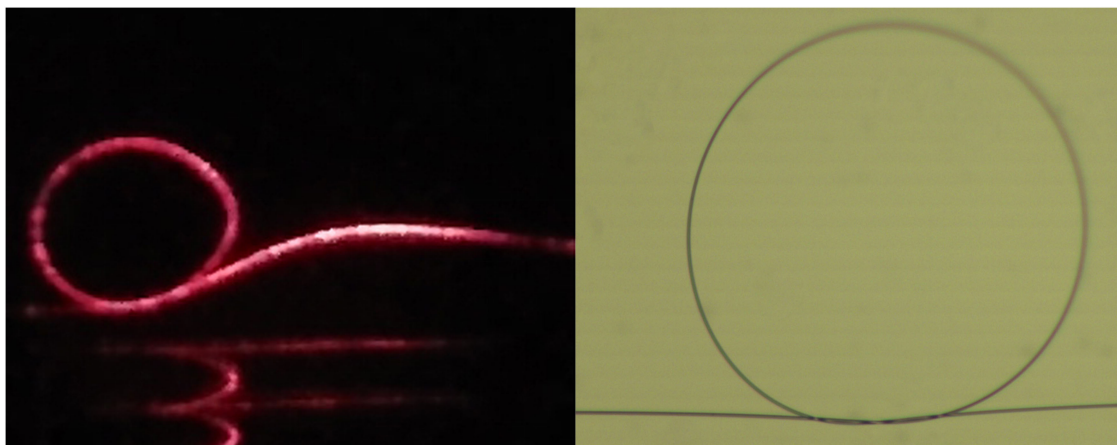


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A Highly-Sensitive Temperature-Sensor Based on a Microfiber Knot-Resonator Packaged in Polydimethylsiloxane

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Abstract: In this paper, a microfiber knot resonator (MKR) was encapsulated in polydimethylsiloxane (PDMS), and its capability to sense temperature was experimentally evaluated. A very high sensitivity of $-12 \text{ nm}/^\circ\text{C}$ was recorded for the MKR device, with a microfiber diameter of $2 \mu\text{m}$ and a ring diameter of $95 \mu\text{m}$. However, light-loss was significant in the small MKR. We attribute this to the low ratio of ring-length to microfiber-diameter. A stable temperature-sensing performance and sensitivity of negative $8.48 \text{ nm}/^\circ\text{C}$ were obtained for the MKRs with a microfiber diameter of $3 \mu\text{m}$ and a ring diameter of 3 mm . A MKR device with a non-uniform microfiber (in diameter) was also fabricated and studied. This compact and miniature temperature-probe will be a promising candidate for wearable devices.

Index Terms: Fiber non-linear optics, fiber optics systems, sensors.

1. Introduction

Optical fiber sensors attract the interest of researchers worldwide because of their electromagnetic resilience, chemical-corrosion resistance, high integration-ability, high sensitivity, remote-sensing ability, low price, and their suitability for mass production [1]–[3]. Essentially made of micro/nano-fiber, miniature fiber-sensors, with their fast response, low transmission-loss, and compact dimensions, currently dominate the sensing field [4], [5]. They are widely used to measure gas, humidity, temperature, stress, magnetic fields, refractive indexes, and other parameters [6]–[8]. Among the various device types, microfiber resonators, especially microfiber knot resonators (MKRs) [9], are promising candidates with the potential to monitor slight temperature fluctuations [10]. Compared with the tapered microfiber, whose sensing performance only depends on the change of the external refractive index, in addition MKR structure introduces another parameter, named ring length, to further improve the sensitivity. As early as 2009, Zeng et al. placed an MKR on a thin layer of polymer, which had a low refractive-index. The group measured a temperature sensitivity of $0.27 \text{ nm}/^\circ\text{C}$ during the reduction process from 28°C to 140°C , and a sensitivity of $-0.28 \text{ nm}/^\circ\text{C}$ during the cooling process from 135°C to 25°C [11]. Wu et al. compared the temperature-sensing performance of silica MKR with that of polymer MKR and used two magnesium difluoride (MgF_2)

sheets to improve their structural stability [12]. Based on their theoretical and experimental results, the temperature response of the MKR depended strongly on the diameter of the microfiber as well as the refractive index and thermal optical-coefficient of the external medium. The sensitivity increases for a thinner microfiber-diameter or a longer detection wavelength. When the diameter range of the optical fibers was between 2.3 and 3.91 μm , and the detection wavelength was 1550-1600 nm, the measurement sensitivity was 5.540 to 22.81 $\text{pm}/^\circ\text{C}$ [13]. Gomes used a bi-conical fiber Mach-Zehnder interferometer and MKR device, and found their temperature sensitivity to be 196 $\text{pm}/^\circ\text{C}$ and 25.5 $\text{pm}/^\circ\text{C}$ [14], respectively. Being packaged in polydimethylsiloxane (PDMS), the temperature sensitivity of the silica MKR was only -41.58 $\text{pm}/^\circ\text{C}$ [15]. Being limited by the thermo-optic and elastic-optic effect of silica, it is difficult to further improve the temperature sensitivity of a pure MKR, without introducing sensitizing materials [16]. To slow-down aging, avoid environmental pollution, and effectively improve their sensitivity, MKRs are usually coated with thin film or embedded within low refractive materials such as polytetrafluoroethylene (PTFE) and PDMS. Lim et al. investigated an MKR temperature-sensor and combined it with a Sagnac circular-reflector [17]. The temperature sensitivity was 20.6 $\text{pm}/^\circ\text{C}$, with good linearity for 30 to 130 $^\circ\text{C}$. By coating a layer of PDMS, with a thickness of 5 μm , on the surface of the MKR, which had a microfiber diameter of 1 μm , the temperature sensitivity for seawater could reach 0.197 $\text{nm}/^\circ\text{C}$ at 1550nm. The experimental results reveal that the sensitivity depends on the ratio of the external power around the microfiber and the bending- and absorption-losses [18]. The MKR device was inserted between two flexible PDMS graphene composite films and had a temperature sensitivity of 0.544 $\text{dB}/^\circ\text{C}$ in the range of 30-60 $^\circ\text{C}$ [19]. Our earlier studies indicate that, by being packaged in PDMS, the temperature sensitivity of the MKR can be increased from 187 $\text{pm}/^\circ\text{C}$ to more than 1 $\text{nm}/^\circ\text{C}$ [20], [21].

The MKR temperature sensors based on silica or polymer materials perform a fast response. In addition to the easy fabrication process and good sensing performance of the polymer MKR or polymer coated silica MKR, the working range will be seriously limited due to the low melting point (usually 200-400 $^\circ\text{C}$) of polymer materials. Materialized inertia, low optical loss and low refractive index are necessary for the polymer, especially for the microfiber resonators with unstable structures, such as microfiber coil resonator (MCR) and microfiber loop resonator (MLR) [22]. For example, MCRs must be twist on the PMMA rod to fabricate the stable temperature probe having the sensitivity of ~ 80 $\text{pm}/^\circ\text{C}$ [23]. The further functionalization and improvement of materials have been studied to explore some new sensors, such as the hydrophobic PDMS reported by Blank et al. The MKR sensor probe can be kept clean when it is used in liquid or high humidity environment [24]. The effect of external strain on temperature sensing performance also needs to be considered [25].

In this paper, by reducing the ring length of the microfiber and ensuring the uniformity of the microfiber diameter, a temperature sensitivity of 8.48 $\text{nm}/^\circ\text{C}$ is experimentally confirmed for a PDMS encapsulated MKR with a ring diameter of 3 mm.

2. Experimental Details

Microfibers were fabricated using a multi-functional fiber taper machine (IPCS-5000-ST, Idealphotonics Inc.) via a one-step high-temperature flame-scanning/stretching method, as shown in Fig. 1(a), the high temperature flame is supplied from a torch by burning the H_2 and O_2 mixture gas. The polymer coating layer removed fiber was fixed by two electrical fiber claps. This torch can be fixed or moving during a region to fabricate the microfiber with a short or long taper, respectively.

In computer software interface shown in Fig. 1(b), one can adjust the process parameters and monitor the transmission power. By controlling the flame heating range, fiber-stretching speed, and heating time, the smooth microfibers with a diameter of 2-49 μm , and a length of at least 1.5cm, can be prepared. Fig. 1(c) indicates that the MKR can be obtained by knotting the microfiber into a ring, which will be precisely controlled by stretching the two ends of the knotted microfiber to gradually reduce the ring diameter under the microscope system in Fig. 1(d), including the microscope (DMM-300C, Shanghai Caikon Optical Instrument Co., LTD.) and two 3D adjusting

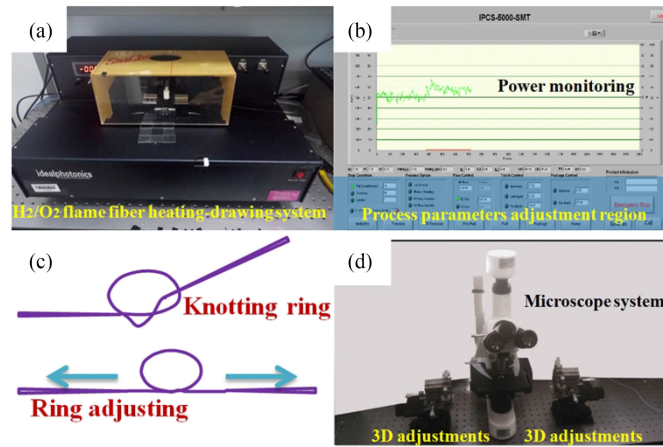


Fig. 1. Fabrication schematic of the microfiber and MKR structures (a) H_2/O_2 flame fiber heating-drawing and (b) process parameters control and power monitoring system; (c) MKR fabrication process through ring knotting and adjusting; (d) Ring micromanipulation system.

frames (APFP-XYZ, precision $<2 \mu\text{m}$, Zolix Instruments Co., LTD). The microscope image is recorded and sent to a computer to precisely measure the diameter of the ring and microfiber using a two-dimensional image measurement software. In this case, either the microfiber diameter or the ring length can be precisely controlled. To improve the structural stability and temperature response of MKR, the substrate was coated by the polymer film with a smaller refractive index compared to microfiber (1.46). In this paper, the packaging material is Dow Corning Sylgard 184 silicone rubber, known as PDMS, which contains the basic components and curing agent in the ratio 10:1. It can become solid after being heated for 90 minutes at 80°C in a constant-temperature oven. The cured transparent PDMS film has some elasticity, a low refractive index (1.4) and stable properties at -55 to 200°C . The high thermo-optic coefficient and thermo-expansion coefficient are beneficial to a highly-responsive temperature sensor.

In this work, the silica MKR structure was packaged by PDMS film with the thickness of $\sim 300 \mu\text{m}$ measured by a Vernier caliper. This thickness was controlled by sandwiched encapsulating the PDMS covered MKR by two glass slides and solidified in a thermostat at 80°C for 1 hour. Before that, each glass slide was spin pre-coated with the PDMS solution to support the MKR and meet the guidance conditions. To verify the quality of knot region and ensure that the structure is not damaged, this MKR was placed on a glass slide and lighted by a red laser at 632.8 nm (Fig. 2(a)). This MKR has the ring diameter of $\sim 3 \text{ mm}$ and the microfiber diameter of $\sim 3 \mu\text{m}$, as shown in Fig. 2(b). The MKR temperature sensor was calibrated using an amplified spontaneous emission light source (ASE output wavelength: $1520\text{--}1580 \text{ nm}$), an optical spectrum analyzer (OSA, Yokogawa AQ6370; detection range: $600\text{--}1700 \text{ nm}$; resolution: 20 pm) and the thermostat (Boxun BGZ-30; temperature range: room temperature to 250°C). The corresponding experiment schematic is shown in Fig. 2(c). The enlarge section of the knot region in Fig. 2(d) indicates the smooth surface and uniform diameter of $\sim 3 \mu\text{m}$ for the microfiber. The microfibers overlap in the coupling region of the MKR device to support the efficient coupling. [26]. The internal and outer transmission power along the microfiber can be expressed to be $P_1 = \int_0^\rho S_{z1} dA$, $P_2 = \int_a^\alpha S_{z2} dA$, where, $dA = r \cdot dr \cdot d\theta$ [26]. Where, S_{z1} and S_{z2} refer to the axial component of the Poynting vector outside and inside the microfiber, respectively. The transmission intensity can be estimated by the following equation based on the coupled mode theory [27]:

$$|T|^2 = (1 - \gamma) \frac{2\kappa_r [1 + \sin(\beta L)]}{1 + \kappa_r^2 + 2\kappa_r \sin(\beta L)} \quad (1)$$

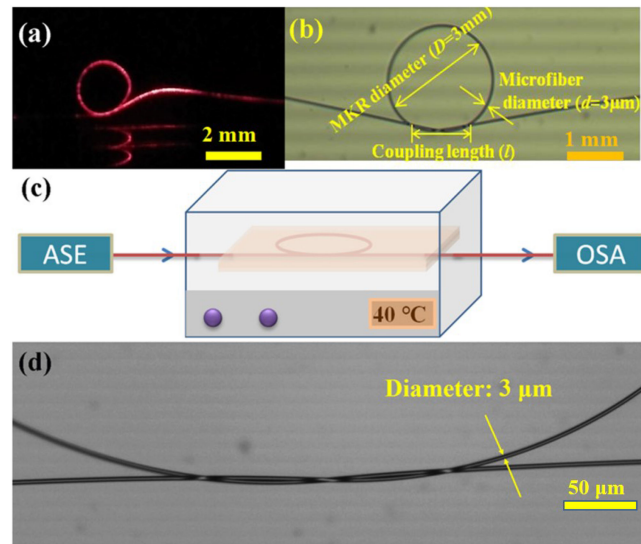


Fig. 2. Experimental schematic and MKR structures (a) MKR structure lighted by red laser; (b) MKR structure picture; (c) temperature calibration experimental schematic and (d) knot region microscope picture.

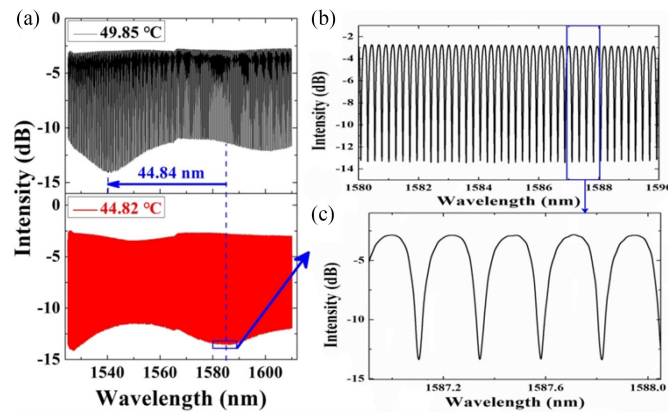


Fig. 3. Transmission spectra of the MKR device (a) Comparison of envelope spectra at different temperatures (49.85 °C vs 44.82 °C); Magnified envelope- (b) and interference-spectra (c).

where, γ refers to the coupling loss because of the knot region, depending by the coupling length l , and the microfiber diameter d . κ_{τ} represents the coupling coefficient, that is, the coupling intensity in different regions of the whole MKR structure. It depends on the microfiber quality (surface smooth, diameter uniform and knot ring length). The propagation constant β is determined by working wavelength λ and mode effective index n_{eff} : $\beta = 2\pi/\lambda Re(n_{eff})$. Due to the long length of the coupling region, a Mach-Zehnder interference effect will be produced, which leads to an envelope spectrum with a larger period in the transmission spectrum, see Fig. 3(a).

Here, the black and red envelope-spectra refer to the transmission spectra of the packaged PDMS MKR device at 49.85 °C and 44.82 °C, respectively. It was observed that, with decreasing temperature, the whole spectrum red-shifted. The shift distance of the peak position of the envelope spectrum was ~ 44.84 nm, showing a temperature-response of > 8 nm/°C. The period, also named free range, can be expressed as $F = C/n\Delta L$, C , n , ΔL refer to the light velocity in vacuum, refractive index of microfiber and the distances difference between the light in microfiber (low

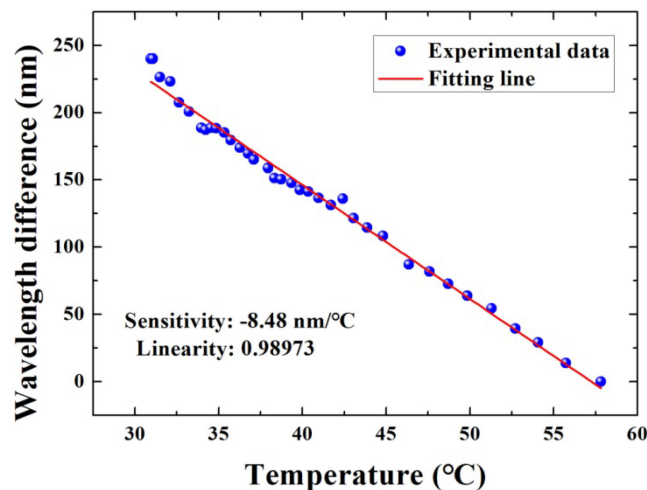


Fig. 4. Temperature-sensing characteristics of the PDMS-packaged MKR, with a microfiber diameter of $3\ \mu\text{m}$ and a ring diameter of 3 mm.

order mode) and the light at the surface of microfiber (high order mode or evanescent field). Due to the small, free spectral range (FSR: 239 pm) and high quality-factor (Q factor: 3×10^4), as indicated in Fig. 3(b-c), WGM can be used to monitor slight fluctuations in temperature accurately. In the experiment, the resonance-depth increased with decreasing temperature. Because the thermo-optic coefficient of the packaging material, PDMS, is negative, its refractive index increased with decreasing temperature, which caused the decrease of the refractive index difference between the inside and outside of the microfiber. Moreover, it reduced the constraining capability of the microfiber core for the optical field. The stronger evanescent field enabled more light to enter the ring and enhanced the resonance depth. In this study, we only used the envelope spectrum to determine the temperature change and verify its high sensitivity.

In practical applications, the large-range temperature measurement and high sensitivity as well as the accurate response to very small temperature fluctuations can be analyzed at the same time, using both the envelope and details of the transmission spectrum. This spectral superposition is similar to the Vernier effect caused by the superposition of two interference devices.

3. Results

To explore the temperature-sensing performance of the PDMS-packaged MKR device, the peak wavelength of the envelope spectrum was continuously tracked and recorded, when the temperature-probe was placed in the thermostat and the ambient temperature rose continuously from 28 °C to 60 °C. The temperature-sensing characteristic, with a linearity of 0.9873, is shown in Fig. 4.

The average sensitivity was calculated to be $-8.48\ \text{nm}/^\circ\text{C}$. Because of the high sensitivity and limitation by the operating wavelength of the light source, only an 8 °C temperature range could be determined by tracing one fixed envelope-peak within the whole spectral-range of 60 nm. In the experiment, when the special envelope-peak moved out of the spectral range, another peak, with a shorter wavelength, was selected. When approaching room temperature, the thermal response of the temperature probe becomes unstable due to the decrease of the temperature change rate of the incubator. In addition, the wavelength shift will have a significant deviation during the process of replacing the characteristic peak. The relative wavelength difference was recorded and used to calibrate the temperature-sensing performance for the whole temperature range.

The two main parameters affect the output spectrum of the MKR: the ring length (diameter) and the microfiber diameter in the coupling region. The smaller diameter of the microfiber has

TABLE 1
Sensing Performance of Four PDMS-Packaged MKRs with Different Parameters

Microfiber diameter (μm)	MKR diameter (μm)	Temperature range ($^{\circ}\text{C}$)	Sensitivity ($\text{nm}/^{\circ}\text{C}$)	Optical loss
5	12000	60-28	-2.36	19 %
3	12000	60-28	-4.66	13 %
3	3000	60-28	-8.48	25 %
2	95	40-34	-12	79 %
2	120	56-40	-3.5	37 %

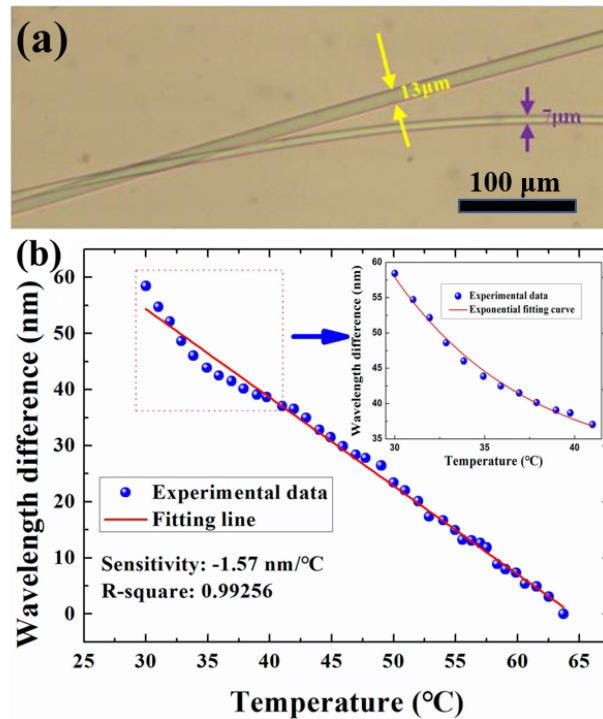


Fig. 5. Schematic of the coupling region microfibers (a) and temperature sensing characteristics (b) of the PDMS-packaged MKR with a microfiber of non-uniform diameter. Inset: Exponential fitting for temperature sensing performance during the low-temperature range (20-41 $^{\circ}\text{C}$).

the better mode-field characteristics, the higher coupling-efficiency and the higher temperature-sensitivity. Meanwhile, the smaller ring produces the greater relative reshape-change and the higher temperature-sensitivity. Table 1 shows the optical characteristic and sensing performance of several MKRs packaged in PDMS. The optical loss is caused by the microfiber bending and coupling. Once the ring diameter become smaller, it will be difficult to promise the production repeatability and linearity of the sensing curve for the MKR structure, that is why the sensitivity for the last sample is lower than the other bigger MKRs.

In addition to the effect of the ring diameter on the temperature sensitivity, a comparison of the results indicates that, when the ring diameter of the MKR is reduced from mm to μm , the operating-temperature-range changes substantially. The shortest range for the mm-scale ring is 32 $^{\circ}\text{C}$, as opposed to only 6 $^{\circ}\text{C}$, for the micrometer scale, which is mainly limited by the relative deformation and FSR. The ring-diameter affects both the FSR of the output spectrum and the measurement range of the MKR sensor. The shorter ring results in a wider FSR but a smaller temperature measurement range. High sensitivity and detection range need to be reasonably balanced for different applications.

TABLE 2
Sensing Performance Comparison Between Reported Works and Our Proposed Temperature Probe

Fiber structure	Function materials	Temperature range (°C)	Sensitivity (nm/°C)	Ref.
MCR	PMMA rod	27-28	~0.08	[23]
MKR	PDMS/Graphene	30-60	0.544dB/oC	[19]
MKR	PDMS film	20-70	0.187	[20]
		24-38	1.79	
MKR	PDMS film	24-38	1.408	[21]
		40-54	0.973	
		60-28	-8.48	
				This work

Due to the varying uniformity and different lengths of the microfiber diameter, as well as the different ring-lengths that are formed using the directly knot method, it is possible to obtain the non-uniform microfiber diameter of elliptically-shaped MKR-devices. For the MKR ring device, with a ring diameter of 7 mm, see Fig. 5(a), the microfiber diameter near the thinnest section was 4 μm , while the two microfibers near the coupling region have diameters of 13 μm and 7 μm , respectively. The temperature-sensing characteristic is shown in Fig. 5(b).

It suggests a corresponding temperature-sensitivity of $-1.57 \text{ nm}/^\circ\text{C}$ and a linear fitting with the R-square of 0.99256 for the whole temperature range. However, the temperature sensing performs an exponential curve characteristic during the low temperature range from 30 $^\circ\text{C}$ to 41 $^\circ\text{C}$, as shown in the Inset of Fig. 5(b). The ring length of the irregular MKR device is difficult to reduce further because of the poor flexibility of the thicker microfiber. However, the structural stability is better due to the large tension in the ring with the tighter combined microfibers of different diameters. Our recent (unpublished) experimental results revealed an even higher sensitivity and stability for the non-circular MKR-devices, which will be reported in future papers. The temperature sensing performance of this proposed MKR structure has been compared with that of similar works reported in recent years, as indicated in Table 2.

It is known that silica MKR can work in a wide temperature range, especially for the high temperature (700 $^\circ\text{C}$ in Ref. [11]), but its temperature sensitivity is limited to several dozens of $\mu\text{m}/^\circ\text{C}$ (Refs. [11]–[17]). The PDMS film packaged MKR has been demonstrated with a 50-200 folds higher sensitivity due to the excellent physical and chemical stability of PDMS, as well as its thermal expansion and thermal optical effect.

4. Conclusion

PDMS-packaged MKR-devices were fabricated and experimentally studied with respect to their temperature response. MKRs with different structural parameters (such as microfiber diameter and ring diameter) were fabricated and packaged, and their temperature-sensing characteristics were compared and analyzed. The results show that either the finer microfiber or the smaller ring-diameter contribute to the higher temperature-sensitivity. Either way, the operating range was reduced significantly. In practical applications, the diameter of either microfiber or ring should be carefully selected to meet the temperature-measurement requirements for specific environments. The temperature responses of the MKRs lie between 1.56-12 $\text{nm}/^\circ\text{C}$. The ring diameter could not be reduced without limits. It is mainly limited by the microfiber diameter, i.e., the microfiber flexibility. Both higher sensitivity and stability of the MKR device can be obtained by reducing the microfiber diameter. The microfiber, with its uneven diameter, can also be used to fabricate MKR devices but the stability is difficult to control. By optimizing the MKR's structural parameters, it is expected to improve both its stability and temperature-sensitivity further, so that it can be used to manufacture small, flexible, and all-fiber sensing-chips.

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