Optical fiber sensor system for remote and multi-point refractive index measurement

Kivilcim Yüksel

Electronics Engineering Department, Izmir Institute of Technology, IZTECH, Gülbahce Kampüsü, 35430 Izmir, Turkey

1. Introduction

Refractive index (RI) is an inherent characteristic of a substance that is closely related to the physical and chemical properties making the refractometry necessary in many fields. Examples include quality control of foods during processing and packaging, adulteration detection (edible oils, gasoline or automotive lubricants) [1], monitoring of environmental pollution [2], biomedical applications, and process monitoring of composite materials [3]. Most of these areas require in-situ, self-calibrated, maintenance-free and easy-to-use refractive index measurement capabilities. Commercially available refractometers based on Abbe configuration present some challenges in addressing these requirements largely due to the manner they are in contact with the specimen, their size and their power requirements.

In parallel to the above-mentioned issues in refractive index measurement, another major concern for many industries is to have a compact system which provides multiple, minimally invasive sensor points interrogated from a remote location. This concern is of particular interest in harsh environments such as chemical and nuclear sites, waste water processing units and river pollution assessment locations. Optical fiber sensors have a great potential for such industrial monitoring tasks thanks particularly to their durability against harsh environments in addition to their small dimensions, fast response, and immunity to electromagnetic interference. Fiber optic sensors which have been widely investigated during the last decades are mainly based on fiber gratings sensors (long period fiber gratings [4,5], tilted fiber Bragg gratings [6]), fiber interferometers [7], and surface plasmon resonance (SPR) [8].

Recently, Fresnel reflection-based sensors gained a renewed interest as they provide simple and low-cost solution [9–14]. The operation principle is basically based on the measurement of Fresnel reflection coefficient at the interface (sensor tip) between the optical fiber and the specimen. Fresnel-reflection-based sensors proposed in the literature differ from each other essentially by their method in interrogating the sensor tip. Among these approaches, there is no universal solution that is standardized for the practical implementations (the related state-of-the-art is summarized in Table 1).

The contribution of this article is twofold: first, the performance parameters of a RI sensor based on a commercial OTDR (single wavelength) has been evaluated which will provide a detailed complementary information to the missing parts of the previous (OTDR-based) work. Second, a multi-wavelength OTDR interrogation scheme is proposed which permits to take measurements on several sensor points from a remote terminal (up to several tens of kilometers). The main advantage of the proposed system compared to the previous work is that it can be used on several sensor tips without imposing limitations on the distances between sensor points. Therefore the system can be easily tailored for different application requirements (i.e. number of sensor tips and the distance between...
Table 1: Comparison of the Fresnel reflection-based RI measurement methods.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Interrogator</th>
<th>Multipoint measurement capability</th>
<th>Reported performance parameters</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>[9]</td>
<td>Diode laser (modulated by a pulse train), photodetector and computer</td>
<td>Not reported but may be implemented by using fiber splitter</td>
<td>Precision: $2.5 \times 10^{-3}$</td>
<td>Double-pulse measurement technique implemented (calibration purpose)</td>
</tr>
<tr>
<td>[10]</td>
<td>Diode laser (modulated by a pulse train), photodetector and digital oscilloscope</td>
<td>Implemented by using 2 x 4 splitter</td>
<td>Short-time precision: $2.8 \times 10^{-6}$</td>
<td>– Fast detectors (5 GHz) and oscilloscope (Gbps) required.</td>
</tr>
<tr>
<td>[11]</td>
<td>Broadband source (centered at 1550 nm) and photodetector</td>
<td>Implemented by using 1 x 2 switch</td>
<td>Short-time measurement precision: $8 \times 10^{-6}$</td>
<td>– Distances between probes should be carefully designed</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Long-time precision: $5 \times 10^{-5}$</td>
<td>+ Eliminates the need of double-pulse measurement technique</td>
</tr>
<tr>
<td>[12]</td>
<td>Broadband source (centered at 1538 nm) and OSA</td>
<td>Implemented by using Array Waveguide Grating (AWG)</td>
<td>Sensitivity: $101.9$ dB/RIU in the RI range of 1.33-1.42</td>
<td>– Optical switch is needed</td>
</tr>
<tr>
<td>[13,14]</td>
<td>Commercial OTDR (single wavelength, 1550 nm)</td>
<td>Not reported but may be implemented by using fiber splitter</td>
<td>Sensitivity: $38.7$ dB RIU</td>
<td>– The number of sensing points limited by the signal attenuation</td>
</tr>
<tr>
<td>This work</td>
<td>Multi-wavelength OTDR and AWG proof-of-concept realised by commercial OTDR and WDM coupler (1550 nm and 1625 nm)</td>
<td>YES (intrinsic property of the system)</td>
<td>Long-time precision: $10^{-6}$</td>
<td>– The distances between sensor points are limited by the spatial resolution of the OTDR</td>
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</table>

The effective index of the fiber $n_f$ can be calculated by using the fibre group index and the dispersion relation [9] 1

2. Sensing principle

Reflection from a discontinuity in the index of refraction is a well known property of light traveling through a dielectric medium. For a fiber tip (perpendicularly cleaved fiber end), the power reflection coefficient is given as

$$R_{end} = \left( \frac{n_f - n_a}{n_f + n_a} \right)^2$$

where $n_f$ is the effective index of the fiber, and $n_a$ is the refractive index of air ($n_a = 1.0002739$ [15]). The reflectivity of such an event can be measured by a commercial OTDR. As represented in Fig. 1, the OTDR-based measurement system functions as follows: the optical source injects short optical pulses (probes) into the Fiber Under Test (FUT). The returning light (test signal) is separated from the probe signal using a coupler and is fed into the receiver where the optical power of the test signal is measured as a function of time. The power evolution with time of the detected signal depends on the presence of localised losses, localised reflections, and distributed fiber attenuation. After some internal signal processing, the OTDR display shows a vertical scale of attenuations and reflections in dB (5log) and a horizontal scale of distance in km. When the sensor tip is exposed to the target liquid (cf. Fig. 1), the measured reflectivity (i.e. the end reflection peak corresponding to fibre–liquid interface appearing on the OTDR trace, $R_{end}$ in dB), can be used to calculate the RI value of the liquid ($n_x$) in

$$n_x = n_f \left( \frac{1 - 10^{R_{end}/20}}{1 + 10^{R_{end}/20}} \right), n_f > n_x$$

2.1. Enhancement by using multi-wavelength OTDR

Even though the sensing principle explained above is not complicated, it brings some challenges when having multiple sensor points (point-to-multipoint configuration) implemented by a passive splitter. That is, fiber lines interrogated by the OTDR should have carefully designed length differences between them (higher than the spatial resolution of the OTDR at a particular pulse width). Moreover, short distances between sensor tips requires the use of an OTDR test equipment optimized for high resolution and short dead zones [18] (Fig. 2).

This problem can be solved by using WDM coupler instead of passive splitter where each test signal at a different wavelength is directed to only one branch after the WDM. In order to realize such configuration, the straightforward approach that one might think is the utilization of a commercial multi-wavelength OTDR. As multi-wavelength OTDRs bring increased complexity and cost, an alternative approach would be the use of tunable OTDR. As represented in Fig. 3, tunable OTDR can be implemented by using a commercially available OTDR and a Wavelength Conversion System (WCS). WCS includes two optical circulators (C1 and C2 in Fig. 3), a tunable laser source (TLS), and an optical/electrical (O/E) converter. The optical pulses emitted by the OTDR are directed onto O/E converter via a first circulator (C1). The electrical pulses obtained at the output of O/E converter are amplified and

1 Using Sellmeier equation with the following $A_i$ and $B_i$ constants for 4.5% GeO$_2$ doped silica fiber, we obtained $n_f = 1.4504$ at 1550 nm, and $n_f = 1.4496$ at 1625 nm.

$$n^2 = 1 + \sum_{i=1}^{3} \frac{A_i \lambda^2}{\lambda^2 - B_i^2}$$

$A_1 = 0.49211$; $A_2 = 0.62925$; $A_3 = 0.59202$; $B_1 = 0.04807$; $B_2 = 0.11275$; $B_3 = 8.29299$ [16,17].
modulate the optical power emitted by the TLS. As a consequence, optical pulses at a desired wavelength are produced at the OTDR repetition rate (with a certain pulse delay) and directed into the network. The standard OTDR receives the reflected and backscattered signals via the two circulators C2 and C1 and stores the associated trace. This WCS which was originally developed for Passive Optical Network (PON) monitoring purposes [19] can readily be implemented in RI measurement context.

3. Experimental work and discussion

3.1. Calibration characteristics

In order to obtain the calibration characteristics, two types of solutions (Glycerol-distilled water, Alcohol-distilled water) having different concentrations (between 10% and 90% with a step of 10%) were prepared and their refractive indices were measured by a state-of-the-art refractometer (Abbe 2WAJ). Fig. 4 represents the evolutions of measured refractive index as a function of Glycerol and Alcohol concentrations. One can observe the linear fit with the computed slopes of $\frac{\partial n}{\partial C} = 0.00147$ (Glycerol), and $\frac{\partial n}{\partial C} = 0.00039$ (Alcohol). The fitting functions on Fig. 4 for Glycerol (dashed line) and Alcohol (solid line) concentrations are: $n = 0.0015 \times C + 1.3338$ (fitting degree = 0.9994), and $n = 0.0004 \times C + 1.3319$ (fitting degree = 0.9986), respectively.
The calibration characteristics were obtained in an air conditioned laboratory environment at room temperature. The temperature dependence of the refractive index, $\partial n/\partial T$ is given as $10^{-4}$ $\text{C}^{-1}$ for most of the liquids [20].

The influence of temperature can be ignored for concentration measurements, as all the measurements were taken at a temperature-controlled laboratory environment (the temperature variation is less than 1–2 $\text{C}$ during the measurements).

The reference measurements presented in the manuscript have been realized by using two state-of-the-art refractometers having light sources at 589.3 nm. These reference values were then converted into values at the wavelengths of 1550 nm and 1625 nm based on the Sellmeier and polynomial fit parameters, provided in Refs. [21,22], respectively.

3.2. Measurement results

The first part of the measurements were performed by our Fresnel reflection-based fiber optic sensor interrogated by a commercial OTDR (cf. Fig. 1). The fibre tip located at 5.3 km is a standard FC/PC connector with ceramic ferrule diameter of 2.5 mm. The OTDR parameters were set as follows: pulse width of 10 ns (the backscatter level just before the sensor point depends on the pulse width but the reflection height does not [23]), distance range of 10 km and averaging time of 30 s. The results presented in Fig. 5 representing the evolution of the sensor deduced vs reference RI values successfully demonstrate the refractive index measurement capability of the sensor. The slopes of the linear fit are: 1.0485 and 1.0195, for Fig. 5a and b, respectively. The end reflection peaks on the OTDR trace for three different Glycerol concentration values (10%, 30%, and 80%) together with the view of complete OTDR trace is shown in Fig. 6.

Fig. 7 represents theoretical values of the Fresnel reflectivity as a function of refractive index (theoretical $R_{\text{ref}}$ values are calculated using the principle explained in Section 2). As can be seen from the inset in the same figure and in Fig. 8, measured reflectivity values agrees well with the theory for the RI span between 1.33 and 1.44. Lastly, the measurements were repeated three times for all the concentration values applied. These repeatability measurements highlighted a standard deviation on the measured refractive index $1.7 \times 10^{-4}$. 

Fig. 6. Enlarged view of the end reflection peak when sensing head (fiber tip) is exposed to 10%, 30%, and 80% Glycerol solutions. Inset: complete OTDR trace.

Fig. 7. Theoretical values of the Fresnel reflectivity versus refractive index. Inset: measured reflectivity values for the RI values between 1.33 and 1.44 using proposed sensor.

Fig. 8. Comparison between theoretical and measured reflectivity values for the RI range between 1.33 and 1.44 (dashed line represents the curve of theoretical values of the Fresnel reflectivity).
3.3. **Proof-of-concept measurements with the enhanced sensor system (multi-wavelength OTDR)**

In the last part of the measurements, proof of concept experiments were realized by using the 2-wavelength OTDR (1550 nm, 1625 nm) and WDM coupler. The following measurement procedure has been applied:

- Both probe signals (1550 nm and 1625 nm) are first taken in air for calibration.
- When the Probe-2 (1550 nm) is measuring air, Probe-1 (1625 nm) is calibrated to distilled-water.
- Probe-2 is then used to measure the RI of different chemical species including Glycerol and Alcohol solutions, Ethanol, Methanol, Acetone and Toluene (meanwhile Probe-1 is kept in distilled-water to determine the precision of the measurements).
- Finally, both probes are used to take simultaneous measurements to check the multi-point measurement capability of the proposed sensor (an example case is represented in Table 2 where probe 1 is taking measurement from Acetone while probe 2 is used for Toluene).

The measurements successfully demonstrated the multi-point measurement capability of the multi-wavelength OTDR scheme even when the two probes are approximately at the same distance from the interrogator unit. By using an AWG, number of sensor probes can be increased without any limitation on the distances between sensor locations. Furthermore, one of the sensor probes can be dedicated to calibration (see the measurement procedure above) while the others can be used to achieve simultaneous temperature and RI measurements.

4. **Conclusions**

A fast and simple technique to measure refractive index of liquids is proposed and experimentally demonstrated. The sensing principle is based on the measurement of Fresnel-reflection from SMF fiber tips by the way of multi-wavelength OTDR from a distant location. The main advantage of the proposed method is that several sensor points can be interrogated without any limitation on the distances between sensor locations. A wide panel of applications requiring the self-calibration and easy implementation features, particularly those in difficult environments imposing the measurements from multiple sensor points located at the outside plant, can be envisaged.

References


**Biography**

Kivlicim Yüksel completed her M.S. degree at the electronics engineering department of the Ege University (Izmir, Turkey) in 2000. Between 2002 and 2005, she worked at Multitel asil (Mons, Belgium) where she was involved in a project dedicated to monitoring methods based on optical time domain reflectometry technique for point-to-multipoint optical access networks. She then joined the electromagnetism and telecommunications department of the Faculté Polytechnique de Mons (FPMs) in 2005 where she worked as teaching assistant. She received a DEA (Diplôme d'études approfondies) and a Ph.D. degree in fiber optics from the Faculté Polytechnique de Mons (FPMs, Belgium) respectively in 2006 and in 2011.

**Table 2**

<table>
<thead>
<tr>
<th>Solvent (Probe-1)</th>
<th>Measured RI</th>
<th>Reference value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone (Probe-1)</td>
<td>1.3556</td>
<td>1.35101</td>
</tr>
<tr>
<td>Toluene (Probe-2)</td>
<td>1.4754</td>
<td>1.47756</td>
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</table>

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Currently she works as an assistant professor at the electronics department of Izmir Institute of Technology.

Her research topics lie in three main areas:

1. Distributed and quasi-distributed optical fibre sensors, fiber-based optical reflectometry.

2. Optical access networks, Passive Optical Networks (PONs), physical layer monitoring of PONs, Fiber to the home (FTTH) technologies.

3. Development of innovative concepts for smart traffic systems based on telecommunication and sensor technologies.