)	1.05
70 (etched)	1.71	340 (etched)	0.36

fibers. However the change in material properties due to etching and its influence on the sensors and grating fabrication is still a research question as the complete scenario of changes are not fully understood. Therefore, to investigate the effect of etching on the material properties of POF, Young's modulus and absorption spectrum of the etched polymer fibers are experimentally obtained [21]. In order to measure Young's modulus, a standard procedure is adopted that is valid for fibers of dimension in the range of optical fibers [22]. The experiment is conducted using Mach-1<sup>TM</sup> mechanical testing system, where each fiber sample is mounted in the testing machine and then stressed to failure at a constant cross-head displacement rate. The estimated Young's modulus from the stress-strain data obtained from the experiment for fibers with different diameters is shown in Table 1. To further verify this result, we have used a different sample (sample-2) of PMMA fibers which have different dopant concentrations and proceed with the same experiment for that fibers. Two different diameters of un-etched fiber, 450  $\mu\text{m}$  and 324  $\mu\text{m}$  are tested which resulted the same Young's modulus of 1.05 GPa. But when we etched down the 450  $\mu\text{m}$  fiber to 340  $\mu\text{m}$ , Young's modulus is reduced to 0.36 GPa. From the table it is clear that Young's modulus of the polymer fiber has significantly reduced with reduction in the fiber diameter through etching.

To further prove the concept, we have used a mathematical equation where the net Young's modulus of the POF is the combination of core and cladding Young's modulus which can be expressed as

$$E_{\text{com}} = \frac{E_{\text{co}}A_{\text{co}} + E_{\text{cl}}A_{\text{cl}}}{A_{\text{co}} + A_{\text{cl}}} \quad (1)$$

where  $E_{\text{co}}$  and  $E_{\text{cl}}$  are Young's modulus of core and cladding, and  $A_{\text{co}}$  and  $A_{\text{cl}}$  are the surface area of core and cladding. We have calculated the possible change in Young's modulus due to a change in cladding diameter which is shown in Fig. 2. In this case, we considered the core diameter as 10  $\mu\text{m}$  and assumed that the cladding diameter varies from 50  $\mu\text{m}$  to 250  $\mu\text{m}$ , and it is also assumed a maximum difference in Young's modulus of 20% between the core and cladding. From the figure, it is clear that due to smaller size of the core, the impact of cladding diameter change and the small difference in the Young's modulus between core and cladding does not have any significant impact in the net Young's modulus of the POF. The estimated difference in the worst-case scenario (20% difference between core and cladding Young's modulus)

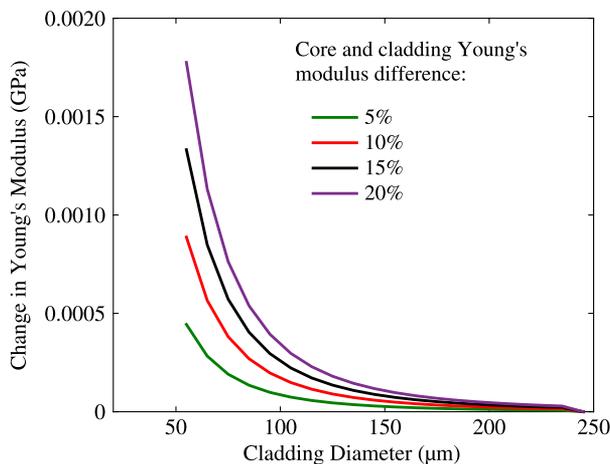


Fig. 2. Change in Young's modulus due to cladding diameter variation.

between the 55  $\mu\text{m}$  and 245  $\mu\text{m}$  cladding diameter is 0.0018, which is negligible. This confirms that the observed change in Young's modulus of the POF with different diameter is mainly due to solvent etching. The observed reduction in Young's modulus is due to two reasons; the irreversible relaxation of the tightly oriented polymer chain of the material due to the solvent absorption, as well as due to the stress relaxation of fiber due to cladding diameter reduction. When the fiber is immersed in the etching solution, the internal stress distribution of the fiber varies with fiber diameter reduction [23] which changes the fiber material properties. Therefore, the combined effect of polymer chain relaxation and fiber stress relaxation can soften the fiber material that reduces the Young's modulus.

The modified material properties of an etched polymer fiber can enhance its intrinsic sensing capabilities (strain, temperature and pressure) which is reported in [21]. To inscribe a grating in etched fiber of different diameters, the exposure time is one of the important parameter that needs to be optimized. Although some significant improvements have been achieved in polymer FBG research including etched polymer FBGs, many questions regarding the grating's formation mechanism and growth behavior still remain unanswered. This study on etched polymer fibers aims to provide the necessary insights on the grating formation mechanism.

### 3. Etched Polymer Fiber Bragg Gratings

Bragg gratings were fabricated by a standard phase-mask technique using a 50 mW Kimmon IK series He-Cd laser emitting light at 325 nm [15]. The phase mask was 10 mm long, which is suitable for a 320 nm wavelength and can produce 1 cm long gratings with a peak wavelength circa 1530 nm in the single-mode POF used in this experiment. To observe the Bragg grating reflection or transmission spectrum, a high power broadband source is used which operates in a wavelength range of 1520–1590 nm with a peak power at circa 1530 nm. In the course of the grating inscriptions, the transmission spectra of the gratings were monitored by an optical spectrum analyzer set with a resolution bandwidth of 0.2 nm.

#### 3.1. Growth Dynamics of Etched Polymer Bragg Gratings

Initially, the growth pattern of a grating in an un-etched polymer fiber with a diameter of 185  $\mu\text{m}$  is obtained for comparison with that of an etched fiber. Fig. 3 demonstrates the dynamic growth process of an un-etched polymer FBG under UV exposure where the transmission spectra at different UV exposure times are shown. In the transmission spectra, a change in transmission at around 1530 nm can be observed after 30 seconds of exposure, which is far quicker than reported in previous papers [7], [14]. The grating continues to grow stronger with further exposure and after 60 seconds of exposure, the highest dip (6.87 dB) in its transmission spectra

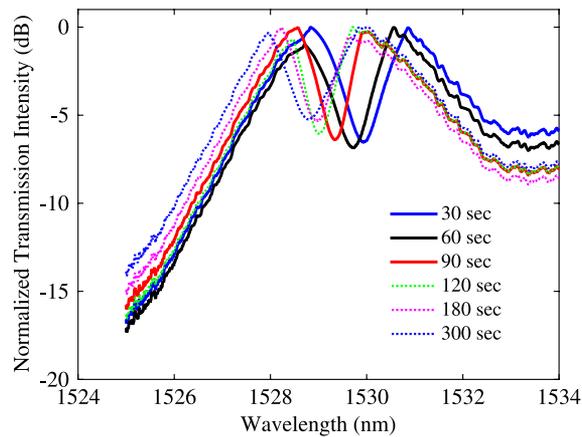


Fig. 3. Transmission spectra of the POF grating fabrication at different UV exposure time.

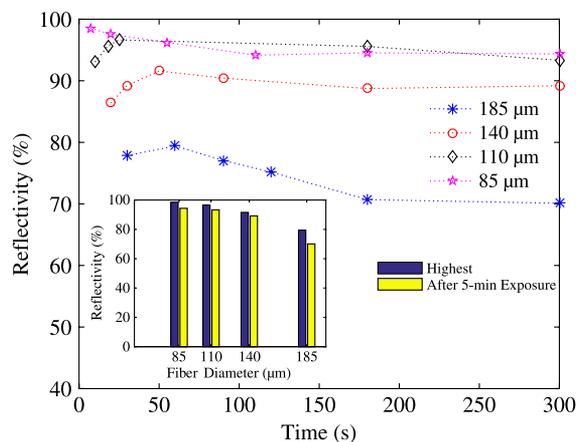


Fig. 4. Measured reflectivity pattern of polymer FBG with different diameters (Inset: Measured highest reflectivity and after 5-min inscription reflectivity).

corresponding to a reflectivity of 79.42% is observed and then decreases with further exposure. After 180 seconds of exposure, it appears stable, and after 300 seconds, this dip becomes 5.24 dB that corresponds to a 70.10% reflectivity. The blue shift observed in the transmission spectrum was due to the UV laser induced temperature change during the grating inscription procedure.

To study the effect of etching on grating fabrication, the polymer fibers are etched to different diameters (140, 110 and 85  $\mu\text{m}$ ). Then the grating is fabricated under the same conditions as of the un-etched POFBG and the same experimental set-up is used. In order to study the evolution of grating, the fiber was exposed to UV light for 5 minutes. Fig. 4 shows a comparison of the reflectivity at different exposure times for three different etched POF gratings, as well as for an un-etched POF grating. From the figure, it is clear that the growth pattern of all the gratings are similar but the reflectivity and exposure times different for the different etched polymer fibers. In addition, a graph comparing the highest reflectivity obtained and the resultant after 5 minutes of exposure is presented in the inset Fig. 4. For 85  $\mu\text{m}$  diameter etched fiber, the highest reflectivity was observed within 7-seconds of exposure with a grating transmission dip of 18.37 dB that corresponds to 98.54% reflectivity. This means that the reflectivity increases by 24.07% with a reduction in diameter of 54.05% through etching and at the same time the exposure time reduces by 88.33%. On the other hand, the highest reflectivity achieved for other two etched fibers are 91.59 and 96.63% for 140 and 110  $\mu\text{m}$  diameters,

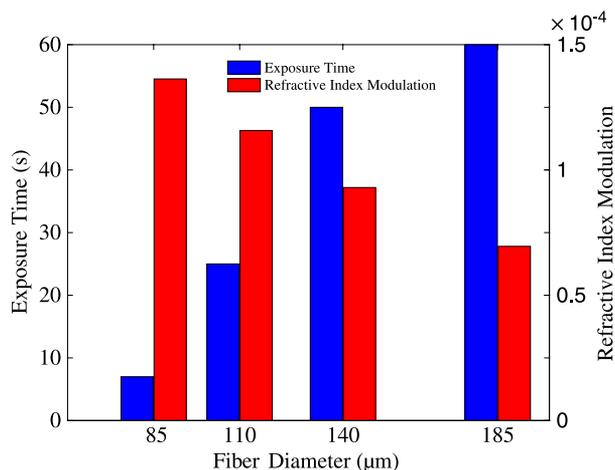


Fig. 5. Exposure time and refractive index modulation for different fiber diameters.

TABLE 2

Measured Parameters of the Bragg Gratings

Fiber Diameter ( $\mu\text{m}$ )	Highest Reflectivity (%)	Exposure Time (sec)	% of Diameter Reduction	% of Reflectivity Increases	% of Exposure Time Reduction
185 (un-etched)	79.42	60	0	0	0
140	91.59	50	24.32	15.32	16.67
110	96.63	25	40.54	21.67	58.33
85	98.54	7	54.05	24.07	88.33

respectively. Whereas the reflectivity after 5-minutes of exposure are 89.19, 93.33, and 94.36% for 140, 110, and 85  $\mu\text{m}$  diameter etched fibers, respectively but for un-etched fiber, it was 70.10%.

So it is important to know the exact exposure time required to ensure the highest reflectivity of a fabricated grating. Moreover it is obvious that the exposure time required to get the highest reflectivity is less for an etched fiber than that of un-etched fiber which is shown in Fig. 5. For etched fibers with 140, 110, and 85  $\mu\text{m}$  diameters, the highest reflectivity was observed at 50, 25, and 7 seconds, respectively, compared with at 60-seconds for the un-etched fiber. Therefore, it is clear that the exposure time required to achieve the highest reflectivity of a grating is reduced through decreasing the diameter of polymer fiber by etching.

For a better understanding of the mechanism of growth behavior of POFBG with different diameters, we have estimated the strength of effective refractive index modulation corresponding to the highest reflectivity and are shown in Fig. 5. From the figure, it is clear that due to etching, the strength of refractive index modulation of the polymer FBG increases. For example, the maximum reflectivity achieved with a 85  $\mu\text{m}$  diameter etched fiber was 98.54%, corresponding to an index change of  $1.36 \times 10^{-4}$  which is approximately 49% higher than refractive index modulation happened in an unetched fiber.

For different diameter polymer fibers, the observed highest reflectivity values, exposure times, percentages of reflectivity increases and exposure time reductions for certain diameter reductions are summarized in Table 2. From the table, it is clear that, high reflectivity gratings are achieved in shorter exposure times by reducing the fiber diameter through etching. Due to

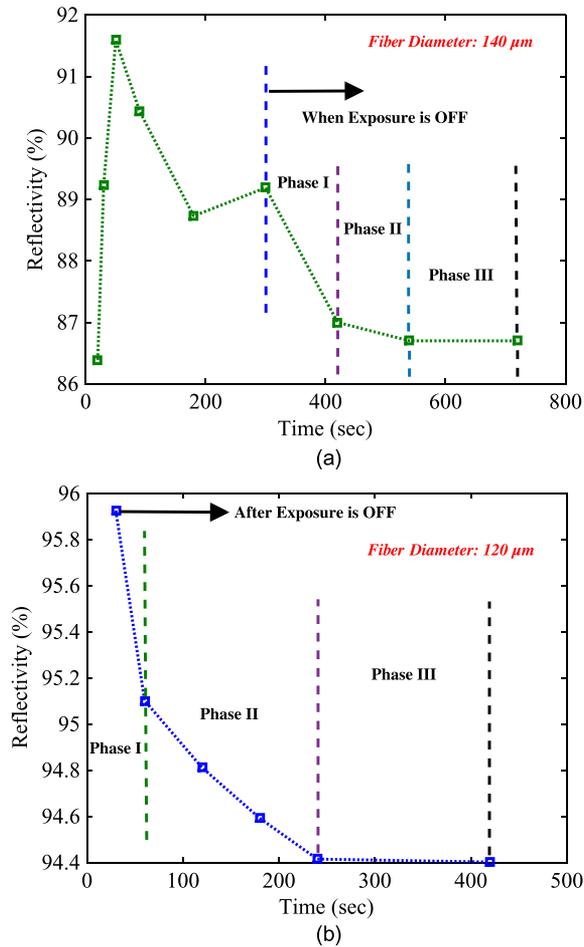


Fig. 6. (a) Grating pattern after 300 seconds exposure. (b) Grating pattern when exposure is OFF after obtaining the highest reflectivity.

experimental limitations, it is difficult to inscribe gratings in etched polymer fibers with smaller diameters.

### 3.2. Stability of Etched Polymer Bragg Gratings

To study the stability of grating behavior, two scenarios are considered: i) the pattern of a grating after 300 seconds exposure and ii) the pattern of a grating when exposure is OFF after obtaining the highest reflectivity.

In the first case, to observe the behavior of a grating after 5-minutes inscription, its transmission spectra are continuously monitored. For example, the behavior of the spectrum for 140 μm diameter etched fiber is shown in Fig. 6(a), in which it is clear that reflectivity is reduced when the exposure is OFF after 5 minutes. It can be due to the chemical stabilization of the polymer materials occurring after inscription on the polymer fiber. It is clearly observed that a sudden drop is occurred when UV irradiation is OFF which is shown in phase-I and the reflectivity reduces by approximately 2.46% within 2 minutes time duration than the last observed reflectivity at 5 minute exposure. Then, the reduction rate is less than before as shown in phase-II and finally reducing by approximately 2.80% within 7 minutes time duration and becoming stable as shown in phase-III. This means that the total reflectivity reduces by approximately 5.34% from the highest reflectivity observed at 50 seconds of exposure.

In the second case, to observe the stability of a grating after obtaining the highest reflectivity, we fabricated a grating into a 120 μm diameter etched fiber. After obtaining the highest

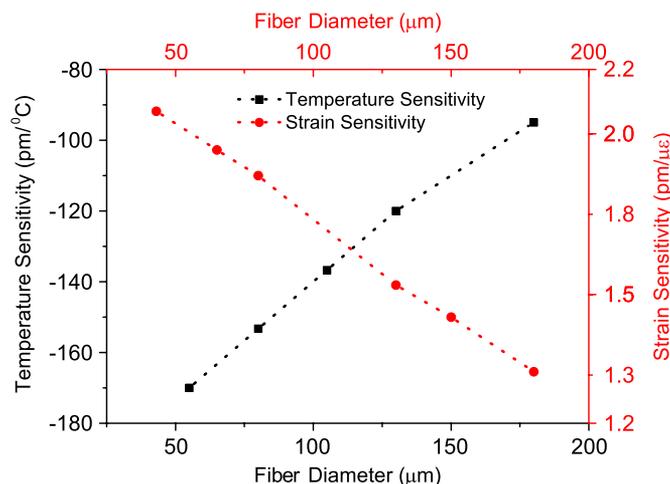


Fig. 7. Measured temperature and strain sensitivity of polymer FBG with different fiber diameters.

reflectivity at 30 seconds, further exposure is stopped and the behavior of the grating observed shown in Fig. 6(b). From the figure, it can be clearly seen that the reflectivity of the grating reduces quickly after the UV exposure is turned off as the previous one and the reflectivity reduces by approximately 0.859% within 30 seconds time after stopping the exposure, as shown in phase-I. Then, the reduction rate reduces (phase-II) and within 5 minutes, it become constant (phase-III). Finally, the reflectivity of the 120  $\mu\text{m}$  diameter etched polymer FBG is 94.402%, which means that it reduces by approximately 1.59% during the 390 seconds observation time.

### 3.3. Analysis and Discussion

From the above observations, it is clear that the reflectivity of a POF grating increases when the fiber diameter reduces through etching. The exposure time is also reduced for an etched POF grating fabrication. This is due to the fact that, in the case of an etched fiber, the UV beam can be focused into the core more effectively, as its reduced cladding will considerably decrease its absorption of UV irradiation. Therefore, the UV interference pattern at its core will have a higher intensity than that of an un-etched fiber. In addition, due to chemical etching, penetration of the solvent can change the properties of the core that can change the photosensitivity. Moreover, the fiber expansion that occurs during etching loosens the matrix of the fiber material, which can allow the UV light to react more in an etched fiber than that of an un-etched fiber. This results point to a direction that the etching procedure has a major role in the enhancement of reflectivity. Therefore from the above results, it is proved that etching has significant impact on grating inscription, which enhances reflectivity while shortening the required exposure time. This means there is an experimental trade-off between the fiber diameter and the exposure time to produce highly reflective gratings.

As we have reported earlier, etching also enhances intrinsic sensing capabilities of etched POFBG and to demonstrate those, in Fig. 7, the measured temperature and strain sensitivity of POFBG with different fiber diameters are shown. Therefore, it can be concluded that etching can improve both reflectivity and sensitivity and make it competitive to use in a range of applications where high sensitivity also matters.

## 4. Conclusion

A detailed experimental investigation on solvent etching effects on solid-core single-mode POF and the characteristic properties of gratings inscribed in etched polymer fibers were carried out. The experimental investigation on the etched POF Bragg grating demonstrated that etched fibers with lower diameter can exhibit high reflectivity and high sensitivity compared to un-etched

ones. This observed phenomenon is credited to the solvent etching process which induces material and mechanical property changes in POF which in turn influenced the reflectivity of the grating. The study showed that the exposure time required for etched polymer fiber is far less compared to unetched one. An exposure time of 7 seconds for an 85  $\mu\text{m}$  fiber produced a POFBG with 98.54% reflectivity. We also studied and reported the stability of gratings under different scenarios, which can provide an insight into the grating behavior after fabrication. These results establish the prospective of etching effects on single-mode polymer fibers to develop high reflective gratings that can lead to further research in this area.

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