

Highly sensitive LPG temperature sensor employing polyamic acid-coating

An Jia^{1,2}, Wang Yongjie^{1,2}, Li Fang^{1,2}, Liu Yuanhui^{1,2}, Peng Dandan^{1,2}

(1. State Key Laboratory of Transducer Technology, Institute of Semiconductors, Chinese Academy of Sciences, Beijing 100083, China;
2. College of Materials Science and Opto-Electronic Technology, University of Chinese Academy of Sciences, Beijing 100049, China)

Abstract: A highly sensitive long period fiber grating (LPG) temperature sensor was designed and demonstrated. Cladding of the LPG was etched and encapsulated in metal to maintain constant strain, then was sealed in a test tube containing polyamic acid (PAA), which had a relatively large thermo-optic coefficient as a temperature-sensitive material. The resonance wavelength shifted with temperature for different PAA refractive index values and different LPG cladding diameters were measured and discussed. The results show that the sensitivity of the LPG temperature sensor becomes higher when the refractive index of PAA becomes larger for a certain LPG of determined diameter. Additionally the sensitivity of the LPG temperature sensor becomes higher when the cladding diameter of the LPG decreases for a determined refractive index of PAA. A sensitivity of 1.26 nm/°C was achieved for this improved LPG temperature sensor, ten times greater than that of the sensor made of common LPG and 100 times greater than that made of common fiber Bragg grating (FBG). This new sensor exhibits good linearity of 99.80% at temperatures of 2–35 °C, which improves its potential application for biomedical measurement and ocean detection, where the temperature range of interest is close to room temperature.

Key words: long period fiber grating; temperature sensor; polyamic acid; sensitivity

CLC number: O439 **Document code:** A **DOI:** 10.3788/IRLA201847.0822002

采用聚酰胺酸层的高灵敏度长周期光纤光栅温度传感器

安佳^{1,2},王永杰^{1,2},李芳^{1,2},刘元辉^{1,2},彭丹丹^{1,2}

(1. 中国科学院半导体研究所 传感技术国家重点实验室,北京 100083;
2. 中国科学院大学 材料科学与光电技术学院,北京 100049)

摘要: 设计并论证了一种高灵敏度的长周期光纤光栅(LPG)温度传感器。将 LPG 的包层腐蚀,封装进金属外壳使 LPG 保持恒定拉力,再密封进盛满聚酰胺酸(PAA)的试管中,其中聚酰胺酸热光系数较大,作为感温材料。测试并讨论了使用不同折射率的聚酰胺酸与不同包层直径的 LPG 时,LPG 共振波长随温度变化的漂移。结果显示,对于一个直径已知的特定 LPG,当使用较大折射率的聚酰胺酸时,

收稿日期:2018-03-05; 修订日期:2018-04-03

基金项目:中国科学院战略性先导科技专项(XDA11040201)

作者简介:安佳(1992-),女,硕士生,主要从事光纤传感方面的研究。Email:anjia@semi.ac.cn

导师简介:王永杰(1979-),男,副研究员,博士,主要从事光纤传感技术与应用方面的研究。Email:wylj@semi.ac.cn

LPG 温度传感器的灵敏度变高。对于使用相同折射率聚酰胺酸的 LPG, 随着 LPG 的包层直径减小, LPG 温度传感器的灵敏度变高。实验制作的 LPG 温度传感器的灵敏度为 $1.26 \text{ nm}/^\circ\text{C}$, 约为普通 LPG 制作的传感器的 10 倍, 以及普通 FBG 制作的传感器的 100 倍。这种新传感器在 $2\sim 35^\circ\text{C}$ 线性度为 99.80%, 这提高了传感器的潜在应用, 特别是在生物医学检测, 海洋监测等这些温度范围接近室温的领域。

关键词: 长周期光纤光栅; 温度传感器; 聚酰胺酸; 灵敏度

0 Introduction

Accurate temperature measurement is important in various fields such as biomedicine, chemical industry, and ocean science. Over the past two decades, there has been significant interest in optical fiber grating temperature-sensing technology due to its small volume, electrical insulation, high sensitivity^[1], and its inexpensive fabrication. Fiber grating is classified into fiber Bragg grating (FBG) or long period fiber grating (LPG) according to the size of the grating period. Common FBG and LPG have temperature sensitivities of about $10 \text{ pm}/^\circ\text{C}$ and $100 \text{ pm}/^\circ\text{C}$, respectively. Strategies to increase the temperature sensitivity of FBG include etching, coating polymer, or binding to substrate with a large thermal expansion coefficient (TEC) to induce a stretching force on the grating region when the temperature changes, and these strategies allow increased sensitivity of 3–6 fold^[2–4]. Common ways to improve the temperature sensitivity of LPG are etching and coating polymer with high thermo-optic coefficient (dn/dT , TOC) which bases on the thermo-optic effect to improve the sensitivity because LPG is very sensitive to the surrounding refractive index^[5–10]. According to previous reports, coating LPG with poly-dimethylsiloxane orOrmocomp (TOC= $-1.3 - -4.5 \times 10^{-4}/^\circ\text{C}$) increased the sensitivities 2–200 times relative to those of LPG without coating, and the temperature sensing range decreased to only 5°C as the sensitivity increased to $24.6 \text{ nm}/^\circ\text{C}$ ^[7–9]. Another coating was similarly able to expand the working temperature range to 15°C with a sensitivity of $1.1 \text{ nm}/^\circ\text{C}$

using proper etching^[10]. However, although these methods can be used to produce an LPG temperature sensor with high sensitivity, the working temperature range in which the resonance wavelength varied linearly with temperature was narrow, limiting its application. Thus, it remains a challenge to achieve both high sensitivity and a wide temperature sensing range.

LPG is easily affected by strain and bending, so both sides of the grating are typically fixed in metal to maintain constant strain during experimentation^[6]. When coating a liquid polymer on LPG, a stainless steel tube full of liquid is used to glue the LPG inside^[9–10]. In the laboratory, silicone and ultraviolet glues are often used as glues to bond fiber and then cured at room temperature. However, this gluing procedure cannot guarantee a constant stretching state of LPG during measurement because the glue deforms with temperature changes. To avoid these problems and construct a sensor that is highly sensitive and also more stable, a new engineering strategy is required.

In this study, an LPG temperature sensor with high sensitivity and relatively wide temperature range was achieved by employing PAA-coated LPG technology. PAA is the precursor of polyimide which has a high TOC, $-3.72 \times 10^{-4}/^\circ\text{C}$ ^[11]. PAA has excellent quantities including anti-oxidation, innocuous and chemical corrosion resistance. Before coating with PAA, etching the cladding of the LPG is needed because common LPG has very low temperature sensitivity. The improvement in the packaging of the LPG also improved the sensor. LPG was fixed in the metal using a superior glue, 353 ND, which has a low

thermal expansion coefficient to ensure constant strain during the experiment. The glass test tube used to seal the LPG ensured long-term measurement in a complex environment, eliminated the interference of the refractive index and pressure of the environment and eliminated contamination of the measured sample. Overall, these improvements allowed preparation of a sensor that is more sensitive, more stable to environmental conditions, less prone to error due to sample contamination.

1 Principle of the LPG temperature sensor

LPG can induce a periodic perturbation when it is fabricated in the fiber. When light travels in the grating region, the wavelength of the light that satisfies the phase matching condition is coupled from core mode to cladding modes. The phase matching condition is given as follows:

$$\lambda_v = (n_{\text{eff}}^{\text{co}} - n_{\text{eff}}^{\text{cl},1v}) \Lambda \quad (1)$$

where λ_v is the wavelength of the light that coupled to the v th cladding mode, $n_{\text{eff}}^{\text{co}}$ and $n_{\text{eff}}^{\text{cl},1v}$ are the effective refractive indexes of the core mode and the v th cladding mode HE1.7. Λ is the grating period. $n_{\text{eff}}^{\text{cl},1v}$ varies with the external environment and $n_{\text{eff}}^{\text{co}}$ does not change with the external environment. As shown in Fig.1, the transmission spectrum of LPG inscribed in a standard Corning SMF-28 fiber was simulated.

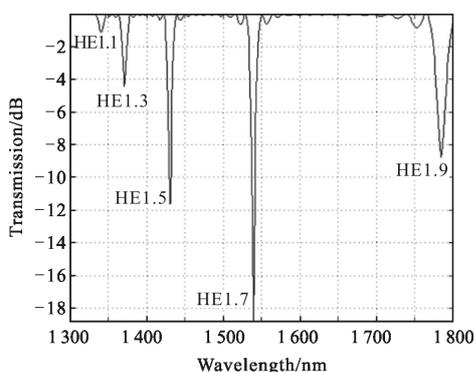


Fig.1 Transmission spectrum of LPG

The grating period is 450 μm and the length of the grating is 3.8 cm. As indicated in the figure, we can see that cladding mode HE1.7 has the strongest coupling.

LPG is very sensitive to temperature, external refractive index, strain, and bending^[12]. In our experiment, the external environment affects LPG through the test tube, allowing the exclusion of the affecting factors of the external refractive index. Both ends of the LPG are fixed on metal, so contributions of stain and bending can also be excluded, allowing us to focus on the temperature. After coating with high TOC material, the wavelength shift of LPG with external temperature is determined by the thermo-optic effect of the material, the thermal expansion effect of the silica, and the thermo-optic effect of the silica. The TEC of silica and the TOC of silica are $5.5 \times 10^{-7}/^\circ\text{C}$ and $8.6 \times 10^{-6}/^\circ\text{C}$, respectively, which is too low to achieve high temperature sensitivity. Thus, the thermo-optic effect of PAA is the major determinant. The temperature sensitivity of bare LPG fabricated in fiber loaded with hydrogen was previously measured as $64.5 \text{ pm}/^\circ\text{C}$ ^[6]. PAA-coated LPG fabricated with the same type of fiber showed sensitivities of $1.08\text{--}2.65 \text{ nm}/^\circ\text{C}$ in this study. The variation of the refractive index of PAA with external temperature is the domain factor as discussed below.

The wavelength shift of LPG with the refractive index of PAA can be expressed as follows:

$$\frac{\partial \lambda_v}{\partial n_3} = \frac{\partial (n_{\text{eff}}^{\text{co}} - n_{\text{eff}}^{\text{cl},1v})}{\partial n_3} \Lambda + (n_{\text{eff}}^{\text{co}} - n_{\text{eff}}^{\text{cl},1v}) \frac{\partial \Lambda}{\partial n_3} \quad (2)$$

$n_{\text{eff}}^{\text{co}}$ does not change with the external environment.

When Λ is determined, Eq.(2) can be written as:

$$\frac{\partial \lambda_v}{\partial n_3} = - \frac{\partial n_{\text{eff}}^{\text{cl},1v}}{\partial n_3} \Lambda = - \frac{n_3 U_\infty \Lambda}{n_2^3 k_3 (n_2^2 - n_3^2)^{\frac{3}{2}}} \quad (3)$$

where n_3 and n_2 are the refractive index of PAA and the cladding, respectively. U_∞ is the v th solution of the zero order of the first kind of Bessel function ($J_0(x)$), and is a constant related to v , the order of the cladding mode, and the larger the order, the higher its

value is. k is the wave number under vacuum. So, we can deduce that a large order of the cladding mode, thin cladding, and the refractive index of PAA near cladding could result in high sensitivity in refractive index sensing. As shown in Fig.3, the peak of mode HE1.7 varying with the refractive index of PAA was simulated. We can see that when n_3 increases from 1.43 to 1.45, the wavelength significantly shifts toward a shorter wavelength. To analyse this refractive index region in detail, the curve of the resonance wavelength of the refractive index at 1.420–1.460 was simulated, as shown in Fig.4. And the refractive

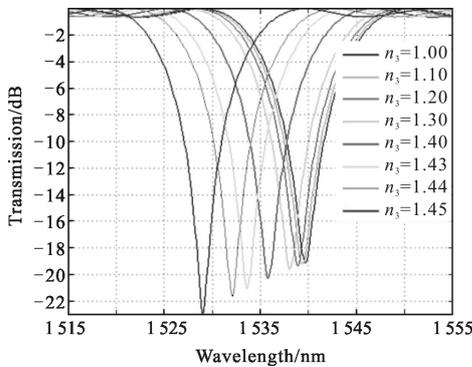


Fig.3 Transmission spectrum for different refractive index of PAA

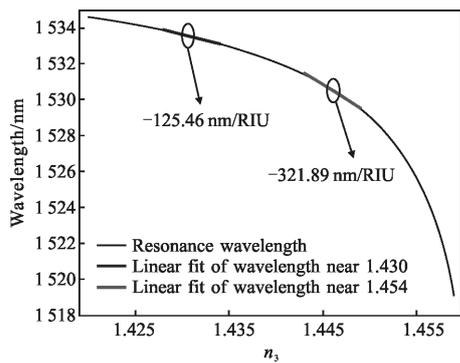


Fig.4 Resonance wavelengths as a function of the refractive index of PAA for HE1.7 mode

index region near 1.430 and 1.445 are analysed by linear fit with linearity of 99.75% and 99.28%, respectively. The slopes are indicated in the figure. We can see that as n_3 increases, the wavelength shift nonlinearly increases. Based on this simulation, in our experiment, the refractive index of PAA is

adjusted to 1.430–1.445 to provide a relatively high external refractive index environment for LPG.

2 Experiments and results

2.1 Etching and packaging of the LPG

LPG samples with a grating period of 287 μm and 300 μm were fabricated using Corning SMF-28 fibers loaded with hydrogen, using gratings 4.2 cm in length. HF acid of 20% concentration was diluted by the addition of deionized water at a volume ratio of 1:1. Figure 5 shows the experimental setup of the LPG etching process. LPG was immersed in a container of diluted HF acid. Both ends of LPG were fixed to remain straight during the experiment. A plastic holder supported the cover of the container to protect the fiber from being pressed. Light from SLED passed through LPG, and was received by an OSA (AQ6370C, Yokogawa Electric Corporation). After etching, the diameters of the three LPG samples were measured by optical microscope (BX51RF, OLYMPUS Corporation). The diameters changed from 127.37 μm to 121.63 μm , 120.34 μm , and 119.04 μm for the three samples.

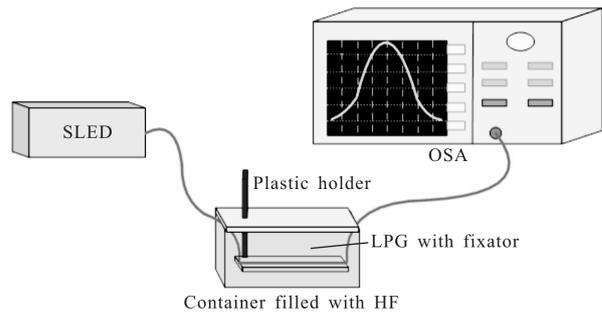


Fig.5 Experimental setup for etching

PAA solutions of different refractive index were prepared by the addition of acetone, and their values were read using an Abbe refractometer. The etched LPG samples were encapsulated in a hollow-out metal shell by glue to maintain constant strain during the experiment, and were then transferred to test tubes full of PAA. The packaged LPG temperature sensor is shown in Fig.6. One end of the

LPG is connected to a fiber patch cord, and the other is connected to a reflector.

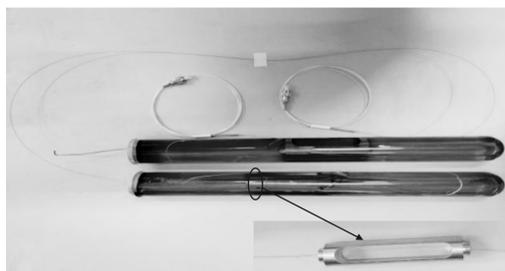


Fig.6 Packaged LPG temperature sensor

2.2 Testing of the packaged LPG temperature sensor

The LPG temperature sensor was immersed into a thermostatic waterbath. Its fiber patch cord was connected to the OSA and SLED by an optical coupler. From the measured transmission spectrum of the LPG temperature sensor, the background spectrum must be subtracted. For each LPG temperature sensor, only one peak in the transmission spectrum at 1 525–1 650 nm is recorded as the sensing peak. The temperature of the thermostatic waterbath was increased 1 °C every half hour from 2 °C to 35 °C. At each temperature, we recorded ten wavelengths of the peak in the five minutes after the temperature of thermostatic waterbath stabilized, and these readings were then averaged to determine the average final wavelength. The precise temperature was measured by a SBE 56 Temperature Logger (Sea –Bird Electronics) with an accuracy of 0.002 °C. This instrument was synchronized with the OSA, allowing the temperature data to be recorded each second for five minutes of measurement. The measured values were then averaged to determine the average final temperature. In this way, we obtained the average wavelength and the corresponding temperature at each temperature. LPG with a diameter of 121.63 μm and grating period of 287 μm was selected, and coated with PAA solutions of 1.443 and 1.445 refractive index, to produce two LPG temperature sensors. For simplicity, we designated these two LPG temperature sensors Sensor1 and Sensor2 corresponding to the PAA refractive index

values of 1.443 and 1.445, respectively. The peak shift of Sensor1 with temperature is shown in Fig.7. To simplify the figure, the peaks at even number temperatures are presented. There is some noise at some peaks that may arise from equipment factors like voltage instability of the SLED or the OSA, temperature instability in the waterbath, or vibrations arising from movement of the waterbath. The variation in the peak wavelengths of these two sensors with temperature were recorded and are shown in Fig.8. The sensitivities of Sensor1 and Sensor2 were determined as 2.48 nm/°C and 2.65 nm/°C, respectively. These two sensors exhibited linearity of 99.72% and 99.58%, respectively, over the temperature range of 12–30 °C. Use of the PAA coating increased the temperature sensitivity of the sensor and the sensitivity became higher when the refractive index of PAA became larger. With the improved sensitivity of the sensor, the linearity of the sensor decreased, especially at low temperatures as shown in Fig.8. This limitation

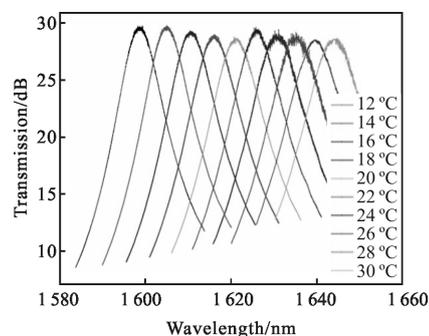


Fig.7 Curve of peak versus temperature of Sensor1 prepared with 1.443 refractive index PAA solution

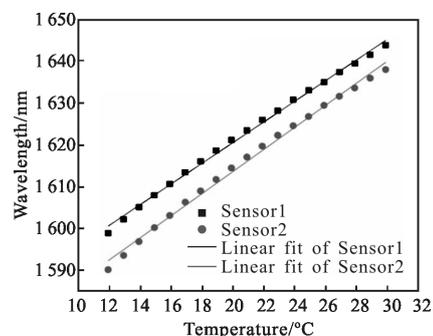


Fig.8 Wavelengths versus temperature of LPG temperature sensors prepared with different PAA solutions and different refractive index values

has prevented researchers from expanding the temperature range of sensors based on the thermo-optic effect. PAA has a larger refractive index at lower temperatures where the resonance wavelength of LPG increases nonlinearly. To solve this problem, we used PAA coatings of relatively low refractive index and we enlarged the etching depth to get a thinner cladding of LPG, which contributed to the decreased sensitivity.

Two LPG specimens were prepared with the same 1.430 refractive index PAA, but with LPG 120.34 μm and 119.04 μm in diameter, corresponding to Sensor3 and Sensor4, respectively. The peak shift of Sensor4 with temperature (four temperatures) is shown in Fig.9. As mentioned above, there is some noise in the data. The variation in the peak wavelengths of these two sensors with temperature were recorded and are shown in Fig.10. The sensitivities

of the twosensors were determined as 1.08 $\text{nm}/^\circ\text{C}$ and 1.26 $\text{nm}/^\circ\text{C}$, respectively, smaller values than those measured for Sensor1 and Sensor2. As predicted by the modification, these two sensors exhibit improved linearity, 99.81% and 99.80%, at temperatures of 2–35 $^\circ\text{C}$ by linear fit. Additionally, we can see that as the diameter of the LPG decreased, the sensitivity was higher, which supports the model that thin cladding leads to higher sensitivity of LPG. Of the four sensors, Sensor4 showed the best properties with sensitivity of 1.26 $\text{nm}/^\circ\text{C}$ and linearity of 99.80% at 2–35 $^\circ\text{C}$.

3 Conclusion

A highly sensitive LPG temperature sensor with relatively wide temperature range at low temperatures was designed and tested. PAA coating with a large thermo-optic coefficient and etching of LPG were used to enhance the temperature sensitivity. To achieve good linearity over a relatively wide temperature range with high sensitivity, materials were prepared with different refractive index of PAA solutions and different diameters of LPG. The results show that use of a relatively low refractive index PAA solution and a relatively large etching depth of LPG results in best performance. One sensor exhibited sensitivity of 1.26 $\text{nm}/^\circ\text{C}$ and linearity of 99.80% at 2–35 $^\circ\text{C}$, a significant improvement over the previous sensors and with proper packaging that are much closer to those needed for effective application.

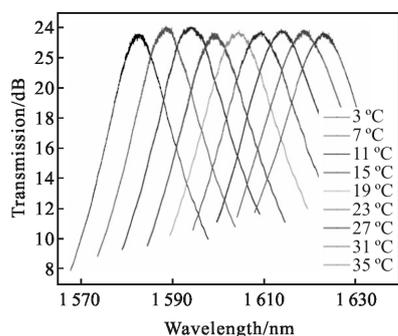


Fig.9 Curve of peak versus temperature of Sensor4 prepared with 1.430 PAA solution and LPG with 119.04 μm diameter

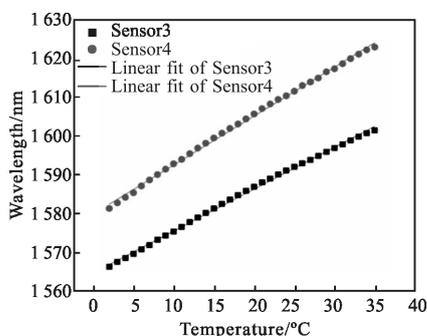


Fig.10 Wavelengths versus temperature of LPG temperature sensors prepared with different LPG and different diameters

References:

- [1] Liu Chao, Zhang Wen, Dong Mingli, et al. Dual-parameter sensing characteristics of long period fiber grating cascaded with fiber MZ structure fabricated by CO_2 laser [J]. *Infrared and Laser Engineering*, 2017, 46(9): 0922001. (in Chinese)
- [2] Zhou K, Chen X, Zhang L, et al. Implementation of optical chemsensors based on HF-etched fibre Bragg grating structures[J]. *Measurement Science & Technology*, 2006, 17(5): 1140.
- [3] Chen G, Zhang W, Webb D J, et al. Enhancing the

- sensitivity of poly (methyl methacrylate) based optical fiber Bragg grating temperature sensors [J]. *Optics Letters*, 2015, 40(17): 4046–4049.
- [4] Lee C H, Kim M K, Kim K T, et al. Enhanced temperature sensitivity of fiber Bragg grating temperature sensor using thermal expansion of copper tube [J]. *Microwave & Optical Technology Letters*, 2011, 53(7): 1669–1671.
- [5] Miao Fei, Zhang Ling, Feng Dejun, et al. Inscription of long period fiber gratings using 800 nm femtosecond laser [J]. *Optics and Precision Engineering*, 2012, 20 (4): 685–691. (in Chinese)
- [6] Wang Q, Liu G, Zhang X, et al. Highly sensitive long period fiber grating refractive index sensor based on thin cladding[C]//International Conference on Optoelectronics and Microelectronics. IEEE, 2016: 455–460.
- [7] Wang Q, Du C, Zhang J, et al. Sensitivity-enhanced temperature sensor based on PDMS-coated long period fiber grating[J]. *Optics Communications*, 2016, 377(20): 89–93.
- [8] Maheshwary S, Mehra R. Comparison of temperature sensors using polymer materials in long period fiber grating [C]// International Conference on Power Electronics, Intelligent Control and Energy Systems, IEEE, 2016: 1–6.
- [9] Tam H Y. Highly sensitive long -period fiber grating temperature sensor[C]//International Symposium on Photonics and Applications. International Society for Optics and Photonics, 2001, 4595: 282–286.
- [10] Zhao Q, Qu Y, Wang Y, et al. A novel temperature sensor based on a long period fiber grating with a unique double-cladding layer structure [C]//International Conference on Electronics and Optoelectronics, IEEE, 2011, 3 (3–4): V3–281–V3–283.
- [11] Qiu Fengxian, Zhou Yuming, Liu Juzheng, et al. Thermal-optical properties based on the fluorine-containing polyimide [J]. *Chinese Journal of Lasers*, 2006, 33 (2): 233–237. (in Chinese)
- [12] Liang Lili, Liu Mingsheng, Li Yan, et al. Solutions of strain and temperature cross-sensitivity of long period fiber grating temperature sensing [J]. *Infrared and Laser Engineering*, 2015, 44(3): 1020–1023. (in Chinese)