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Fiber Bragg Grating Sensor for Detection of Nitrate Concentration in Water

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Abstract: The concentrations of chemical species in drinking water are of great interest. We demonstrated etched fiber Bragg grating (FBG) as a concentration sensor for nitrate by analyzing the Bragg wavelength shift with concentration of chemical solution. The FBG is fabricated by phase mask technique on single mode Ge-B co-doped photosensitive fiber. Sensitivity of FBGs to the surrounding solution concentration can be enhanced by reducing diameter of the cladding with 40 % HF solution. The maximum sensitivity achieved is 1.322×10^{-3} nm/ppm. The overall shift of Bragg wavelength is of the order of 6.611×10^{-2} nm for 10 to 50 ppm concentration. *Copyright © 2011 IFSA.*

Keywords: Concentration sensor, Photosensitive fiber, Fiber Bragg grating, Etching, Nitrate.

1. Introduction

Today precise measurement of chemical species in drinking water has a great importance in the society, because of the toxic effects of these chemicals cause to humans. Development of simple low cost, portable sensors capable of direct measurement of water pollution is of considerable interest in this context. Excess quantity nitrogen in water creates health hazard and their accurate estimation is very important. The total nitrogen content of water samples can be present in many chemical forms such as nitrites, nitrates, ammonia etc. In the process of decomposition, raw sewage undergoes a chemical change. Among the end products is nitrate nitrogen. When nitrate nitrogen occurs, it is considered evidence of pollution either from septic tank fields, cesspools or other sewage sources. Inorganic nitrogen (nitrate NO_3) is a major source of pollution in ground water, surface water and the air. Applications of nitrate containing fertilizers can create distributed or non point source pollution problems. Scalable nitrate sensors (sensors which are small and inexpensive) would enable us to better assess non point source pollution processes in agronomic soils, ground water and rivers [1, 2]. Where a groundwater is known to contain little or no nitrate nitrogen naturally, the appearance of any significant increase is a probable indication of pollution. When nitrogenous matter is oxidized by the environment, the nitrogen remains mainly in the form of nitrites and nitrates.

Many of the methods like chemical, electrochemical and spectroscopy methods are adopted in finding the concentration of nitrate [3]. Here we are presenting a simple method using Fiber Bragg Grating (FBG) to determine the concentration of nitrates in water in ppm level.

Fiber optic sensors offer numerous advantages over electric transducers due to their small size, high sensitivity and possibility of distributed measurements. The research field in optical fiber grating technology has opened a new platform in both communication and sensor field. These fiber optics have originally developed to multiple signals in optical networks and are now being widely used in the field of sensors, such as to measure strain, temperature, pressure and as a chemical sensor [4-6]. Specifically FBG sensors have attracted considerable attention.

Gratings are simple intrinsic sensing elements which can be photo-induced into a silica fiber [7]. The fiber gratings are classified as Long Period Grating (LPG) and Fiber Bragg Gratings (FBG) depending on their grating period. The grating period in LPG is of the order of more than 100 μm, where as in FBG it is of the order of less than 1μm [8]. The FBG is a periodic modulation in refractive indices of the core of the fiber. When a broadband light is connected to FBG a narrow band of wavelength centered at one certain particular wavelength known as Bragg wavelength λ_B is reflected. The FBG works on the principle of reflected wavelength λ_B and is given by

$$\lambda_B = 2\, n_{eff}\, \Lambda,$$

where n_{eff} is the effective refractive index of the core and Λ is the grating period.

The reflected wavelength mainly depends on parameters like grating pitch and effective refractive index. When cladding region is reduced along the length of grating region, the n_{eff} is significantly affected by surrounding refractive index (SRI) [9]. By reducing the cladding part of fiber, grating can be made to act as a chemical sensor [10]. The change of wavelength due to the changes in measurand is used for sensing applications. Our particular interest is the use of FBGs as chemical sensors where the change of wavelength is induced by changing the chemical composition around the sensor. In order to measure small changes in composition, it is important for the optical mode to penetrate evanescently into the surrounding solution. A sensing scheme for chemical sensing was proposed based upon Bragg grating in- and out-coupling for increased fluorescence excitation [11]. A method of increased sensitivity to surrounding index by etching the fiber close to the core diameter was presented by [12]. Sensors have been developed for propylene glycol and sugar solutions down to 0.067 % and 0.027 % respectively by the half etched FBG [13]. We demonstrated here the completely cladding etched FBG as a chemical sensor for nitrates having 10-50 ppm sensitivity.

2. Fabrication Technique

Two methods are widely adopted for fabrication of FBG, interferometric technique and phase mask technique [4-6, 14, 15]. Interferometric method is extremely difficult to produce FBG on large scale because of the high sensitivity of the interference condition to environmental disturbance. Phase mask technique is proved to be advantage over interferometric technique [14, 15]. Phase mask is used to photo-imprint the gratings on photosensitive fibres and which is relatively simple, flexible, and low-cost and can be produced in large-volume. So phase mask technique is being most reliable method and is being now widely adopted for the fabrication of FBG.

A single mode Ge-B co doped photosensitive fiber (Newport F- SBG -15, step index profile of NA 0.12 - 0.14, cladding diameter 125 ± 1 µm, and operating wavelength 1550 nm) is chosen to form gratings, which are formed using phase mask technique. Phase mask is a diffractive element (on silica slab) exposed through UV source to produce interference pattern, which in turn produces a permanent change in refractive index of the photosensitive fiber core, which is immediately kept behind the phase mask in proximity and parallel [5].

The acralyte coating of the Ge-B co doped photosensitive fibers is removed carefully. The cleaved Ge-B co-doped photosensitive fiber is placed on a platform for the exposure to the KrF source of wavelength 248 nm. The schematic arrangement for the fabrication of FBG is shown in Fig. 1. The cleaved Ge-B co doped photosensitive fiber, which is placed behind very close to phase mask is exposed to KrF source. The grating formation can be monitored using optical spectrum analyzer. The gratings are formed in a very small time interval of 20 seconds. The advantages of Ge-B co doped photosensitive fiber is that the exposure time for grating formation is remarkably reduced to a UV source [6].The reflected spectra is shown in Fig. 2.

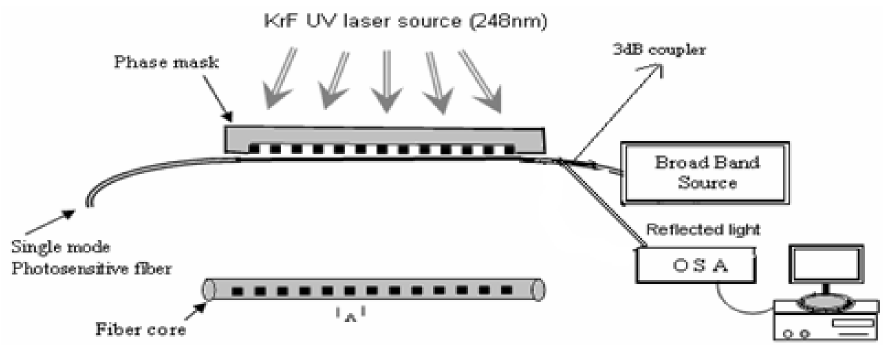

Fig. 1. The schematic diagram FBG on photosensitive single mode fiber.

3. Design of FBG Based Chemical Sensor

For proper designing of FBG to act as a concentration sensor the cladding region has to be etched where the gratings are formed. This leads to direct interaction of core mode with surrounding media. Thus results in a wavelength shift of FBG and varies in accordance with change in refractive index of surrounding media. The 40% HF is used for etching the cladding region for around 60 minutes. The changes in wavelength were closely monitored during etching process as shown in Fig. 3.

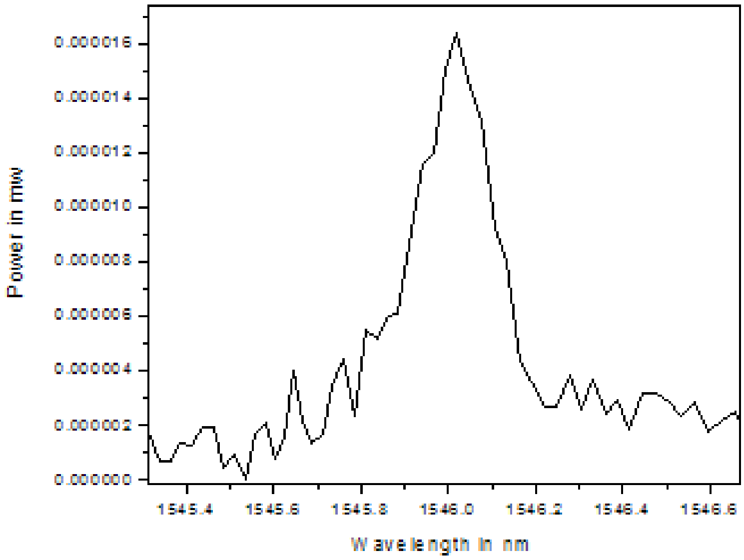

Fig. 2. The reflected spectrum of FBG.

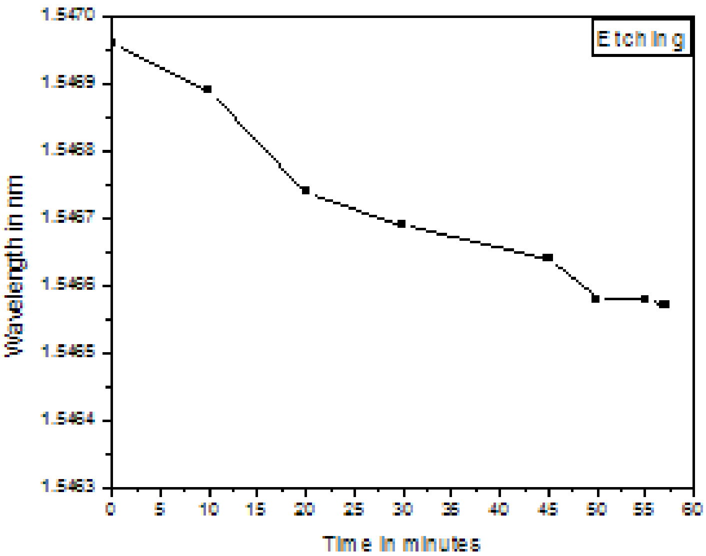

Fig. 3. Etching Process of FBG.

4. Experimental Setup

The experimental setup for nitrate concentration sensor using FBG is as shown in Fig. 4. In order to characterize the FBG sensor sensitivity to the surrounding media for concentration changes, the different solutions of nitrate concentrations varying from 10 ppm to 50 ppm are prepared by dissolving KNO_3 in water. Added reagents mainly as sulphuric acid, and O-phosphoric acid with appropriate volumes. These test solutions are allowed for measurements. FBG is immersed in test tube containing nitrate solution, which is connected to broadband source through 3 dB coupler. The care has to be taken that FBG should be completely dipped in the nitrate solution. The broadband source was injected into the fiber and the reflected spectrum from FBG is observed through optical spectrum analyzer. As the concentration of surrounding nitrate solution is changed, it changes the effective refractive index of FBG and cause shifts in Bragg wavelength (λ_B). Reflected spectra were recorded for different concentrations of nitrate solution ranging from 10 ppm to 50 ppm. Each time the grating region is cleaned with acetone solution properly before exchanging the different concentrations of nitrate solution to avoid contamination.

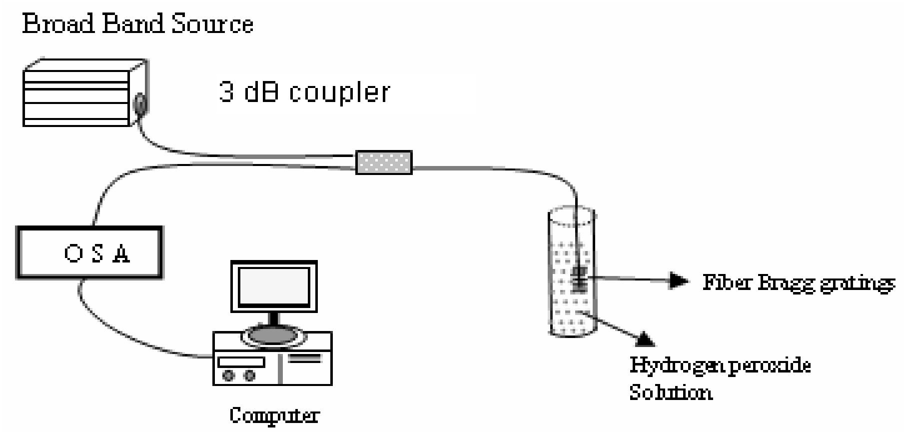

Fig. 4. Experimental setup for concentration measurement of Nitrate solution.

5. Results and Discussion

The spectral response of change in wavelength with concentration is shown in Fig. 5. By measuring the shift in Bragg wavelength λ_B with the corresponding concentration of nitrate solution, we can plot a graph of Bragg wavelength shift versus concentration (ppm) of nitrate solutions as shown in Fig. 6. From Fig. 5, it can be observed that the concentration of surrounding media increases the reflected spectrum shifts towards longer wavelength. This can be visualized interms of an increase in the n_{eff} of the cladding which changes to the resonance condition leading to the decrease in resonance wavelength. From Fig. 6, the sensor is used to detect the concentration of nitrate from 10-50 ppm. The sensitivity of sensor is 1.322×10^{-3} nm/ppm. The overall shift of Bragg wavelength is 6.611×10^{-2} nm for 10 to 50 ppm. As the concentration of nitrate increases, difference in wavelength increases given in Fig. 7. From the linear fit to Figs. 6 and 7, the calculated values for slope, intercept and correlation coefficient are in Table 1. From these results, it is able to discriminate wavelength shifts with a resolution of Pico meter range at lower concentrations.

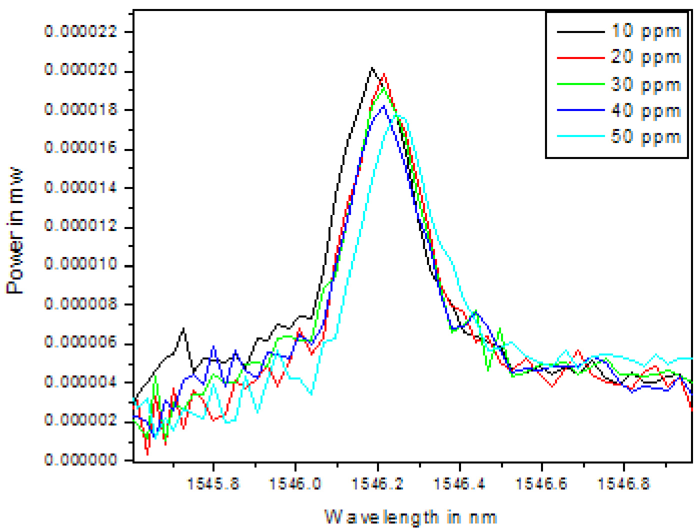

Fig. 5. The spectral response of FBG for different concentrations of Nitrate solutions.

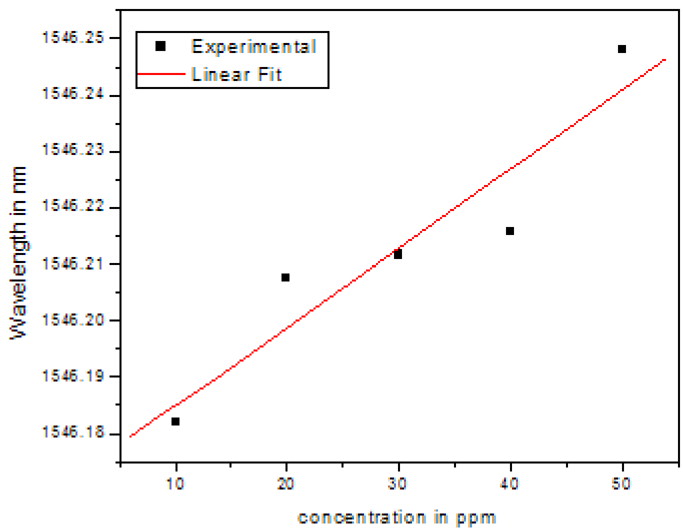

Fig. 6. Wavelength shift versus concentration of nitrate solution.

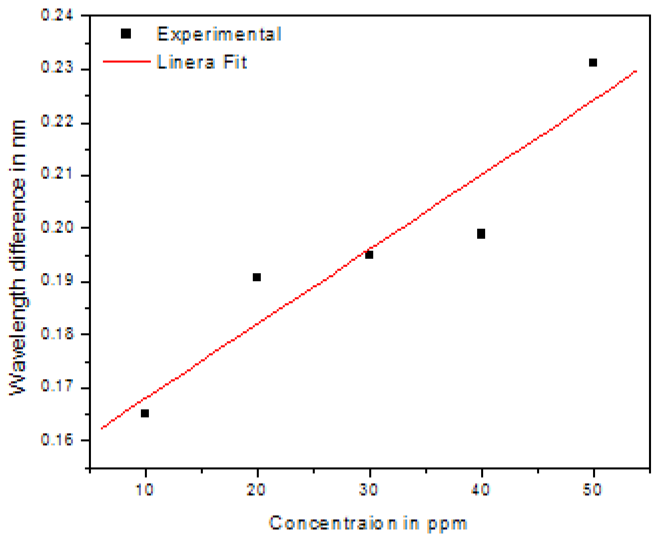

Fig. 7. Wavelength difference Vs Concentration.

Table 1. Slope, Intercept and correlation coefficient from Fig. 6 -7.

Parameter	Fig. 6	Fig. 7
Slope	0.0014	0.0014
Intercept	1546.18 nm	0.154 nm
Correlation Coefficient	0.93918	0.93918

6. Conclusions

We have designed the etched FBG sensor to detect the concentration of nitrate in water. This system can be used for measuring the nitrate compounds in water with good sensitivity in the lower concentration range. Values for nitrate measurement of two samples are given Table 2. The measured values are compared with some other sophisticated methods like Ions Elective Electrode method. The determined values are in good agreement with the WHO standards. This establishes that, the system can be used for measuring fiber grating chemical sensor, where the changes in concentration of nitrate content in water for monitoring the water quality. These sensors can be used in agriculture, industrial fluids and food industry.

Table 2. Comparative measurements for nitrate concentrations in water.

Chemical Species	Samples	Fiber Bragg grating	WHO* Standard	Indian Standard
Nitrate	1 2	32.442 ppm 22.603 ppm	45 ppm	45 ppm

*WHO-World Health Organization.

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