Discussion

Dispersion measurement of liquids with a fiber optic probe based on a bi-functional lensed photonic crystal fiber

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Abstract

We report a fiber optic probe suitable for measuring the wavelength dispersion of liquids. The measuring probe is made from an extrinsic fiber-optic cavity formed by a lensed photonic crystal fiber (PCF) and a micro-mirror. The custom-designed lensed PCF plays a dual role of collimator to help realize a long cavity length of the order of millimeters and as an efficient reflector to form the reference beam. The length of the cavity used in the experiment is about 1620 μm. In this article, we present the analytical method of obtaining dispersion of the liquid sample from the spectral interferogram of a single-arm or common path interferometer. Sodium chloride salt solutions with varied concentrations are used as an example to demonstrate the efficacy of the instrument. The index change with concentration is found to be linear at low concentrations and deviates from linearity as it approaches saturation.

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1. Introduction

The refractive index (RI) is one of the important properties of substance and can be used for identifying liquids or measuring the concentration and purity of solutions. The rampant increase in adulteration of liquids, particularly cooking oils [1,2] and fuels [3,4], demands a simple and cost-effective technique for characterizing the liquids. Ever since its invention in late 19th century, Abbe refractometer, which is based on the critical angle measurement, has still remained one of the standard techniques for measuring the RI of liquids in laboratories and even in the industries [5]. In the Abbe refractometer, a liquid sample is sandwiched into a thin layer between an illuminating prism and a refracting prism. The refracting prism is made of a glass with a high refractive index and the refractometer is designed to be used for liquid samples with RI smaller than that of the refracting prism [6]. The Pulfrich refractometer based on a single prism was considered to be a more flexible instrument with higher precision than the standard Abbe refractometer. However, in spite of the long history and a high precision of about $10^{-4}$ to $10^{-5}$, the Abbe and Pulfrich refractometers have the disadvantage of being based on prisms. Since, the temperature sensitivities of the RIs of liquids and glass material (of the prism) are different, the prism based refractometers have the inherent drawback of requiring the measurement be made only at specific temperatures (typically 20 or 25 °C) at which the RI of the prism involved is known. The refractometers therefore require internal water-cooling and a temperature-regulation system, thus making them bulky. Compact, hand-held refractometers referred as Brixmeters [7] have been developed for measuring the sugar levels in the sugars canes and fruits. Although Brixmeters have no cooling system, they have a limited measurable range for which the temperature compensation is done and hence are suitable for a specific application.

Although, several optical refractometers which do not depend on the optical prisms have been reported, fiber-optic refractometers are undoubtedly preferred due to the innumerable advantages of optical fibers such as immunity to electromagnetic interference, compactness, flexibility to reach samples and corrosion resistance. Fiber-optic refractometers can be broadly classified as evanescent, reflective and interferometric. The evanescent-type fiber refractometers work on the interaction of the evanescent field in the fiber with the external RI and obtain the refractive RI by measuring the change in the resonance wavelength with the change in RI. Fiber-optic evanescent refractometers based on surface plasmon resonance sensor [8–10], etched fiber Bragg gratings [11–13], structurally induced LPG [14] have been reported. Although these have high sensitivity, their main limitation is non-linear response to the RI, in addition to the limited measurement range, relatively large temperature cross-sensitivity and fragile nature due to the need for stripping the protective jacket and the cladding region to provide access to the evanescent fields. The reflective-type works on the principle of Fresnel reflection. The RI of the external liquid sample is obtained from the change in reflected power when the fiber tip is in contact with the sample. However, these refractometers
are not reliable as the measurements are susceptible to the power fluctuations of the optical source [15] and also affected by the quality of the reflecting surface. Interferometric refractometers are the natural choice when high sensitive measurement is required. In this type of refractometers, the liquid sample, held in a quartz sample cell is placed in one of the arms of an usually dual-arm Michelson or Mach–Zehnder interferometer [16]. The interferometer essentially converts the RI information encoded in the phase to amplitude. However, the high sensitivity of interferometers also means high alignment and polarization sensitivity. Often fiber-optic interferometers use a polarizer in one of the arms [17] to improve the fringe contrast by ensuring both the beams have the same polarization. Fiber-optic single arm interferometers also referred as common-path interferometers are of interest for the refractometers, since they overcome the alignment and polarization sensitivity of conventional dual-arm configurations while still holding the advantage of the inherent sensitivity of the interferometric techniques. Further, they are convenient for real-time monitoring since they are an immersion-type and do not require a separate sample cell. They are also immune to the fluctuations in the optical source as the measured interferometric sensors modulate the phase and not the amplitude of the beam. The interferometer converts the phase into amplitude. Since the phase difference between the two beams at any wavelength is dependent only on the cavity length and not on the power of the source, it is immune to the power fluctuations of the source.

Intrinsic or extrinsic fiber cavities are suitable fiber-optic configurations for the single-arm and immersion-type refractometers. In the intrinsic type, the two reflections to form the reference and the sample beams of an interferometer are obtained from the two surfaces of a cavity formed in a piece of fiber by etching a hole [18]. The extrinsic type on the other hand is typically composed of a fiber-tip and an external micro-mirror/another fiber-tip enclosed in a tube to form a cavity [19]. In the absence of reflective coatings on the fiber-tips, both configurations are equivalent to a two-beam interferometer, since multiple reflections can be neglected due to the weak Fresnel reflection at the fiber tip. Compact integrated cavities based on micro-fluidic channels have also been reported [20].

In this paper, we report an extrinsic cavity based refractometer. The length of extrinsic or intrinsic fiber optic cavity sensors reported so far is limited to typically less than 150 μm, beyond which the coupling loss increases rapidly [21]. However, liquids particularly require larger cavity lengths due to their property of surface tension, which inhibits their free-collapse over a short length behind the lens surface, which acts as a beam-expansion region. Thus, with the PCF a collimator can be formed in a single-step (applying fusion arc). Further, by choosing appropriate arc parameters, the radius of curvature of the lens surface can be made large to maximize the reflection along with the collimation of the PCF lens [19]. Experimentally, we have found [19] that an arc power and arc duration of 120 mW and 500 ms as optimum parameters to obtain well-collimated beam for a cavity of the order of millimeters and at the same time have strong reflection at the lens surface for getting good interference contrast. The schematic of the sensor-head is shown in Fig. 2(a) while Fig. 2(b) shows the actual microscopic image of the lensed PCF. The air-hole collapsed region which helps in beam-expansion can be easily distinguished from the unaffected PCF by the absence of hairy diffraction pattern caused by the air-holes.

Interferometric methods reported so far to measure the dispersion of optical fibers and liquids are based on dual-arm Michelson interferometers [16]. Here, we present the theory for obtaining the dispersion of the liquid sample from the spectral interference measured using an optical spectrum analyzer. The interferogram measured with respect to wavenumber, \( \sigma = 1/\lambda \) can be represented as,

\[
l(\sigma) = I_0(\sigma)[1 + \gamma \cos(\phi(\sigma))]
\]

where, \( I_0(\sigma) \) is the intensity profile of the source, \( \gamma \) is the visibility or the contrast of the fringes and \( \phi(\sigma) \) is the phase difference between the reference beam \( E_R \) and the sample beam \( E_S \) shown in Fig. 2(a). The phase difference between the two beams has the contribution from the propagation in the cavity, \( \phi_{\text{cavity}}(\sigma) \) and also from the reflection at the mirror, \( \phi_{\text{mirror}}(\sigma) \). Unlike the dual-arm interferometer, where a sample cell made of quartz is placed in one of the arms, there is no separate sample cell for the liquid sample in the single-arm interferometer. Therefore, in order to protect the metal coatings on the mirror, non-corrosive protective coatings are used in general, which have strong wavelength dependent phase change [22,23]. Thus, the phase difference, \( \phi(\sigma) \) can be written as,

\[
\phi(\sigma) = \phi_{\text{cavity}}(\sigma) + \phi_{\text{mirror}}(\sigma)
\]

Since the propagation induced phase change is \( \phi_{\text{cavity}}(\sigma) = 4\pi n_l(\lambda) L_c \), if the cavity length \( L \) is known, the dispersion of the sample \( n(\sigma) \) can be obtained from the extracted phase of \( \phi(\sigma) \), provided the effect of the phase change due to the mirror can be removed. In order to remove the effect of mirror, two interference signals, \( I_1 \) and \( I_2 \) are measured when the sensor-head is not dipped in the liquid followed by dipping it in the liquid sample, respectively. The phases of the corresponding interference signals can be written as,

\[
\begin{align*}
\phi_1 &= 4\pi n_1(\lambda) L + \phi_{\text{mirror}}(\sigma) \\
\phi_2 &= 4\pi n_2(\lambda) L + \phi_{\text{mirror}}(\sigma)
\end{align*}
\]

2. Theory

Lensed PCF is used as the sensor-head for measuring the wavelength dependence of RI or simply, the dispersion of the liquid samples as shown in Fig. 1. In this section, we briefly explain the principle of using PCF in the sensor-head along with the method of extracting the dispersion from the interference signal. Lensed fibers aid in increasing the coupling efficiency between two fibers or between a laser source and its pigtail. In these applications the reflection from the lens surface is not desirable and often anti-reflection coatings are employed to reduce the insertion loss. However, in order to realize an extrinsic cavity sensor with a long cavity length, we need a strong reflection to form the reference beam along with a collimating lens to realize a long cavity. Collimation in general is a two step process involving beam-expansion and focusing. However, recently we have reported [19] a monolithic bi-functional PCF lens formed in a single-step using fusion-arc method. We call it bi-functional because, it acts both as a reflector as well as a collimator. When a fusion arc is placed at the tip of a fiber, the molten silica helps in shaping the fiber-tip. However, when a PCF is used, in addition to the lens formation at the fiber-tip, it causes the air-holes in the PCF to collapse over a short length behind the lens surface, which acts as a beam-expansion region. Thus, with the PCF a collimator can be formed in a single-step (applying fusion arc). Further, by choosing appropriate arc parameters, the radius of curvature of the lens surface can be made large to maximize the reflection along with the collimation of the PCF lens [19]. Experimentally, we have found [19] that an arc power and arc duration of 120 mW and 500 ms as optimum parameters to obtain well-collimated beam for a cavity of the order of millimeters and at the same time have strong reflection at the lens surface for getting good interference contrast. The schematic of the sensor-head is shown in Fig. 2(a) while Fig. 2(b) shows the actual microscopic image of the lensed PCF. The air-hole collapsed region which helps in beam-expansion can be easily distinguished from the unaffected PCF by the absence of hairy diffraction pattern caused by the air-holes.

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\phi_1 &= 4\pi n_1(\lambda) L + \phi_{\text{mirror}}(\sigma) \\
\phi_2 &= 4\pi n_2(\lambda) L + \phi_{\text{mirror}}(\sigma)
\end{align*}
\]
Then, the dispersion of the liquid sample is simply obtained from the difference of the phases of the two interferograms. By defining $\Delta \Phi_{\text{ABS}} = \Phi_{+} - \Phi_{-}$, we have

$$n(\sigma) = 1 + \frac{\Delta \Phi_{\text{ABS}}}{4\pi n d L}.$$  \hspace{1cm} (4)

The cavity length $L$ can be obtained from the first measurement $I_1$ by locating the peak of the Fourier transform of the interferogram measured without the liquid [19]. Therefore, the role of the first measurement is two-fold: obtaining the cavity length $L$ and removing the phase change due to the mirror reflection $\Phi_{\text{mirror}}(\sigma)$.

There are three major techniques reported to extract the phase of a spectral interferogram; inverse-cosine [24], Fourier transform [25], and peak-finding [26] methods. Since the inverse-cosine and the Fourier transform techniques require a complicated phase unwrapping process, we chose the peak-finding method to extract the phase of the interferogram. The phase is constructed by locating the peaks of the interferogram. The phase is written as a polynomial of $\Phi_{\text{corr}}(\sigma)$ reported by Sainz et al. [27]. The absolute phase of the spectrum can be written as,

$$\Phi_{\text{abs}} = 4mL \sigma n(\sigma)$$  \hspace{1cm} (5)

By using the Taylor’s series expansion of RI, at the central wavenumber $\sigma_0$, $n(\sigma) = n(\sigma_0) + (dn/d\sigma)(\sigma - \sigma_0)$ in Eq. (5), the absolute phase can be written as a polynomial of,

$$\Phi_{\text{abs}} = A \sigma^2 + B \sigma C$$  \hspace{1cm} (6)

where $A = 4nL \frac{dn}{d\sigma}$ and $B = 4nL[n_0 - \sigma_0 \frac{dn}{d\sigma}]$. The relative phase obtained from the experimental data can also be expanded as a polynomial using a second order polynomial. A second order polynomial is used since a quadratic fit is known to be better in the wavelength range greater than 550 nm [28].

$$\Phi_{\text{rel}} = A \sigma^2 + B \sigma + C$$  \hspace{1cm} (7)

The phase correction is done at one of the peaks in the interferogram so that, we can use, $\Phi_{\text{abs}}(\sigma_0) = m \pi$. The integer $m$ can be obtained from Eq. (6) as,

$$m = \frac{A \sigma_0^2 + B \sigma_0}{\pi}.$$  \hspace{1cm} (8)

Therefore, the corrected absolute phase can be obtained from the relative phase by using the following two corrections. The first correction is equivalent to correcting the intercept term $C$, while the second term is for correcting the phase to the actual absolute phase;

$$\Phi_{\text{corr}} = \Phi_{\text{rel}}(\sigma) - \Phi_{\text{rel}}(\sigma_0) + m \pi$$  \hspace{1cm} (9)

Therefore, using the corrected absolute phase obtained from Eq. (9) for both measurements, the RI dispersion of the liquid sample with respect to wavenumber and hence wavelength can be obtained by substituting in Eq. (4).

3. Experiment

The fabrication of the sensor-head shown in Fig. 2(a) is done by taking a small piece of PCF of about 5–10 cm length and splicing one of its ends to a suitable patch-cord. Then, a fiber lens is formed on the other end of the PCF piece by placing it near the fusion arc of a conventional arc-splicer. We have used the arc time and power of 500 ms and 120 mW, respectively, for making the lens. The lensed fiber-tip is inserted into two metal tubes of different diameters to protect the fiber and tightly fit into a glass-tube GT as shown in Fig. 2. Before inserting into the GT, a hole is made on one end of the GT with the aid of a diamond cutter and a mirror M is fixed. Then, the mirror and the tubes were carefully aligned to obtain a good fringe contrast and a proper cavity length. Once, the components are aligned, all the components of
the sensor-head are permanently glued in position with the help of a thermal epoxy (EPO-TEK 353ND). Fig. 2 shows the schematic of the proposed dispersion measurement system. The interference signal from the two reflections in the sensor-head is measured using an optical spectrum analyzer (OSA) with the aid of a fiber-coupler. Fig. 3 is the normalized spectral interference signals obtained when the sensor-head is in air and in distilled water, respectively. The length of the cavity used in our experiment was found to be 1620 μm. It can be seen that, when there is no liquid in the cavity, the fringe contrast is very high (greater than 3 dB) demonstrating the performance of the bi-functional lensed PCF used in the sensor head. However, the fringe contrast can be seen to have reduced for the case of liquid sample (distilled water) as shown in the inset of Fig. 3. The reduction in the fringe contrast can be attributed mainly to the reduced Fresnel reflection at the lens surface due to the reduced RI difference between the lens material and the cavity, when the air is replaced by water. Of course absorption of the light beam at the liquid also worsens the fringe contrast. At least in principle, the coupling efficiency and hence the operational range of measurement can be increased by considering the shape of the lens surface along with the radius of curvature. Further, by sacrificing a little bit of the fringe contrast in air, it is possible to have rather good and similar fringe contrasts not only in air but also in liquid. We can also expect an increase in the realizable cavity length as well as a lower reduction in the fringe contrast for liquids by utilizing a curved mirror instead of the flat mirror. Cavities with curved mirrors have better angular tolerance.

In order to verify the proposed procedure for obtaining dispersion, we have measured the spectral fringes for sodium chloride salt solutions with different concentrations. Including pure distilled water (zero concentration), measurements have been made by adding 1, 2, 4, 10, 20, 30 and 40 g of salt in 100 ml of distilled water. Although, data for comparison is not available in this wavelength region and at 20 °C, the measurement results in Fig. 4 show, as expected, increment in RI with salt concentration. Further, the change in the slope for each concentration indicates that the dispersion of the solution changes with concentration. In order to see the trend of RI change with concentration, RI at an arbitrary wavelength (1.3 μm) is shown in Fig. 5. The result elucidates that the RI change is linear at low concentrations and deviates from linearity at higher concentrations as already reported [30]. The change in the RI of a solution is related to the dissolution process which involves a series of microscopic processes involving the breaking of intermolecular bonds of the
solute and solvents and forming inter-molecular bonds between the solute and the solvent [31]. The linearity at low concentration for some solutions indicates the ease and spontaneity of the dissolution process, which will reduce as it reaches saturation. Since, there was no analytical expression or data for comparison of the results shown in Fig. 5, it is difficult to conclude on the resolution, sensitivity and errors involved in the procedure. The liquid samples with known dispersion data and varied concentrations are required to obtain the performance parameters. In our previous work [19], with a cavity of 1000 μm, the sensor is found to have a resolution and precision of 2.6 × 10^{-5} and 6.2 × 10^{-5} respectively for the group index measurement. Therefore, since the sensitivity is proportional to the cavity length, a better performance is expected.

4. Conclusions

We have proposed and presented a long length fiber-optic extrinsic cavity sensor suitable for refractive index (RI) measurement of liquids. The interference fringe measured in air had more than 3 dB contrast even with a cavity length as long as 1.6 mm. The fringe contrast was reduced a little bit when the sensor head was immersed in distilled water. From the spectral phase constructed from the spectral peaks of the interferogram, we could obtain the dispersion of salt solutions. The RI change with concentration is found to be linear at low concentration as expected. We believe that the reduction in the fringe contrast can be handled to some extent by sacrificing fringe contrast for air and also by using a curved mirror instead of plane mirror.

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