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A Fiber Optic Sensor for Measurements of Solute Concentration in Fluids

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A new and simple calibration technique that greatly enhances the measurement sensitivity of conventional fiber-optic reflectometry based on Fresnel reflection from the tip of a fiber is used for demonstrating the feasibility of measuring solute concentrations and index changes in fluids to very high precision. The amplitude of pulses originating from reflection from the fiber-fluid interface is compared in real-time with the amplitude of reference pulses from a fiber-air interface such that errors caused by pulse amplitude fluctuations and slightly varying detector responses are corrected. Using solutions of salt and water, it is demonstrated that the technique is capable of measuring index changes of about 1×10^{-5} corresponding to a salt concentrations of 0.01 %.

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I. INTRODUCTION

The measurement of salt concentration in real time has been of great interest to ocean engineering. In the study of fluid dynamics, not only is it important to obtain the concentration field precisely, it is often essential to obtain the concentration as a function of time and space in a dynamic problem. For example, the concentration measurement of a turbulent jet is important in the study of pollutant mixing and transport [1]. The problem is strongly temporal and spatial dependent and requires precise concentration data with high temporal and spatial resolutions. To preserve the flow characteristics a small probe is also necessary to minimize the disturbance caused by the introduction of the probe.

High-performance liquid chromatography (HPLC) using interferometric and flow-cell techniques [2]- [4] for chemical analysis have extremely high sensitivity, but their constructions and geometries are cumbersome and inappropriate for some applications such as in-situ sea-water salinity measurement in a water tank. A number of optical techniques, implemented with bulk optics, that may be appropriate for studying the salinity of seawater either in the open sea or in a water tank where the spatial and temporal dependence of salt concentration needs to be determined, have been reported. They measure the refraction angle as a function of liquid index [5], and intensity change caused by Fresnel reflection between a glass plate and a liquid. [6] By allowing an air-gap between two optical fibers, liquid solutions can be placed between the two fibers. Solute concentrations can then be determined from the transmitted signal spectra through the two fibers when the fluid to be measured fills the gap. [7,8] Seawater salinity [7] and electrolytes concentration [8] have been measured by this technique.

Here, we introduce a new calibration technique that greatly enhances sensitivity when applied to conventional fiber optic reflectometry based on Fresnel reflection from the fiber tip. The overall technique provides accuracy, simplicity, near non-invasiveness because of the small dimension of the optical fiber, high spatial resolution of less than 10 μm, and almost unlimited temporal resolution.

II. PRINCIPLE AND EXPERIMENTAL SETUP

The idea of this experiment is to measure the reflected optical power due to Fresnel reflection from

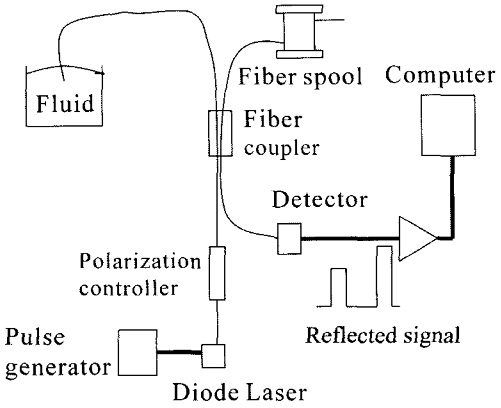

FIG. 1. The experimental setup for concentration measurement. The light source is a 1.3 μm wavelength multi-longitudinal diode laser.

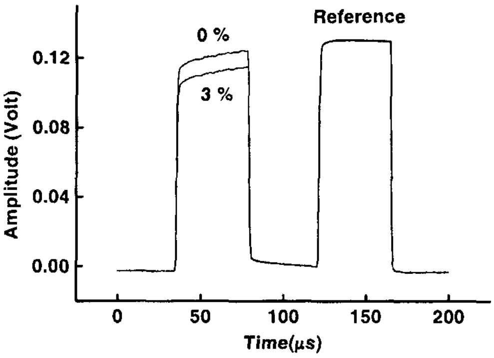

FIG. 2. Typical reflected pulses from the fluid-fiber and air-fiber interface.

the fiber-fluid interface. A change in the reflected optical power measures the change in the refractive index of the fluid. However, to increase measurement sensitivity, the reflected power is normalized by a similar reflected power from a reference fiber-air interface, with both reflected signals derived from the same optical source, and detected by a common detector.

The idea mentioned above was implemented by the experimental setup shown in Fig. 1. A train of optical pulses, derived from a diode laser driven by a pulse generator, is launched into a standard telecommunications graded single-mode optical fiber (SMF-28 fiber). The optical pulse is divided into two paths by a 2 × 2 single-mode fused fiber coupler with a nominal 50% : 50% splitting ratio. The end of the signal fiber is immersed in the fluid while the end of the reference fiber rests free in air. Returned pulses due to Fresnel reflections from both fiber ends are detected by a dc-coupled detector. The pulses are subsequently amplified and processed by a computer. The experiment is conducted in the pulse mode so that the reflected power from the signal and reference fibers can be temporally separated. Thus, the signal path consists of only a few meters of fiber while the reference path is a spool of long fiber. The length of the long fiber used is arbitrary as long as the returned signal and reference pulses can be temporally separated.

A pair of time-separated optical pulses reflected from the fiber-fluid and fiber-air interface is shown in Fig. 2. It is important to note that both pulses are derived from a common optical pulse but delay in time by $2L/c$, where L is the length difference between the two optical paths and c is the speed of light in the fiber. This assures that any pulse amplitude fluctuation will not limit the measurement precision. The first pulse which represents the overlay of two reflected pulses from the fiber-fluid interface with two different salt concentration of 0 and 0.03 gram – salt/cm^3 (3% salt) shows the amplitude dependence on solute concentration. The second pulse is the reference pulse. It is noted that, unlike the second pulse, the first pulse does not have a flat top. This is due to the additional contribution originating from backscattered Rayleigh light from the reference pulse traveling in the long fiber. However, the Rayleigh backscattered light does not affect the differential index measurement. The amplitude of each pulse is made almost equal by adjusting the splice loss while splicing the reference arm to the fiber coupler.

The quantity of interest is the amplitude ratio R between the signal and the reference pulse. By taking the ratio, drifts in laser optical power, potential non-polarization dependent temporal variations in the splitting ratio of the fiber coupler and, to a good degree, the drift in the detector response can be calibrated out. To demonstrate the merit of our ratio technique, we measured the amplitude and their ratio immediately after the system was switched on. Fig. 3 shows the plot of the amplitude of the individual signal and reference pulse and the amplitude ratio R as a function of time upon the turn-on of the system. It is noted that the ratio R stays relatively constant despite the drift in the individual amplitude. By taking the ratio, it is shown that our method is effective in calibrating out the drift.

One issue is the somewhat polarization dependent loss of the fiber coupler as the fiber used are standard telecommunication fiber which are birefringent in nature. Therefore, the polarization state of the returned pulse cannot be maintained indefinitely in a routine environment, causing the relative polarization between the signal and reference pulse to vary in time. This effect, which causes the amplitude ratio R to drift slightly in time, can be simulated by adjusting the po-

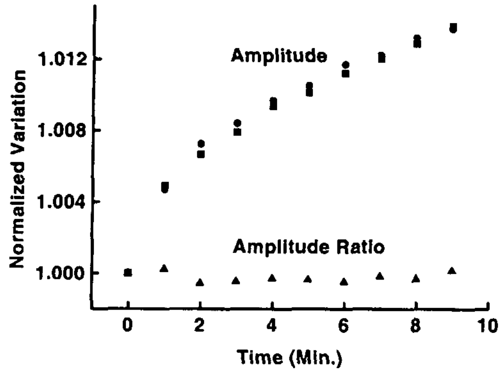

FIG. 3. Squares and circles show the drift of the signal and reference pulse amplitude normalized to the initial value. Triangles show the normalized amplitude ratio.

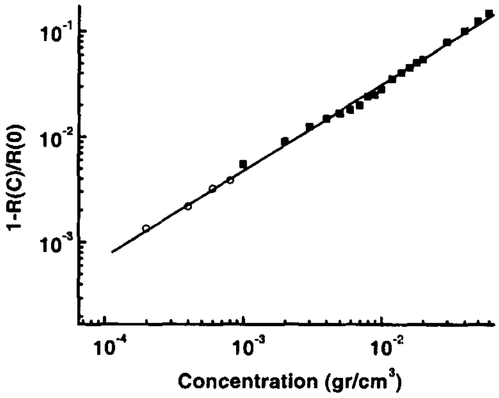

FIG. 4. A plot of $\{1 - R(C)/R(0)\}$ versus concentration in log scale. The open circles are low concentration data taken on a different day.

larization controller shown in Fig. 1. The data shown in Fig. 3 indicates a slightly varying amplitude ratio R due to the polarization effect. This effect limits the index resolution to about 2×10^{-5} in the present experiment.

The formula used in this experiment for calculating the concentration dependent refractive index change is given below. Taking the derivative of the Fresnel reflection formula [9] with respect to the refractive index of the solution, one obtains the change in the amplitude ratio δR due to a change in the refractive index δn_c of the solution.

$$\frac{\delta R(C)}{R(C)} = -\left(\frac{4n_f}{n_f^2 - n^2(C)}\right)\delta n(C) \tag{1}$$

where n_f is the refractive index of the fiber and $n(C)$ is the refractive index of the solution at a solute concentration of C. Writing $R(C) = R(0) + \delta R(0)$, and using Eq. (1) for $\delta R(0)$, one obtains,

$$1 - \frac{R(C)}{R(0)} = \left(\frac{4n_f}{n_f^2 - n^2(0)}\right)\frac{dn}{dC}\delta C \tag{2}$$

where $n(0)$ is the refractive index of pure water and $R(0)$ is the ratio at zero concentration. Eq. (2) is used for analyzing the data.

Two separate sets of data were taken on different days. One set of data was taken for salt concentration ranging from 1×10^{-3} to 6×10^{-2}gram − salt/cm^3. The other set was taken for concentration varying from 2×10^{-4} to 1×10^{-3}gram/cm^3. The data for the lower concentration set was taken within 5 minutes to minimize temperature and polarization effects, since the refractive index of water is a fairly strong function of temperature. [10] The higher concentration set was taken within 2.5 hours. Each set of data for $R(C)$ was normalized by $R(0)$ measured once at the beginning of each data set. For water, $dn/dT = 9 \times 10^{-5}/°\text{C}$, and for the fiber, $dn_f/dT = 1 \times 10^{-5}/°\text{C}$. From the temperature coefficients, the calculated error in the ratio $R(C)/R(0)$ is 1.5×10^{-3} per degree due mainly to the temperature coefficient of water. The corresponding error in determining the index change is about $8x10^{-5}$ per degree. However, within the 5 minutes measurement time the temperature is constant to about 0.1°C, the resolution of the thermocouple used in the temperature measurement. Thus, the corresponding index resolution limited by temperature effects is about 8×10^{-6} for 0.1°C stability. The measured data are plotted in Fig. 4 as $\{1 - R(C)/R(0)\}$ versus C in log scale to better show the data at very low concentration and its connection to the higher concentration data. We use Eq. (2) to calculate dn/dC from the measured data. Using a value of $n_f = 1.45$ for the fiber and $n(0) = 1.33$ for water, we found that $dn/dC = 0.13$/gram/cm^3 in good agreement with a value of 0.12/gram/cm^3 from reference 4. From Fig. 4 it is observed that the resolution of the present technique is about 1×10^{-4}gram − salt/cm^3. This corresponds to an index resolution of 1×10^{-5}. This resolution is about a factor of 5 better than the best bulk-optic technique. [5,6]

III. CONCLUSION

In conclusion, we introduce a new calibration technique that greatly enhances measurement sensitivity when applied to conventional fiber optic reflectometry based on Fresnel reflection from the fiber tip. The technique was used for demonstrating measurement of salt concentration in water. The technique gives an index measurement resolution of about 1×10^{-5} and salt concentration resolution of about 0.01gram/cm^3

limited by temperature and polarization effects. This ratio-meter should be appropriate for various measurements, in particular for measurements of the spatially and temporally dependent concentration field in an environment such as the open sea or a water tank.

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