ULTRAFINE TEMPERATURE MEASUREMENT USING OPTICAL WHISPERING-GALLERY MODES

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ABSTRACT
This keynote provides a review of the research and development of optical whispering-gallery modes on the measurement and monitoring of temperature at ultrafine and micro scales.

INTRODUCTION
Optical whispering-gallery mode (WGM) resonators have seen increasing study over the past two decades in both physics and engineering [1-27]. The small mode volume confined at the surface of the resonators allows for strong interactions between resonators and the surrounding environment. Additionally, the potential for high quality factors (Q-factor) of WGM resonators allows for devices making use of WGM resonance to perform with high-precision and high-resolution. WGM devices have been developed in the fields of solid state physics and electrical engineering for the purposes of creating micro-disk lasers [1], studying quantum electrodynamic interactions [5, 6], and for the manipulation of optical signals [7, 8]. One of the most popular applications of WGM devices is for sensing. Sensors designed around WGM resonators are able to take full advantage of both their small mode volume and high quality factor. WGM sensing devices have been demonstrated in a number of different applications including: electric field [9], biological/chemical [10-16], humidity [17], pressure [21], and temperature sensing [18-20, 25-27]. In these applications WGM sensors demonstrate extremely high-resolution. Single molecule and individual RNA viruses [11, 12], as well as, pico-molar chemical residues [16] for biological/chemical sensing have been reported in the literature. Pressure sensors with milli-newton force [21] and electrical field sensors [9] with resolutions as high as 0.027pm/V m⁻¹ have been demonstrated. Temperature sensors capable of detecting milli-kelvin temperature changes have also been exhibited [18].

In this article we look to review the work done on developing accurate, on-chip, high-resolution temperature sensors. The work begins with studying resonators fabricated from fused silica. These resonators act as independent sensing devices, and are studied at both the room temperature and cryogenic temperature regimes. After these independent fused silica resonators are studied and understood, the development of sensors for on-chip temperature measurements took place. The goal of these experiments was to find suitable techniques for the fabrication of WGM sensors directly onto electrical components of interest and determine of these sensors could be used for real-time temperature monitoring. In order to perform these experiments innovative techniques capable of creating on-chip WGM resonators needed to be developed. After fabrication experiments were performed to understand the physics of these on-chip devices and to test their sensitivities as WGM temperature sensors in both the room temperature and cryogenic temperature regimes. Once the sensors sensitivity was discovered it was then possible to determine if those sensors were capable of on-chip dynamic temperature monitoring.

EXPERIMENTAL SETUP
Before discussing the results of the various experiments that took place, a brief discussion of the experimental setups used needs to take place. Though a number of different experiments will be reviewed in this paper, there were a number of common elements contained within all the experimental setups used. A 1516nm distributed feedback laser (NEL NLK1556STG) was used as the excitation source. The laser is controlled with a ILX Lightwave LCD-3724B laser controller and a function generator (Agilent 33220A). The standard practice for experiments conducting was to use a ramping function with frequency of 100Hz and amplitude of 3.5Vpp. The laser was injected into a fiber optic cable (Fiber Instruments Sales Inc. SMF28E+). The light was coupled into the WGM resonator via a tapered fiber technique [28], and the final signal was collected using a photo detector (Thorlabs PDA400). The signal from the photo detector was recorded either using a DAQ card and a LabVIEW program, or using a digital oscilloscope (Picoscope 3206B). The standard procedure...
for data acquisition was to record 10 WGM spectra per data point.

There are two additional components that varied depending on the experiment conducted. The first of these was the temperature cell. Different temperature cells were designed and used depending on if the WGM sensor was an independent device or an on-chip sensor. Different temperature cells were also used depending on the target temperature regime. For cryogenic experiments it was also necessary to use a nitrogen purging chamber to remove any frosting that would occur while cooling the system. The other component that varied throughout experiments was the WGM resonator. Resonators were fabricated as both independent and on-chip devices and out of both silica glass and polymer materials. Details of these two different components will be discussed in greater detail during the discussion of experimental results. Figure 1 shows a schematic of the experimental setup with all possible components used.

**FIG. 1.** Schematic of experimental setup [27]

**RESULTS AND DISCUSSION**

In this section we will discuss the results of a number of experiments performed on both independent sensors fabricated from pure silica as well as composite polymer sensors used for on-chip measurements.

**Independent fused silica sensors**

One of the simplest and most common material used for the fabrication of WGM resonators is fused silica. A simple technique for the fabrication of fused silica WGM resonators is through the melting of a fiber optic cable. A stripped and cleaned fiber optic cable is introduced into a heat source, in this case an oxy-hydrogen torch. The high temperatures, \( >1600^\circ C \), of the torch will melt the fiber optic and, under surface tension, the melted silica will take a spherical shape. Once the resonator is removed from the heat source the silica will return to a solid and the resulting silica microsphere can be used as a WGM resonator. Figure 2 shows a scanning electron microscope image of a fused silica resonator.

As can be seen in Fig. 2 fused silica resonators made with this technique remain attached to the fiber optic cable they are fabricated from. We refer to sensors fabricated in this way as independent sensors since they are not fabricated onto a region of interest. Two different working cells were used to test these independent sensors. Both made use of a tube, either plastic or copper, to surround the resonator/fiber taper coupling point. The purpose of this surrounding tube is to isolate the system from any air currents that may occur in the lab. The room temperature working cell had a base made from a large copper slab. This slab can be heated using a torch or electric heater, in turn, increasing the temperature within the working cell. In the case of the cryogenic working cell a long copper rod is attached to the base of the working cell. This copper rod can be placed within a liquid nitrogen (LN2) bath cooling the environment within the working cell to a temperature nearing 100K. In both the room temperature and cryogenic temperature experiments data is collected during the warming of the temperature cell. Data is collected simultaneously from both a thermocouple located in close proximity to the WGM coupling point and the photodetector monitoring the WGM spectra. Figure 3 shows sample spectra, with a trackable resonance peak, at different temperatures obtained during a cryogenic temperature experiments.

Figure 4 shows the wavelength shifts versus temperature changes of 5 different diameter resonators tested from 110K to nearly 300K. Artificial vertical shifts are added to the figure so that all 5 resonators can be clearly distinguished. This behavior can be explained by the equation:

\[
\frac{d\lambda}{dT} = \lambda \left( \frac{1}{n} \frac{dn}{dT} + \frac{1}{D} \frac{dD}{dT} \right) = \lambda \left( \frac{1}{n} \alpha + \beta \right)
\]

In this equation \( T \) is temperature, \( \lambda \) is the wavelength of the injection laser, \( n \) is the index of refraction, \( D \) is the resonator diameter, \( \alpha \) is the thermal optical coefficient of the resonator material, and \( \beta \) is the thermal expansion coefficient of the resonator material. It can be observed in Fig. 4 that the...
The curvature of this data is very similar regardless of resonator diameter.

\[ \frac{d\lambda}{dT} = -(4.48 \pm 0.30) \times 10^{-5}T^2 + (5.31 \pm 0.12) \times 10^{-2}T - (1.08 \pm 0.12) \]

The overall quality of these fused silica microresonators was \(>10^7\). This high Q-factor allows for the overall resolution of these sensors to be on the order of micro-kelvin.

### Composite silica and PDMS sensors

The experiments performed in the previous section are useful to verify the physical model used to understand resonance shifts based on temperature changes, but in order to take full advantage of the unique qualities of WGM temperature sensors it was necessary to develop techniques for the direct fabrication of a WGM resonator onto a region of interest. The key factor in the creation of these new techniques is that the must not require any temperatures or chemical processes that would adversely affect the region of interest. Two effective techniques were found in the lab. The first of these techniques made use of polydimethylsiloxane (PDMS). PDMS is sold in a base agent and curing agent that when unmixed are liquids at room temperature. Once mixed, a 10:1 base to curing agent ratio was used, cross-linking will take place and the PDMS will solidify. At room temperature it can take between 24-48 hours for the polymer to cure, but this time can be substantially reduced by increasing the PDMS temperature. A temperature of around 70°C will reduce the curing time to a matter of minutes.

Once the PDMS is mixed it is placed in a vacuum to remove any air bubbles that occurred during mixing. If these air bubbles are not removed it is unlikely the PDMS resonator will be able to excite WGM resonance. The vacuumed PDMS can now be applied to a region of interest. In this case we used a 127\(\mu\)m diameter nichrome wire as the region of interest. This wire represents an electrical component to be monitored. To fabricate the PDMS resonators onto the nichrome wire a stripped and cleaned fiber optic is dipped into the PDMS and removed so that a droplet of PDMS is left on the tip of the fiber optic. This drop can than be transferred to the nichrome wire by placing it into contact with the nichrome. Using this method, droplets can be placed on the wire with diameters as small as 200\(\mu\)m. It was found that if a current of 300mA was run through the wire it would cause droplets <500\(\mu\)m to cure in place. Droplets >500\(\mu\)m would slide down the wire with the current running through it and leave a thin cylindrical PDMS coating, 10-15\(\mu\)m in thickness, along the wire. This process could be repeated to increase the thickness of this coating. These coatings can be seen in Fig. 4.

A second method was developed in an effort to form glass based resonators in order to compare the potential quality of polymer and glass on-chip resonators. In this method high temperature glasses such as borosilicate or those found in...
optical fibers, such as the one used to fabricate the fiber taper, cannot be used. The temperatures required to cause fusion of these glasses would destroy the nichrome wire.

To resolve this problem optical fibers were fabricated in the lab from low temperature glasses such as BK7 and flint glass. By running a current of approximately 1.5A through the wire, the nichrome would get hot enough to melt these glass fibers attaching them to the wire and eventually fusing them into ellipsoid shell resonators. Under optical microscopy these resonators look the same as though seen in Fig. 4(a).

With resonators fabricated the next step was to determine the resonator’s room temperature sensitivity. A new working cell based on the design of the previously used cell was constructed. This cell consisted of a copper tube to enclose the coupling point. This copper tube was wrapped with a separate nichrome wire that could be used to control the internal temperature. Using this heating method the internal cell temperature could be raised from room temperature to 10°C above room temperature at a rate of 1°C every 3-5 minutes. Two clamps are attached to the mount holding the copper tube in place, these mounts are positioned to hold the on-chip electrical component in place so that it can be aligned with the fiber taper. A thermocouple is also placed inside this cell a few millimeters from the coupling point to monitor the local temperature. Figure 5 shows the sensitivities of a number of different diameter PDMS resonators. Table 1 shows the the sensitivity and linear correlation coefficient of different PDMS and glass resonators fabricated onto nichrome wire [26].

An examination of Fig. 5 demonstrates that the sensitivity of these on-chip resonators depends on diameter. Both negative and positive sensitives can be seen depending on resonator thickness, and an asymptotic relationship appears to exists as the overall resonator size increases. In order to explain the behavior Eq. 1 needs to be adapted. In previous works found in the literature a diameter dependent sensitivity was seen for composite WGM resonators that consisted of a fused silica core with PDMS coating. In this case this behavior is explained by adapting an effective index of refraction and thermal optical effect based on the energy fraction of the WGM mode located in each of the resonator’s layers. In the case of the work contained here, this explanation does not work. Not only are the PDMS coatings used in these experiments thick enough that they would contain roughly 100% of the energy fraction. Our system also consists of a dielectric coating on conducting core, and since the conducting core cannot support optical waves, an effective thermal optical coefficient and index of refraction would not make sense to use in this system. Instead an effective thermal expansion coefficient must be adopted. In this case Eq. 1 becomes:

\[
\frac{dx}{dT} = \lambda \left( \frac{1}{n} \alpha + \beta_{\text{eff}} \right)
\]

(3)

where \(\beta_{\text{eff}}\) is found using the equation [29]:

\[
\beta_{\text{eff}} = \beta_2 - V_1 \frac{(1 + \nu_2)/2E_2}{[(1 + \nu_2)/2E_2] + [(1 - 2\nu_1)/E_1]} \left( \beta_2 - \beta_1 \right)
\]

(4)

In this equation subscript 1 and 2 represent the core and coating layer respectively. \(V_i\) is the volume fraction of the core within the coating layer. \(E\) and \(\nu\) are the Young’s modulus and Poisson ratio respectively. The volume fraction contained within Eq. 3 along with the fact that PDMS has a negative
thermal optical coefficient explains the sensitivity behavior observed in Fig. 5.

<table>
<thead>
<tr>
<th>Resonator Diameter (μm)</th>
<th>Sensitivity (pm/K)</th>
<th>Correlation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>172 (PDMS cyl. annulus)</td>
<td>-65.7 ± 3.6</td>
<td>0.999 81</td>
</tr>
<tr>
<td>194 (PDMS cyl. annulus)</td>
<td>16.0 ± 0.9</td>
<td>0.998 37</td>
</tr>
<tr>
<td>295 (PDMS elp. shell)</td>
<td>82.0 ± 4.5</td>
<td>0.999 30</td>
</tr>
<tr>
<td>345 (PDMS elp. shell)</td>
<td>87.4 ± 4.8</td>
<td>0.999 94</td>
</tr>
<tr>
<td>360 (PDMS elp. shell)</td>
<td>93.2 ± 5.1</td>
<td>0.999 90</td>
</tr>
<tr>
<td>400 (Flint Glass elp. shell)</td>
<td>11.9 ± 5.7</td>
<td>0.996 62</td>
</tr>
<tr>
<td>512 (PDMS elp. shell)</td>
<td>107.8 ± 5.9</td>
<td>0.999 79</td>
</tr>
</tbody>
</table>

With the sensitivity of the resonators determined it was then possible to test the resonators for direct on chip temperature monitoring. In order to perform this test two electrical leads were attached to the nichrome wire with the WGM sensor fabricated to it. A small current ranging from 0.03A to 0.05A was run through the wire. Once the current was turned on data from both the WGM sensor and thermocouple were acquired roughly every 2 seconds. The system could then be cooled by turning off the current and letting it return to equilibrium with the local environment. Figure 6 illustrates the results of such an experiment.

The data displayed in Fig. 6 shows the temperature determined from the thermal couple inside the chamber as well as from the calibrated shifts in the WGM spectra. A theoretical transient heating curve is also plotted based on the equation:

\[ \frac{\pi}{4} \left[ D_1^2 \rho_1 C_1 + (D_2^2 - D_1^2) \rho_2 C_2 \right] \frac{dT}{dt} = \frac{4T^2 \sigma_e}{\pi D_1^2} - \pi D_2 h(T - T_\infty) \]  

where \( D \) is diameter, \( C \) is specific heat \( I \) is current, \( \sigma_e \) is the nichrome resistivity, \( \rho \) density, and \( h \) is the heat transfer coefficient found using a correlation for a horizontal cylinder in free convection. The subscripts 1 and 2 are again used to represent the core and coating layers respectively. It is clear from Fig. 6 that in both the heating and cooling cases the temperature measured by the WGM sensors matches much better with the theoretical curve than the temperature measured by the thermocouple. Most likely, this is due to the small size of the electric component used. The thermocouple bead has roughly the same size diameter as the nichrome wire, therefore it is impossible for the two objects to be in perfect contact where as the PDMS WGM resonator surrounds the nichrome wire giving it excellent thermal contact, but it is also thin enough to not overly effect the systems temperature in the way an insulator might [26]. Lastly, it should be noted that the PDMS resonators tended towards a Q-factor of \( 10^4 \) to \( 10^6 \) giving the system a potential resolution on the order to 0.01K-0.001K.

Having demonstrated the effectiveness of on-chip WGM temperature sensors at room temperature, it was of interest to see if they would also be effective at cryogenic temperatures. At the time of the experiments, to the authors knowledge, PDMS had not been tested as a WGM resonator at cryogenic temperatures. Therefore, before any direct temperature monitoring could be done it first needed to be determined if a PDMS resonators would work at these temperatures. A purging chamber and cryogenic working cell were designed for these experiments. The cryogenic working cell was capable of reaching temperature below 100K by using a liquid nitrogen reservoir [27]. Figure 7 shows the WGM spectra of a PDMS sensor at both room and cryogenic temperatures. It is clear from the figure that PDMS can support resonance at low temperatures, but that there is a drop in quality can most likely be explained by a change in the intrinsic properties of PDMS as it is cooled to these low temperatures. A number of different diameter sensors were tested at cryogenic temperatures. To determine the resonators sensitivity, the system was cooled by filling the LN2 reservoir. After about 10 minutes the LN2 reservoir would become
depleted and the system would begin to naturally warm. Both thermocouple and WGM spectra data could be collected at regular intervals during this warming period to determine the resonators sensitivity. The calibration curves can be seen in Fig. 8 and the sensitivities and linear correlations can be found in Table 2.

![Fig. 7: Room temperature and cryogenic temperature spectra from a PDMS ellipsoid shell resonator [27]](image)

![Fig. 8: Calibration curves for 4 PDMS ellipsoid shell resonators [27]](image)

<table>
<thead>
<tr>
<th>Shell Diameter (μm)</th>
<th>Sensitivity (nm K⁻¹)</th>
<th>Correlation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>190</td>
<td>0.032</td>
<td>0.9974</td>
</tr>
<tr>
<td>542</td>
<td>0.025</td>
<td>0.9964</td>
</tr>
<tr>
<td>562</td>
<td>0.023</td>
<td>0.9937</td>
</tr>
<tr>
<td>639</td>
<td>0.021</td>
<td>0.9985</td>
</tr>
</tbody>
</table>

Examination of the cryogenic temperature sensitivities shows that a different relationship exists between diameter and sensitivity in the cryogenic temperature regime than did at room temperature. At room temperature thin coatings had a negative correlation and the value of the sensitivity increased with diameter. In the cryogenic temperature regime thin coatings have a positive sensitivity and the value of sensitivity decreases with increase in diameter. Unfortunately, the material properties of PDMS and nichrome at cryogenic temperature were not available in the literature and so Eqs. 3 and 4 cannot be used to determine the theoretical diameter based sensitivity of the PDMS resonator in this temperature regime. Although using the experimental data found we can make inferences about the potential cryogenic properties of the materials used. The fact that the sensitivity is always positive regardless of the diameter implies that either the thermal optical coefficient of PDMS is positive at these temperatures or that thermal expansion term dominates at these temperatures. Additionally, the sensitivity decreasing with increasing diameter implies that $\beta_{nichrome} > \beta_{PDMS}$ in the cryogenic regime. Note that at room temperature the opposite is true.

Having calibrated the sensors, it was then possible to test them for on-chip temperature monitoring at cryogenic temperatures. This was done in the same way as at room temperature. A small current was introduced into the nichrome wire so that it would undergo Joule heating. The major difference here was that there was only a small window to conduct the experiment in. Once the LN2 reservoir is depleted the system will naturally warm, therefore any experiment to test for on-chip sensing should be conducted before the system begins to naturally warm, so as to isolate the Joule heating effect. Figure 9 shows the results of such an experiment. Without the material properties of PDMS it is not possible to to a full transient theoretical analysis instead a simple steady-state analysis can be performed. Table 3 shows both the experimental and analytically determined values for the steady state temperature, based on a simple energy balance of the system. The results show excellent agreement between experimental and theoretical results.
CONCLUSIONS

A lot of work in our research group has been done related to the use of WGM resonators as potential temperature sensors. Fused silica resonators have been demonstrated capable of high resolution temperature sensing from room temperature to cryogenic temperatures. These simple devices can easily achieve temperature resolutions on the order of milli-Kelvin. PDMS based WGM sensors have the added advantage of being capable of direct on-chip fabrication. On-chip PDMS sensors are not only capable of resolutions of about 10 milli-Kelvin, but they can also be used for real time temperature monitoring of a region of interest. These devices out performed common temperature monitors such as thermocouples in both resolution and accuracy for on-chip measurements. Additionally, PDMS was demonstrated capable of supporting WGM resonance at cryogenic temperatures. This opens up a number of new polymer applications, including high-accuracy real-time temperature measurement.

ACKNOWLEDGMENTS

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REFERENCES


TABLE 3. Steady-state analysis of cryogenic system undergoing Joule heating

<table>
<thead>
<tr>
<th>Current (A)</th>
<th>Experimental ΔT (K) ± Error</th>
<th>Analytical ΔT (K) ± Error</th>
<th>Difference (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0375</td>
<td>4.11 ± 0.12</td>
<td>3.84 ± 0.52</td>
<td>0.27</td>
</tr>
<tr>
<td>0.043</td>
<td>4.83 ± 0.13</td>
<td>4.86 ± 0.64</td>
<td>0.03</td>
</tr>
<tr>
<td>0.052</td>
<td>6.79 ± 0.14</td>
<td>6.76 ± 0.86</td>
<td>0.03</td>
</tr>
</tbody>
</table>

FIG. 9. Transient heating curve for three different currents at cryogenic temperatures [27]