Polymer-Coated Fiber Bragg Grating Sensors for Simultaneous Monitoring of Soluble Analytes and Temperature

Ping Lu, Liqiu Men, and Qiying Chen

***Abstract*—A new fiber-optic sensor for simultaneous measurement of water-soluble analytes and temperature with polymer-coated fiber Bragg gratings (FBGs) is proposed. As an application of the approach, simultaneous monitoring of the concentration of sugar or potassium chloride (KCl) and temperature has been achieved. Changes in these environmental parameters result in different extents of either red- or blue-shifts of the Bragg resonance wavelengths of the gratings. It has been found that polyimide-coated FBG responds to variations of both temperature and concentrations of soluble analytes, while acrylate-coated FBG is sensitive to environmental temperature only. The experimental results showed that the temperature sensitivity of the acrylate-coated FBG, temperature, sugar, and KCl concentration sensitivities of the polyimide-coated FBG are 0.0102 nm/°C, 0.0094 nm/°C, 0.0012 nm/°Bx, and 0.0126 nm/M, respectively. The sensing mechanism of the polyimide-coated FBG lies in the hygroscopic properties of the polyimide coating, which result in the change of the strain of the fiber and, thus, the optical properties of the grating. Since the sensor detects the analytes that swell the polyimide coating and different analytes induce different swelling effects, the sensor can detect different analytes without prior knowledge once a calibration curve is developed.**

***Index Terms*—Chemical sensor, fiber Bragg grating (FBG), polymer, temperature sensor.**

I. Introduction

FIBER BRAGG GRATING (FBG) sensors have attracted considerable attention recently [1], [2]. Compared with conventional electric sensors, FBG sensors exhibit many unique advantages, for instance, immunity to electromagnetic interference, compact size, low cost, compatibility with the existing fiber-optic systems, and possibility of distributed measurement over a long distance. FBG sensors have been reported to be effective in monitoring various environmental parameters, including temperature, strain, tilt, torsion, flow, humidity, chemicals, gas, and structural health monitoring. For environmental conservation, food industries, and biomedical applications, *in situ* monitoring of physical, chemical, and biological parameters in water is of great importance, in which versatile sensors are highly demanded to achieve simultaneous measurement of temperature and different chemical parameters, for example, concentrations of water-soluble analytes including sugar, metal ions, metal compounds, and chemical traces. In practical applications, it is necessary for the sensor system to be capable of measuring more than one of these parameters.

This paper reports a new scheme constructed with two different polymer-coated FBGs to achieve simultaneous measurement of temperature and concentrations of water-soluble analytes such as sugar and potassium chloride (KCl). Previous research on the use of FBG sensors to measure the concentrations of soluble analytes adopted etched fibers to measure the refractive indices of the materials [3]–[7]. Bennion *et al.* proposed a dual-parameter optical sensor based on a hybrid long-period grating—FBG structure in a D fiber to deduce the concentration of an aqueous sugar solution from the measurement of its refractive index [3]–[5]. Iadicicco *et al.* reported the use of nonuniform thinned FBGs for self temperature compensated refractive index measurement, while the sugar concentration measurement was carried out in nonisothermal condition [6], [7]. However, in these techniques, part of the cladding layer was removed by wet chemical etching in a buffered hydrofluoric acid (HF) solution, which made the sensors to be more fragile and susceptible to damage by external force. In our approach, two polymer materials, i.e., polyimide and acrylate, are used as the fiber coating materials. Polyimide and acrylate have excellent strength and can resist breakage, which are innocuity and harmless to human-beings and the environment. On the other hand, polyimide polymers are hygroscopic and swell in aqueous media as the water molecules migrate into them [8]. In order to achieve simultaneous measurement of temperature and concentrations of water-soluble analytes, our sensor system consisted of two FBGs, in which an acrylate-coated FBG is not sensitive to sugar or KCl concentration and functions as a temperature sensor, while a polyimide-coated FBG is sensitive to sugar or KCl concentration and acts as a chemical sensor. The experimental results indicate that other water-soluble analytes can be detected by this sensor system as well.

II. Theory

The Bragg resonance wavelength of a grating, which is the center wavelength of the light back-reflected from the Bragg

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P. Lu and L. Men are with the Department of Physics and Physical Oceanography, Memorial University of Newfoundland, St. John's, NL A1B 3X7, Canada.

Q. Chen is with the Department of Physics and Physical Oceanography, Memorial University of Newfoundland, St. John's, NL A1B 3X7, Canada, and also with the Faculty of Engineering and Applied Science, Memorial University of Newfoundland, St. John's, NL A1B 3X5, Canada (e-mail: qiyingc@mun.ca).

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grating, depends on both the effective refractive index $n_{\rm eff}$ of the fiber core and the periodic spacing between the grating planes Λ. The effective refractive index and the periodicity of the grating are susceptible to the changes in temperature and strain. For a polymer-coated FBG, i.e., polyimide-coated FBG, subject to changes in temperature and water concentration of the environment (relative humidity), the shift in the Bragg grating center wavelength is given by [8]

$$\begin{aligned}\frac{\Delta\lambda_B}{\lambda_B} &= S_T\Delta T + S_{RH}\Delta RH \\ &= \left[\alpha_{cf} - \hat{P}_e(\alpha_{cf} - \alpha_f) + \zeta\right]\Delta T \\ &\quad + \left[\beta_{cf} - \hat{P}_e(\beta_{cf} - \beta_f)\right]\Delta RH \end{aligned} \tag{1}$$

where S_T and S_{RH} are the sensitivities of temperature and relative humidity, respectively. ΔT and ΔRH are the changes in temperature and relative humidity accordingly. The subscript stands for bare fiber $(i = f)$ and coated fiber $(i = cf)$. α_i is the thermal longitudinal expansion coefficient, and β_i is the hygroscopic longitudinal expansion coefficient, which is zero for a bare fiber [8]. ζ is the thermo-optic coefficient of the fiber core, and $\hat{P}_e$ is the effective photoelastic coefficient of the coated fiber.

The temperature sensitivity of the grating, S_T, is

$$S_T = \alpha_{cf} - \hat{P}_e(\alpha_{cf} - \alpha_f) + \zeta. \tag{2}$$

Since the hygroscopic longitudinal expansion coefficient of the bare fiber β_f is zero, the relative humidity sensitivity of the sensor, S_{RH}, can be expressed as

$$S_{RH} = (1 - \hat{P}_e)\beta_{cf}. \tag{3}$$

The polyimide coating swells after absorbing water, which will stretch the fiber. The relative humidity around the polyimide-coated FBG can alter the longitudinal length of the grating area and hence the water concentration in the polyimide coating. The water accumulation and leakage in the coating layer of the FBG is a diffusion process, which can be described by Fick's second law

$$\frac{\partial c}{\partial t} = D\nabla^2 c \tag{4}$$

where c is the water concentration, D is the constant diffusion coefficient, and t is the diffusion time.

For radial diffusion in a cylinder as illustrated in Fig. 1, the general diffusion equation shown in the above equation can be expressed in cylindrical coordinates

$$\frac{\partial c}{\partial t} = D\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial c}{\partial r}\right). \tag{5}$$

The boundary condition for the diffusion mass entering the polyimide coating at the position r along the direction of diffusion at time t is

$$\begin{aligned} c(a < r < b, t = 0) &= C_0 \\ c(r = b, t \geq 0) &= C_{\rm ext} \\ \frac{\partial c}{\partial r}(r = a, t \geq 0) &= 0 \end{aligned} \tag{6}$$

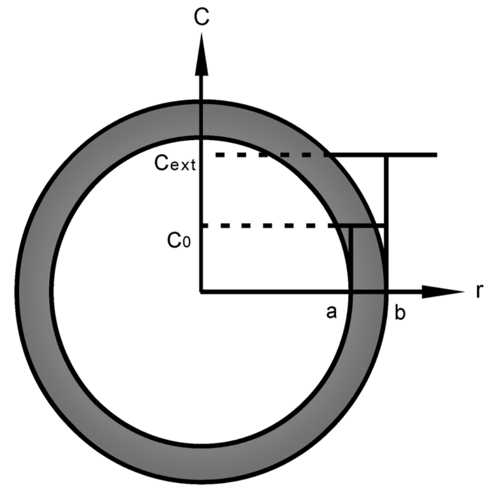


Fig. 1. Schematic illustration of the distribution of the water concentration along the cross section of the polymer-coated fiber.

where C_0 and $C_{\rm ext}$ are the water concentrations corresponding to different radial positions.

When the FBG is immersed in the water bath, it is reasonable to consider the environmental medium as a source of constant water concentration, in which the water outside the fiber will diffuse into the fiber coating until it reaches equilibrium. The solution of (5) is

$$c(r,t) = C_{\rm ext} erfc\left(\frac{r}{\sqrt{4Dt}}\right). \tag{7}$$

In the case when a soluble analyte is gradually added into the water bath, the water concentration in the fiber coating will be higher than that in the environmental medium and the water will diffuse into the environmental medium from the fiber coating until it reaches a new equilibrium. The solution of (5) in this case is

$$c(r,t) = C_{\rm ext} erf\left(\frac{r}{\sqrt{4Dt}}\right). \tag{8}$$

III. Experimental Details

A standard telecommunication single-mode optical fiber (Corning SMF-28) was soaked in high-pressure hydrogen atmosphere (1900 psi) at room temperature for two weeks and then stored in a freezer at −70 °C before use. Two FBGs with a grating length of 1 cm for each were inscribed on the hydrogen-loaded fiber using a KrF excimer laser and a phase mask. After the grating fabrication, the FBG sample was baked at 150 °C for overnight to eliminate the residual hydrogen and the unstable UV-induced index changes. One section of the grating area was then recoated with polyimide polymer, while the other grating area recoated with acrylate polymer. The recoating process resulted in a fiber diameter of 173 μm at the grating section. From an optical microscopy, the thickness of the polyimide coating was observed to be 24.1 ± 1.0 μm. The FBG sensor system is illustrated in Fig. 2(a) with its transmission spectrum shown in Fig. 2(b). The transmission spectrum, measured by an optical spectrum analyzer (Ando 6315E), shows two Bragg wavelengths at 1549.020 and 1550.244 nm

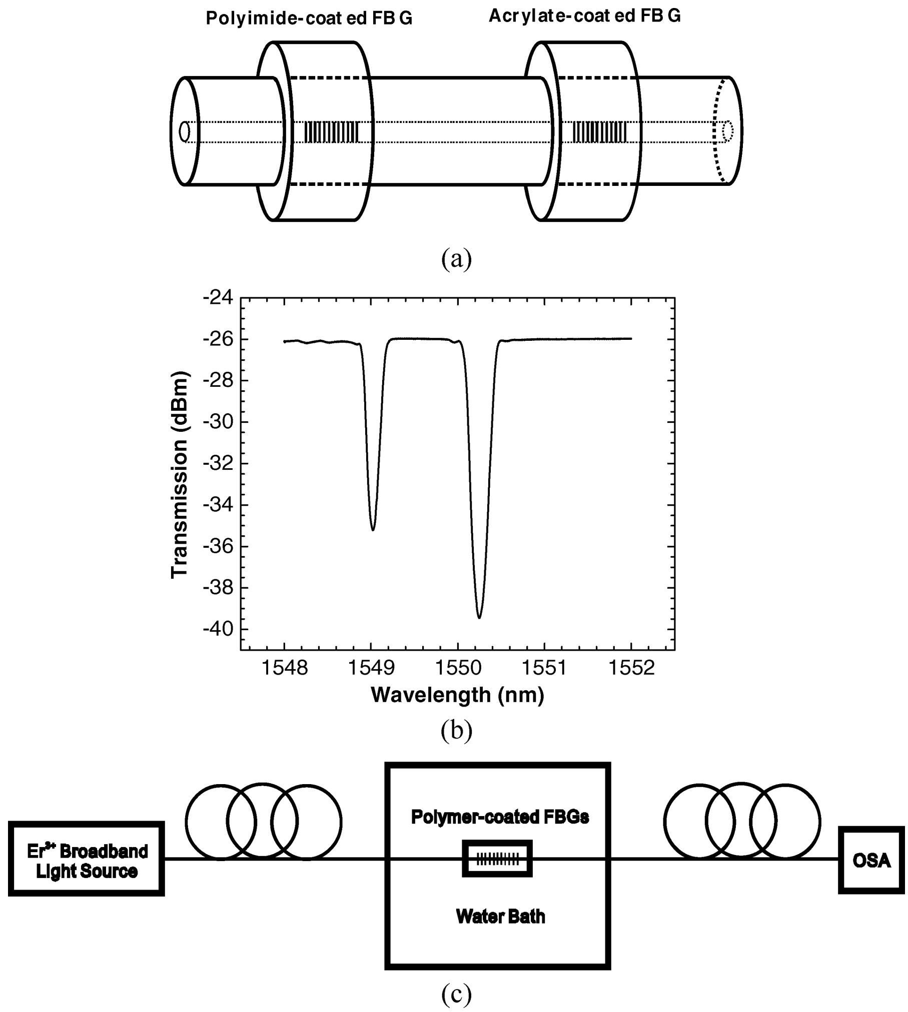


Fig. 2. FBG sensor and measurement: (a) illustration of the FBG sensor used in this study, (b) transmission spectrum of the FBG sensor, and (c) experimental setup for the measurement of sensing performance in a water bath.

TABLE I
CHARACTERISTICS OF THE SENSOR SYSTEM WITH TWO POLYMER-COATED FBGS

	Fiber Type	Fiber Recoating Type	Physical length (mm)	Bragg Wavelength (air, 20 °C) (nm)	Thick-ness (μm)	Bandwidth (nm)	Isolation (dB)	Reflection (%)
Polyimide-coated FBG	SMF-28	Polyimide	10	1550.244	24.1 ± 1.0	0.290	13.47	97.2
Acrylate-coated FBG	SMF-28	Acrylate	10	1549.020	24.1 ± 1.0	0.170	9.24	90.0

with reflection signals of 9.24 and 13.47 dB, corresponding to the acrylate- and polyimide-coated FBGs, respectively. The characteristics of the FBG sensor are listed in Table I.

In the study of the temperature response of the FBG sensor, a microcomputer-controlled water bath was used to control the variations of the environmental temperature, in which the water is the corresponding ambient medium. Fig. 2(c) illustrates the experimental setup used to determine the temperature-induced shift of the FBG transmission spectrum in the water bath. When these two sections of the polymer-coated FBGs had been com-

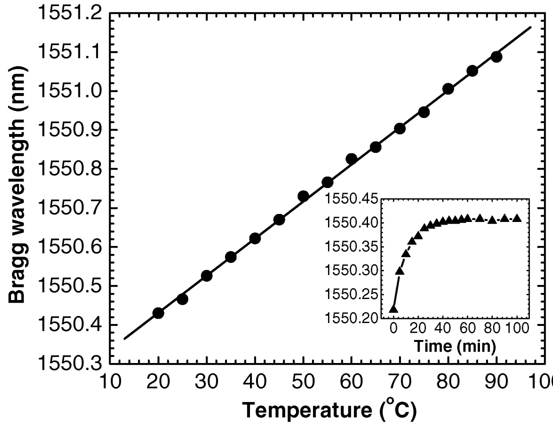


Fig. 3. Bragg wavelength of the polyimide-coated FBG as a function of temperature in the water bath. The inset is the time evolution of the shift of its Bragg wavelength when the grating is transferred from an environmental chamber to the water bath.

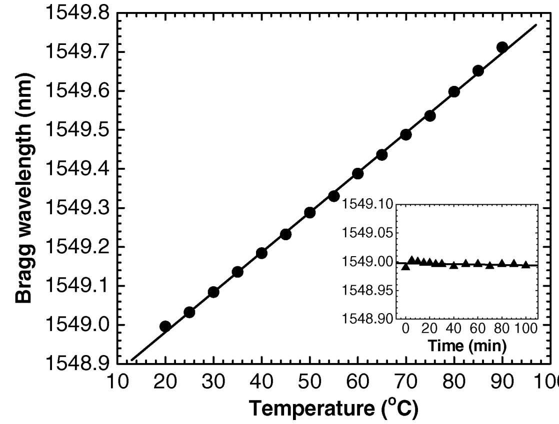


Fig. 4. Bragg wavelength of the acrylate-coated FBG as a function of temperature in the water bath. The inset is the time evolution of the shift of its Bragg wavelength when the grating is transferred from an environmental chamber to the water bath.

pletely immersed in the water bath, certain amount of sugar or KCl was added into the water to adjust the concentration of the solution. The relationship between the concentration of sugar or KCl and the Bragg wavelengths of the FBG sensor will be investigated.

IV. Results and Discussion

A. Measurement of Temperature Sensing Performance

In order to eliminate the strain or bending cross effects [9], the section of the polyimide-coated grating was fixed and tightened during the temperature sensing measurement. When the polyimide-coated FBG was transferred from an environmental chamber with the ambient medium of air to the water bath at a constant temperature of 20 °C, it was found that the Bragg wavelength red-shifted from 1550.218 to 1550.408 nm and stabilized after half an hour (inset of Fig. 3). The increase in the Bragg wavelength was due to the expanding grating period, which was caused by the stretched fiber. After the transmission spectrum of the polyimide-coated FBG in the water bath stabilized, the Bragg wavelength red-shifted from 1550.430 to 1551.088 nm when the temperature was increased from 20 °C to 90 °C (Fig. 3). The temperature coefficient of the polyimide-coated FBG in water, $k_{T\text{water}}$, is 0.0094 $\text{nm}/^\circ\text{C}$.

Following the same procedure mentioned above, the temperature response of the acrylate-coated FBG in water was also studied. During the process of transferring the acrylate-coated FBG from the environmental chamber to the water bath, no apparent shift in the Bragg wavelength was found (inset of Fig. 4). From Fig. 4, the temperature coefficient in water, $k_{T\text{water}}$, was measured to be 0.0102 $\text{nm}/^\circ\text{C}$.

B. Measurement of Sugar Concentration

After both of the polyimide- and acrylate-coated FBGs had been completely immersed in the water bath with a constant temperature of 20 °C for half an hour, a certain amount of natural granulated sugar was added into the water to prepare desired sugar solutions of different concentrations ranging from 0 to 50°Bx. The transmission spectra of the polyimide-coated

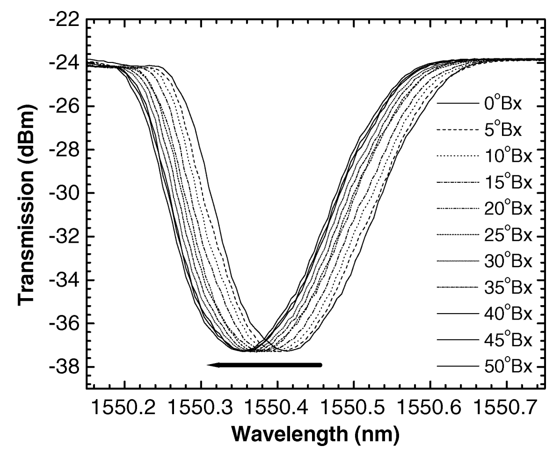


Fig. 5. Transmission spectra of the polyimide-coated FBG as a function of sugar concentration.

FBG as a function of sugar concentration are shown in Fig. 5. When the sugar was added into the water bath, the water concentration surrounding the grating decreased, which resulted in the decrease of the corresponding water concentration in the polyimide coating. In the experiments, for each time the sugar was added, sufficient waiting time was observed to make the FBG spectrum immovability. Fig. 6 shows the Bragg wavelengths of the two FBGs as functions of sugar concentration. The sensitivity decreases when the polyimide-coated FBG is exposed to solutions of higher sugar concentrations as the solubility approaches saturation. It shows that the average sensitivity of the polyimide-coated FBG on sugar concentration is $0.0012\text{nm}/^\circ\text{Bx}$.

The shrinkage of the polyimide coating in sugar solutions results in the blue-shift of the Bragg wavelength of the polyimide-coated FBG, as shown in Fig. 6. In contrast, the acrylate-coated FBG is not sensitive to the change of sugar concentration. The relationship between the Bragg wavelengths of the FBGs and the changes in the temperature and sugar concentration can be described with a matrix equation

$$\begin{bmatrix} \Delta\lambda_{\text{acrylate}} \\ \Delta\lambda_{\text{polyimide}} \end{bmatrix} = \begin{bmatrix} 0.0102 & 0 \\ 0.0094 & -0.0012 \end{bmatrix} \begin{bmatrix} \Delta T \\ \Delta C_{\text{sugar}} \end{bmatrix} \quad (9)$$

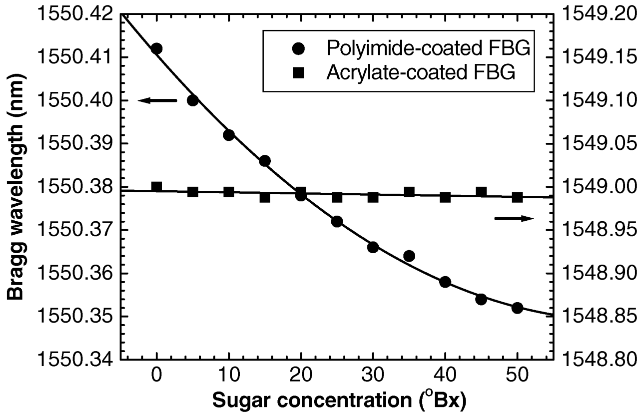


Fig. 6. Bragg wavelengths of polyimide- and acrylate-coated FBGs as functions of sugar concentration.

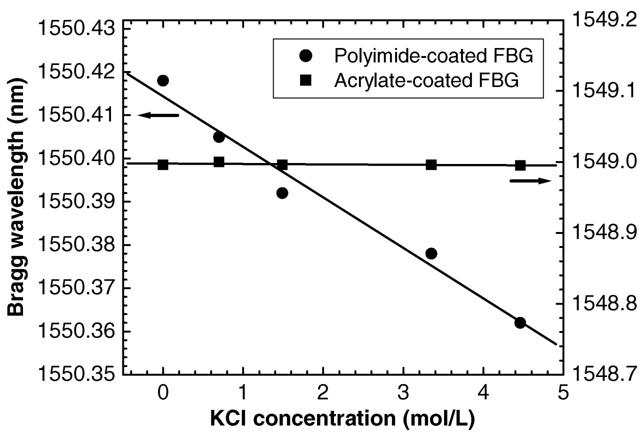


Fig. 7. Bragg wavelengths of polyimide- and acrylate-coated FBGs as functions of KCl concentration.

where $\Delta\lambda_{\rm acrylate}$ and $\Delta\lambda_{\rm polyimide}$ are the shifts in the Bragg wavelengths of the acrylate- and polyimide-coated FBGs, respectively. ΔT and $\Delta C_{\rm sugar}$ stand for the changes in temperature and sugar concentration, respectively. The positive and negative signs of the matrix elements correspond to the red- and blue-shifts of the Bragg wavelengths of the gratings, respectively.

C. Measurement of Potassium Chloride Concentration

Since the sensing mechanism of the FBG sensor discussed here is based on the hygroscopic properties of the polyimide coating, water-soluble analytes other than the concentration of sugar could have the similar swelling effects on the polyimide coating. However, different analytes will result in the different extents of responses manifested in the changes of the Bragg wavelengths. Therefore, it is possible to detect other soluble analytes with the same sensor system. In order to validate the feasibility, an experiment on the determination of the KCl concentration has been performed. Fig. 7 shows the Bragg wavelengths of the two FBGs as functions of KCl concentration, which gives the sensitivity of the KCl concentration of the polyimide-coated FBG as 0.0126 nm/M. The figure also shows the blue-shift of the Bragg wavelength indicating the shrinkage of the polyimide coating in KCl solutions, however, the Bragg wavelength of the acrylate-coated FBG is not sensitive to the change in the KCl concentration. The dependence of the Bragg wavelengths on the temperature and KCl concentration ΔC_{KCl} is

$$\begin{bmatrix}\Delta\lambda_{\rm acrylate}\\ \Delta\lambda_{\rm polyimide}\end{bmatrix} = \begin{bmatrix}0.0102 & 0\\ 0.0094 & -0.0126\end{bmatrix}\begin{bmatrix}\Delta T\\ \Delta C_{\rm KCl}\end{bmatrix}. \quad (10)$$

The experimental results demonstrate that not only sugar concentration but also KCl concentration can be identified by the FBG sensor. Furthermore, it can distinguish different analytes from the difference in the corresponding concentration sensitivities. For a general case described by (11), a character matrix $M_{\rm TA}$ related to the sensitivities of measurands is defined to represent the sensing performance of the sensor system, for example, temperature and analytes, and different character matrix $M_{\rm TA}$ can be used to determine different species and the concentration of the analyte

$$\begin{bmatrix}\Delta\lambda_{\rm acrylate}\\ \Delta\lambda_{\rm polyimide}\end{bmatrix} = M_{\rm TA}\begin{bmatrix}\Delta T\\ \Delta C\end{bmatrix}. \quad (11)$$

This scheme offers a number of advantages over other techniques to measure chemical analytes. With the FBG sensor system proposed here, it is possible to measure different water-soluble analytes by utilizing the polyimide-coated FBG, while simultaneously measure the temperature with the acrylate-coated FBG. For other parameters of widespread interest, such as $\rm Na^+$, $\rm Li^+$, and $\rm Ca^{2+}$ ions, that can swell the polyimide coating in solutions, the same FBG sensor system discussed here can differentiate and quantify these analytes from the different responses of the grating (sensitivity, rate, etc.) once a calibration curve for different substances is developed. Since the FBG fabrication process and recoating technique required for the sample preparation are quite simple, the approach proposed here is promising to realize low-cost substance or chemical sensors. In addition, it is possible to introduce more wavelength channels other than two in this study to realize quasi-distributed measurement.

V. Conclusion

A new scheme for simultaneous measurement of soluble analytes and temperature has been proposed. The approach has been demonstrated with *in situ* monitoring of temperature and sugar or KCl concentration. The sensor system consisted of two in-line polymer-coated FBGs with one acrylate-coated grating sensitive to temperature only and the other polyimide-coated one sensitive to the soluble analytes. The experimental results showed that the temperature sensitivity of the acrylate-coated FBG, temperature, sugar, and KCl concentration sensitivities of the polyimide-coated FBG are 0.0102 $\rm nm/^\circ C$, 0.0094 $\rm nm/^\circ C$, 0.0012 $\rm nm/^\circ Bx$, and 0.0126 nm/M, respectively. As long as the soluble analytes swell the polyimide coating, different responses from the grating (sensitivity, rate, etc.) are expected, which enables the possibility to differentiate and quantify different analytes. In practice, through extensive experimentation and accumulation of data, a calibration curve for different substances can be developed before routine uses, even though the experiments performed here revealed the concentrations of sugar and KCl only. The fiber-optic sensor discussed here is a versatile chemical sensor to monitor different soluble analytes.

Since the two sensing elements have been integrated on one standard single-mode telecommunication fiber, it is possible to achieve quasi-distributed *in situ* monitoring of temperature and analytes over a long distance.

REFERENCES

[1] M. G. Kuzyk, *Polymer Fiber Optics: Materials, Physics, and Applications*. Boca Raton, FL: CRC, 2006.

[2] *Handbook of Optical Fibre Sensing Technology*, J. M. López-Higuera, Ed. West Sussex, U.K.: Wiley, 2002.

[3] X. Chen, K. Zhou, L. Zhang, and I. Bennion, "Simultaneous measurement of temperature and external refractive index by use of a hybrid grating in D fiber with enhanced sensitivity by HF etching," *Appl. Opt.*, vol. 44, no. 2, pp. 178–182, 2005.

[4] K. Zhou, X. Chen, L. Zhang, and I. Bennion, "High-sensitivity optical chemsensor based on etched D-fiber Bragg gratings," *Electron. Lett.*, vol. 40, no. 4, pp. 232–234, 2004.

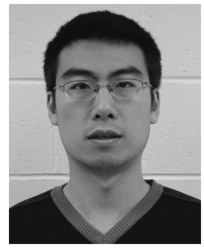
[5] X. Chen, K. Zhou, L. Zhang, and I. Bennion, "Optical chemsensors utilizing long-period fiber gratings UV-inscribed in D-fiber with enhanced sensitivity through cladding etching," *IEEE Photon. Tech. Lett.*, vol. 16, no. 5, pp. 1352–1354, 2004.

[6] A. Iadicicco, S. Campopiano, A. Cutolo, M. Giordano, and A. Cusano, "Self temperature referenced refractive index sensor by non-uniform thinned fiber Bragg gratings," *Sens. Actuators B*, vol. 120, no. 1, pp. 231–237, 2006.

[7] A. Iadicicco, A. Cusano, S. Campopiano, A. Cutolo, and M. Giordano, "Thinned fiber Bragg gratings as refractive index sensors," *IEEE Sensors J.*, vol. 5, no. 6, pp. 1288–1295, 2005.

[8] P. Kronenberg, P. K. Rastogi, P. Giaccari, and H. G. Limberger, "Relative humidity sensor with optical fiber Bragg gratings," *Opt. Lett.*, vol. 27, no. 16, pp. 1385–1387, 2002.

[9] N. Mohammad, W. Szyszkowski, W. J. Zhang, E. I. Haddad, J. Zou, W. Jamroz, and R. Kruzelecky, "Analysis and development of a tunable fiber Bragg grating filter based on axial tension/compression," *J. Lightwave Technol.*, vol. 22, no. 8, pp. 2001–2013, 2004.



Ping Lu received the B.Sc. degree in applied physics from Jilin University, Jilin, China, in 2003, and the M.Sc. degree in condensed matter physics from the Memorial University of Newfoundland, St. John's, NL, Canada, in 2007. He is currently working towards the Ph.D. degree at Memorial University of Newfoundland with research interest on the design, fabrication, and application of fiber Bragg gratings in sensor networks.

Liqiu Men received the B.Sc. degree (Honors) in physics, the M.Sc. degree in condensed matter physics from Nankai University, China, and the Ph.D. degree in optics from the Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Shanghai, China, in 1991, 1994, and 1997, respectively.

She is now with CREAIT Network, Memorial University of Newfoundland. Her research interests include bio-imaging, biophotonics, fiber optics, nonlinear optics, and optical data storage.

Qiying Chen received the B.Sc. degree (Honors) in applied physics from Shanghai Jiao Tong University, Shanghai, China, and the M.Sc. and Ph.D. degrees in optics from the Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Shanghai, in 1989, 1992, and 1996, respectively.

He is an Associate Professor in the Department of Physics and Physical Oceanography, Faculty of Science, and cross-appointed to the Faculty of Engineering and Applied Science at Memorial University of Newfoundland, Canada. He has been Canada Research Chair in Photonics since 2004.

Currently, he is leading a research group working on ultrafast nano-photonics with topics on fiber gratings and sensors, polymer solar cells, femtosecond laser physics, near-field optics and their interdisciplinary applications in environmental and bio-sciences. He has 100 publications in refereed journals and conferences and six patents. His research experience includes optical waveguides, laser physics and technology, optical data storage, optical materials, nano-electronics, scanning probe microscopes, nonlinear optics, laser microfabrication, and photonic lightwave circuits.