

Lasers in Manufacturing Conference 2015

## Investigation on Bragg grating formation in a perfluorinated polymer optical fiber

Simon Kibben<sup>a\*</sup>, Michael Koerdt<sup>b</sup>, Frank Vollertsen<sup>c</sup>

<sup>a</sup>BIAS – Bremer Institut für angewandte Strahltechnik GmbH, Klagenfurter Straße 2, 28359 Bremen, Germany

<sup>b</sup>Faserinstitut Bremen, Am Biologischen Garten 2, 28359 Bremen, Germany

<sup>c</sup>BIAS – Bremer Institut für angewandte Strahltechnik GmbH and University of Bremen, Klagenfurter Straße 2, 28359 Bremen, Germany

### Abstract

We present first results of ultraviolet laser induced fiber Bragg gratings (FBG) in a commercially available perfluorinated polymer optical fiber (POF) and their characterization results. FBG have gained interest in the past few years as the fiber is very thin and therefore easily can be integrated into composite structures and monitor the temperature and strain state. The type of polymer used in this study shows a much higher transparency compared to Polymethylmethacrylate (PMMA) based optical fibers. Up to now, only gratings in thin slabs of CYTOP<sup>®</sup> (cyclic transparent optical polymer) were successfully detected. The gratings presented were inscribed using a krypton fluoride excimer laser and the well-known phase mask method. The polymer optical fiber (POF) type used in the investigations was GigaPOF-62SR from Chromis Fiberoptics, Inc. The fabricated gratings have a reflection maximum at 1450 nm and show many reflection peaks over a bandwidth of 10 nm. Prior to the inscription, an overcladding was removed using dichloromethane. The gratings were inscribed with 40 mJ/cm<sup>2</sup> pulse fluence and a total fluence of 2.5 to 5 kJ/cm<sup>2</sup>. A pre-treatment of the POF in an evacuated steel tube followed by an oxygen atmosphere at 80°C, each for one day, and a temper step after the grating inscription show promising results for a much lower total fluence of 1 kJ/cm<sup>2</sup> and lower. This also means shorter grating inscription duration. The inscription time is crucial for a commercial use of this new type of POF-FBG in the field of structural health monitoring of composite structures.

*Keywords:* polymer optical fiber, fiber Bragg grating

---

\* Corresponding author. Tel.: +49-421-218-58066; fax: +49-421-218-58063.  
E-mail address: kibben@bias.de.

## 1. Introduction

Fiber Bragg gratings have gained interest in the past decades, as they can act as sensing elements for measuring strain and temperature. This was discovered by Hill et al., 1978 in glass optical fibers (GOF). The phenomena were precisely characterized by Morey et al., 1990. Most of today's FBG are inscribed into SiO<sub>2</sub>-based optical fibers. Polymer optical fibers (POF) can be advantageous compared to glass based fibers in some special cases. This is especially the case for embedding the optical fibers into fiber reinforced plastics (FRP), where glass based optical fibers often break (Kang et al., 2006).

First FBG in POF were reported by Peng et al., 1999, where a FBG was formed in the core of a dye-doped Poly(methyl methacrylate) (PMMA) based fiber. A major disadvantage of the PMMA-based POF compared to GOF is their very high absorption in the infrared (IR) spectral range. This inhibits the use of these POF for using the cheap and well-established inscription and measurement equipment from IR telecommunications, which mostly uses the wavelengths around 1550 nm.

A new type of POF is available since a few years, which has quite low absorption in the telecom IR. This fiber bases on a perfluorinated polymer, which does not have the IR absorption bands arising from C-H bonds. The polymer material is called CYTOP®, which stands for cyclic transparent optical polymer. The fiber is commercially available from Chromis fiberoptics under the name GIPOF. This fiber is a promising candidate enabling POF-FBG measurements in the IR range over 10 meter and more.

Until recently, it was not possible to inscribe FBG directly into the core of the CYTOP fiber, however the photosensitivity of the polymer was shown by Liu et al., 2001, where the polymer material was thermoformed to thin slabs which then were irradiated with a frequency-tripled Nd:YAG laser.

In 2014, Koerdet et al. used the well-known approach from the GOF with a KrF excimer laser as ultraviolet (UV) source and a phase mask which forms a sinusoidal intensity pattern, in which the CYTOP fiber is placed. A FBG was inscribed over the whole core and a reflectivity of about 5 dB ( $\approx 70\%$ ) was measured. Several Bragg transmission dips could be observed, which are believed to correlate with the principal modes of the CYTOP multimode fiber.

In 2015, Lacraz et al., 2015 presented a femtosecond laser inscription approach. The grating was inscribed layer by layer using a 517 nm femtosecond laser with 220 fs pulse length and repetition rate of 1 kHz. The inscribed FBG also shows a reflectivity of about 70 % but only has a single transmission dip.

Here we present a more efficient method for the Grating inscription based on the research of Koerdet et al., 2014.

## 2. Experimental

As POF, GigaPOF-62SR manufactured by Chromis Fiberoptics Inc. was selected. The POF was cut with a razor blade in a length of about 300 mm. Each piece was put into an extraction thimble, which then was placed in a Soxhlet extractor. The extraction was done according to DIN 54278-1: 1978-02 using dichloromethane. The soxhlet extraction was carried out to remove the over-cladding, which is not UV transparent.

The POF samples could be placed in a vacuum chamber to let a gas diffuse into the POF. For this, the POF was placed in a stainless steel tube with an inner diameter of 6 mm, which could be evacuated down to 10<sup>-4</sup> mbar. The steel tube could be flushed with oxygen with a pressure of 1 bar. The steel tube could be heated up to 80° C.

The POF sample without over-cladding was fixed on one side of the sample holder using adhesive tape. The other end was fixed to a weight of 2 g and was let hang lose on the other side of the sample holder to keep the POF straight. A phase mask with a grating period of 1084.45 nm and an active area of 1 cm x 1 cm

for a illumination wavelength of 248 nm manufactured by Ibsen Photonics A/S was employed. The mask was placed with its grating side in direct contact to the POF.

As a UV laser source, a krypton fluoride excimer laser Lambda Physik LPX 305i emitting at 248 nm was used. The laser had an unstable resonator. The radiation has a spatial coherence of about 500  $\mu\text{m}$  and a temporal coherence of about 50  $\mu\text{m}$ , the pulse length is 20 ns (according to the manufacturer). The beam profile was reduced to 1 cm x 1 cm via an aperture. During the irradiation process, the pulse fluence was measured and stabilized by a computer-controlled attenuator which was integrated in the beam path. For the pulse energy measurement, a beamsplitter was placed in the beam path which directed 10 % of the pulse energy onto a calibrated detector, which was connected to the computer controlling the attenuator. During the irradiations, each pulse was recorded and the laser was automatically switched off when the predefined total fluence was reached. Behind the beamsplitter, a 45°-mirror was placed to direct the beam perpendicularly onto the phase mask. The sample holder with the fixed POF and the phase mask was placed in the beam and was precisely adjusted, so that the UV radiation passes through the active region of the phase mask. This position was marked with mechanical stops to guarantee a reproducibility of the positioning. The pulse fluence for the grating inscription was adjusted to 40  $\text{mJ}/\text{cm}^2$ , the laser was operated at a repetition rate of 50 Hz. After irradiation, the sample was placed on a hotplate for 18 h.

After the irradiation and the thermal treatment, the samples were prepared for optical characterization. The sample was cut to a length of ca. 2 cm with the grating region in the center. This was carried out with a one-sided razor blade manufactured by Lutz Blades type 0402. The short fiber piece then was placed on a polymer block as a holder and fixed with small pieces of adhesive tape.

The optical characterization of the samples was carried out with a superluminescent light emitting diode (SLED) broadband source manufactured by Denselight emitting from 1420 nm to 1500 nm and an optical spectrum analyzer (OSA) from EXFO model IQ5240. The measurement of the OSA had a measurement inaccuracy of 0.1 dB (~2.2 %). The light from the broadband source was launched into the sample using a standard singlemode fiber and a Physik Instrumente F-206.01 Hexapod. On the outcoupling side, also a singlemode fiber fixed on a Hexapod was used. For measuring the transmitted power, a photodiode by Rifoc model 675RE could be used instead of the OSA. The grating samples were fixed between the two hexapods for launching the broadband IR radiation into the sample and collecting the transmitted IR radiation. The coupling was first done by eye. After rough positioning of the launch and output fiber, the coupling was done with automated scanning in a parallel plane to the fiber end faces and a simultaneous measurement of the output power. This was done iteratively for the output and launch fiber starting with a step width of 10  $\mu\text{m}$ , 5  $\mu\text{m}$  and 1  $\mu\text{m}$ .

Gratings were inscribed in two differently treated POF. The first type of fiber was put in the Soxhlet extractor with dichloromethane for the removal of the UV-absorbing over-cladding and had no further treatment. The gratings were inscribed with a pulse fluence of 40  $\text{mJ}/\text{cm}^2$ . Samples with total fluences of 0.5  $\text{kJ}/\text{cm}^2$ , 1  $\text{kJ}/\text{cm}^2$ , 1.5  $\text{kJ}/\text{cm}^2$ , 2.5  $\text{kJ}/\text{cm}^2$  and 5  $\text{kJ}/\text{cm}^2$  were fabricated. The samples were put on a hot plate at 80 °C for 18 h before the optical characterization was carried out.

The second type of fiber was put in the steel tube at 80 °C after the Soxhlet extraction. The fiber was stored for 24 h in a vacuum of  $10^{-4}$  mbar and another 24 h in an oxygen atmosphere of 1 bar. The gratings were inscribed with a pulse fluence of 40  $\text{mJ}/\text{cm}^2$ . The total fluences of the gratings were 0.5  $\text{kJ}/\text{cm}^2$  and 1  $\text{kJ}/\text{cm}^2$ . The samples were put on a hot plate at 90 °C prior to characterization.

### 3. Results

#### 3.1. Gratings in non-oxygen treated fibers

Due to the serious mode mismatch of the launch and output fiber compared to the graded index perfluorinated fiber with the inscribed grating, the principle of energy conservation could not be applied to the measurement. This means that the sum of the measured reflection and transmission spectrum does not represent the whole energy provided by the SLED. Also, as a consequence, the reflection spectrum does not represent a value for the maximum reflectivity of the grating. This only can be extracted from the transmission spectra by comparing the depth of the transmission dips with the rest of the spectrum, where no reflection occurs. However, of each sample a reflection and a transmission spectrum was recorded.

The gratings with total fluences below  $2.5 \text{ kJ/cm}^2$  showed a low reflection in the reflection spectrum, but the dips in the transmission spectrum were not significant (reflection smaller than 2.2 %). The gratings fabricated with  $2.5 \text{ kJ/cm}^2$  and  $5 \text{ kJ/cm}^2$  both showed a significant reflection. The grating inscribed with a total fluence of  $2.5 \text{ kJ/cm}^2$  had a reflection of 0.8 dB (~17 %), the grating inscribed with a total fluence of  $5 \text{ kJ/cm}^2$  had a reflection of 3.1 dB (~51 %). Both gratings had more than one transmission dip. Each of them is believed to correspond to a principal mode of the graded index polymer optical fiber, which is being excited by the launch fiber. In the transmission spectrum of the fiber inscribed with  $5 \text{ kJ/cm}^2$  13 transmission dips can be identified. The dip with the shortest wavelength has its minimum at 1448.42 nm, the dip with the longest wavelength is at 1456.81 nm. Each dip has a distance of about 0.65 nm to each other. The overall bandwidth of the transmission dips is about 9 nm. The grating inscribed with a total fluence of  $2.5 \text{ kJ/cm}^2$  only shows six clearly identifiable transmission dips in the range from 1447.0 nm to 1450.32 nm. The dips have a distance of about 0.66 nm to each other. On each side two more dips can be estimated, however their reflectivity is very low.

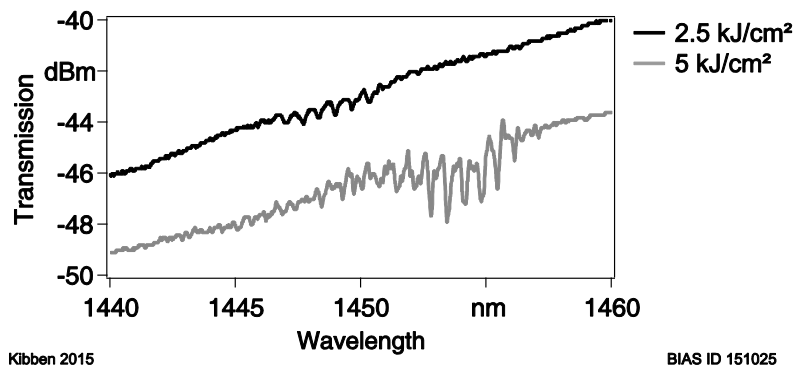


Fig. 1. Transmission spectra of both gratings inscribed in a POF without previous oxygen loading of the fiber. For better visibility, the reflection spectrum of the  $2.5 \text{ kJ/cm}^2$  sample was shifted for 3 dBm.

#### 3.2. Gratings in oxygen treated fibers

The two samples were optically characterized and a transmission and reflection spectrum was recorded. Both spectra are shown in Fig 2. In the transmission spectrum of the sample fabricated with a total fluence of  $0.5 \text{ kJ/cm}^2$ , the highest reflectivity of 9.4 dB (~88.5%) is at 1453.97 nm. On the lower wavelength side of the main transmission dip, eleven further transmission dips can be identified, the farthest is at 1447.28 nm. On the long wavelength side of the main transmission dip, no clearly separated transmission dips are

observable, although the reflectivity is quite high and gets to zero at about 1455.5 nm. In that area, it can be estimated that there are three transmission dips. In sum, 14 transmission dips are spread from 1447.28 nm to 1455.28 nm with an average spacing of 0.615 nm. In the transmission spectrum of the grating inscribed with 1 kJ/cm<sup>2</sup>, the transmission dips are more clearly compared to the sample fabricated with 0.5 kJ/cm<sup>2</sup>. The maximum reflectivity of 19.1 dB (~98.8 %) is at 1452.98 nm. The transmission dip with the shortest wavelength is at 1449.57 nm. The transmission dip with the longest wavelength is at 1455.37 nm. Overall, 13 transmission dips are recognizable with an average spacing of 0.45 nm.

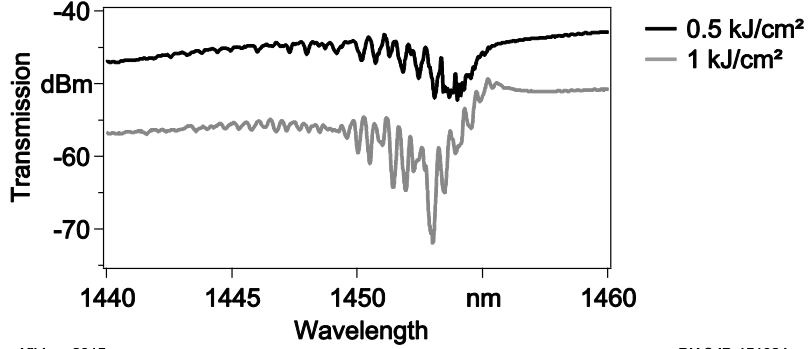


Fig. 2. Transmission spectra of both gratings inscribed in a POF which previously was loaded with oxygen. For better visibility, the reflection spectrum of the 0.5 kJ/cm<sup>2</sup> sample was shifted for 10 dBm.

#### 4. Discussion

It is clearly observable that the gratings in the oxygen-loaded fibers have a much higher reflectivity although the total fluence necessary for the inscription of the high reflectivity gratings is much lower. A measure for the efficiency of the inscription process is the comparison of the reflectivity in dB and the necessary total fluence, see table 1 for the actual calculated values.

It is estimated that the oxygen loading speeds up ongoing chemical reaction processes, which are induced by the UV photons inside the core of the POF. It is believed that the photosensitivity of the CYTOP polymer inside the core of the POF only depends on the dopants used for the forming of the graded index refractive index profile of the POF, as CYTOP itself only has C-C, C-O, and C-F bonds, which do not absorb in the far UV. The dopants contain C=C double bonds and benzene rings, which represent chromophores with absorption in the far UV (200 nm to 280 nm) Naritomi et al., 2004. However it has to be mentioned, that the authors only can give estimations about the ongoing processes. The refractive index increase by the gratings is very small and can be calculated from the grating parameters. The formula for this is

$$\Delta n = \frac{\lambda \tanh^{-1} \sqrt{\eta_g}}{\pi L \eta_c} \quad (1)$$

$\Delta n$	Refractive index difference
$\eta_g$	Reflectivity of the grating
$L$	Length of the grating along the fiber axis (1 cm)
$\lambda$	Central wavelength of the FBG (~ 1450 nm)

and is taken from Kogelnik, 1969. The formula was extended by the amount of energy guided inside the core of the fiber  $\eta_c$ , which can be calculated via the normalized frequency parameter  $V$ ,  $\eta_c = 1 - 1/V^2$ . In our case, the normalized frequency parameter is  $V=25.05$  and  $\eta_c=0.96$ . This is a necessary step as it is estimated, that the grating is only present inside the core and only can reflect the part of the light guided inside the core. The calculated refractive index differences for the four gratings are mentioned in table 1.

Table 1. Efficiency of grating inscription and refractive index difference of the gratings

Grating	(J/cm <sup>2</sup> ) / dB	$\Delta n$
No O <sub>2</sub> loading, 2.5 kJ/cm <sup>2</sup>	3125	0.000021
No O <sub>2</sub> loading, 5 kJ/cm <sup>2</sup>	1612.9	0.000043
O <sub>2</sub> loading, 0.5 kJ/cm <sup>2</sup>	53.19	0.000084
O <sub>2</sub> loading, 1.0 kJ/cm <sup>2</sup>	52.36	0.000140

## 5. Conclusion

The pre-treatment of the POF in an oxygen atmosphere with elevated temperature is a simple method to strongly gain the efficiency of FBG inscription in the investigated graded index perfluorinated polymer optical fiber. The reflectivity of the grating could be increased and at the same time, the inscription time was reduced by a factor of 10. This is a main step on the way to make FBG in POF commercially available for using these FBG as sensing elements for temperature or strain.

## Acknowledgement

The authors gratefully acknowledge the financial support of this work by the Federal State of Bremen in the framework of the ISIS (Integrated Solutions in Sensorial Structure Engineering) Sensorial Materials Scientific Centre ([www.isis.unibremen.de](http://www.isis.unibremen.de)) and by the Deutsche Forschungsgemeinschaft (DFG) under the contract number VO530/59-1.

## References

- Hill, K. O., Fujii, Y., Johnson, D. C., Kawasaki, B. S., 1978. Photosensitivity in Optical Fiber Waveguides: Application to Reflection Filter Fabrication. *Applied Physics Letters* 32, p. 647.
- Kang, D. H., Hong, C. S., Kim, C. G., 2006. Characteristics of Fiber Bragg Grating Sensors with Various Grating Lengths Embedded in Composite Materials. *Key Engineering Materials* 321-323, p. 152.
- Koerdt, M., Kibben, S., Hesselbach, J., Brauner, C., Herrmann, A. S., Vollertsen, F., Kroll, L., 2014. „Fabrication and characterization of Bragg gratings in a graded-index perfluorinated polymer optical fiber.” *Proceedings of the 2nd International Conference on System-Integrated Intelligence: Challenges for Product and Production Engineering*, Bremen, Germany, p. 133.
- Kogelnik, H., 1969. Coupled Wave Theory for Thick Hologram Gratings, *The Bell System Technical Journal* 48, p. 2909.
- Lacruz, A., Polis, M., Theodosiou, A., Koutsides, C., Kalli, K., 2015. Femtosecond laser inscribed Bragg gratings in low loss CYTOP polymer optical fiber. *Photonics Technology Letters, IEEE* 27 (7), p. 693.
- Liu, H., Peng, G., Chu, P., Koike, Y., Watanabe, Y., 2001. Photosensitivity in low-loss perfluoropolymer (cytop) fibre material. *Electronics Letters* 37 (6), p. 347.
- Morey, W. W., Meltz, G., Glenn, W. H., 1990. Fiber Optic Bragg Grating Sensors. *Proceedings of SPIE* 1169, p. 98.
- Naritomi, M., Murofushi, H., Nakashima, N., 2004. Dopants for a perfluorinated graded index polymer optical fiber. *Bulletin of the Chemical Society of Japan* 77 (11), p. 2121.
- Peng, G. D., Xiong, Z., Chu, P. L., 1999. Photosensitivity and gratings in dye-doped polymer optical fibers. *Optical Fiber Technology* 5 (2), p. 242.