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Highly Sensitive Cadmium Concentration Sensor Using Long Period Grating

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Abstract: In this paper we have proposed a simple and effective Long Period Grating chemical sensor for detecting the traces of Cadmium (Cd^{++}) in drinking water at ppm level. Long Period gratings (LPG) were fabricated by point-by-point technique with CO_2 laser. We have characterized the LPG concentration sensor sensitivity for different solutions of Cd concentrations varying from 0.01 ppm to 0.04 ppm by injecting white Light source and observed transmitted spectra using Optical Spectrum Analyzer (OSA). Proper reagents have been used in the solutions for detection of the Cd species. The overall shift in wavelength is 10 nm when surrounding medium gradually changed from water to 0.04 ppm of cadmium concentrations. A comparative study has been done using sophisticated spectroscopic atomic absorption spectrometer (AAS) and Inductively Coupled Plasma (ICP) instruments. The spectral sensitivity enhancement was done by modifying grating surface with gold nanoparticles. *Copyright © 2011 IFSA.*

Keywords: Long period grating (LPG), Point by Point technique, Chemical sensor, Cadmium, Aqueous gold nanoparticles.

1. Introduction

Regular supply of drinking water to the growing population is really tough task. A number of measures like digging bore wells to get ground water are being taken on large scale. The accuracy in detection and determination of concentration of chemicals / contaminants (especially in ppm / ppb level) present in the ground water are very important to meet the drinking water standards of World Health Organization (WHO). If the concentration of dissolved chemicals in water crosses its limit, it may lead to harmful effects on human health. Out of many dissolved chemicals, Cadmium is an important one. A very large amount of cadmium is released into the environment every year (about 25,000 tons a year). About half of this is released into rivers through weathering of rocks and some cadmium is released into air, through forest fires and volcanoes. The rest of the cadmium is released through human activities, such as manufacturing, zinc ore processing etc. Contamination of drinking-water may occur as a result of the presence of cadmium as an impurity in the zinc galvanized pipes or cadmium-containing solders in fittings, water heaters, water coolers and taps. Short-term consumption (over days or weeks) of high levels of cadmium (Cd) through drinking water can cause nausea, vomiting, and diarrhea, while long-term (over years or decades) consumption may cause kidney dysfunction and osteoporosis [1, 2]. As per World Health Organization, the maximum permissible limit of Cd in water is 0.005 ppm. So, precise measurement of Cd in drinking-water at low concentration is very important.

Many of the methods like chemical, electrochemical and spectroscopy methods are adopted in finding the concentration of dissolved chemicals in water [3]. Atomic Absorption Spectroscopy (AAS) is one of the techniques for determining the concentration of a particular metal element within a sample. The sample is atomized in the flame, through which radiation of a chosen wavelength is sent. A disadvantage of the AAS technique is the non linearity of the calibration curves when absorbance becomes higher than 0.5 to 1 and its detection limit is 0.001 ppm. The relative standard deviations are between 0.3 % and 1 % for absorbance of 0.1 to 0.2. Detection limits for flame AAS vary enormously from 1 - 5 ppb to more than 1000 ppb and hence some elements cannot be measured at all. Inductively Coupled Plasma (ICP) is another technique used for the detection of trace metals in samples. But certain unstable elements require special facilities for handling the radioactive fume of the plasma. Its detection limit is 0.002 ppm. Also, an ICP has difficulty in handling halogens special optics for the transmission of the very short wavelengths become necessary [3, 4].

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 [5-7]. Recently Long Period Grating (LPG) sensors have attracted considerable attention. They are mainly produced by a periodic refractive index change in the core of an optical fiber. In LPG coupling is between the modes traveling in the same direction, which are very sensitive to changes of the external environment [8, 9]. This character allows its use without etching the cladding, being more attractive than other fiber optic sensors [10, 11].

The unique sensitivity to the external refractive index of a Long Period Fiber Grating (LPFG) structure has invoked some discussion on using LPFGs for chemical/biochemical sensing. Patrick et al. demonstrated that LPFGs could be used in effectively measuring the concentration of ethylene, a principal compound, in the antifreezer [9]. They fabricated LPFG of grating 275 μm could detected a tiny change (less than 1 %) in that concentration. Allsop and co-workers [12] performed the detection of organic aromatic compounds in paraffin by an LPFG sensor with optimized sensitivity. They suggested that an LPFG approach in this application may be more attractive than the conventional

HPLC and UV spectroscopy methods, as an LPFG could potentially offer in-situ process control measurements for oil refinery industrial applications [12]. Many chemical concentration sensors have been proposed previously based on LPG by many workers [13-16]. In this paper, we have presented the design and development of LPG based concentration sensors for cd^{++} . A Comparative study has been done using Atomic Absorption Spectrometer (AAS) and Inductively Coupled Plasma (ICP) for ground water samples collected in this geological region.

2. Theory of LPG Operation

In 1996 Vengsarkar et al. reported the principle of operation and some characteristics of long period gratings (LPG) [8]. LPG is a periodic modulation of the refractive index in the fiber core typically has a period 100 micrometer to 1 mm and length of a few cm. LPG uphold coupling between the propagating core mode and co-propagating cladding modes as shown in Fig. 1. The high attenuation of cladding modes results in the transmission spectrum of the fiber containing a series of attenuation bands centered at discrete wavelengths, each attenuation band corresponding to the coupling to different cladding mode. Phase matching between the mode propagating in the core of the fiber and a co-propagating cladding mode is achieved at the wavelength λ_m is [8]

$$\lambda_m = (n_{co} - n_{cl}^m) \Lambda, \quad (1)$$

where λ_m is the peak wavelength of the resonance band between the core mode and the cladding mode. n_{co} and n_{cl}^m are the effective refractive indexes of the core mode and of the m^{th} order cladding mode respectively. Λ is the period of LPG. The minimum transmission of the attenuation bands is governed by the expression [13].

$$T_i = 1 - \sin^2(\kappa_m L), \quad (2)$$

where L is the length of the LPG and κ_m is the coupling coefficient for the m^{th} cladding mode.

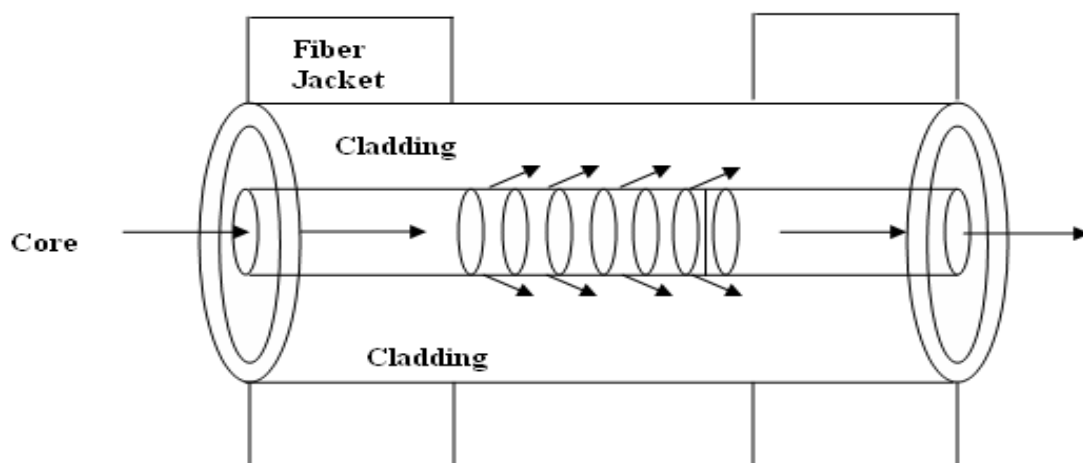


Fig. 1. Coupling of modes in Long Period Grating.

The refractive index sensitivity of LPGs arises from the dependence of the phase matching condition upon the effective refractive index of the cladding modes. LPG is very useful as a surrounding medium refractive index sensor when the refractive index of the external medium changes. The change in ambient index changes the effective index of the cladding mode and will lead to wavelength shifts of the resonance dips in the LPG transmission spectrum. The effect of refractive index of the surrounding medium on the loss resonance wavelength is expressed by [14]:

$$\frac{d\lambda_m}{dn_{sur}} = \frac{d\lambda_m}{dn_{cl}^m} \left[\frac{dn_{cl}^m}{dn_{sur}} \right], \quad (3)$$

where n_{sur} is the refractive index of the surrounding material. For each cladding mode, the term $\frac{dn_{cl}^m}{dn_{sur}}$ is distinct and hence an LPG is expected to have a strong dependence on the order of the coupled cladding mode.

3. Fabrication of Long Period Grating

Fabrication of long period grating was made point by point technique. The schematic diagram of fabrication of LPG is as in Fig. 2. The experiment configuration consisted of a computer controlled translational stage with optical beam scanner. A white light source in the range of 600-1700 nm and spectrum analyzer were used to monitor the transmission spectrum during the formation of the grating. The singlemode Ge-B photosensitive fiber (Newport F- SBG -15, with step index profile of NA 0.12-0.14, cladding diameter $125 \pm 1 \mu\text{m}$, and operating wavelength 1550 nm) is chosen to form Long Period Grating. The gratings of period $410 \mu\text{m}$ have been made along the fiber of length 1.4 cm using CO_2 continuous laser ($10.6 \mu\text{m}$) of output power $\approx 20 \text{ W}$. For grating of length of 1.4 cm totally 35 shots of laser beam have given during grating formation. Fig. 3 shows the grating transmission spectrum. Diffraction of core guided light into cladding modes produces characteristic transmission minima or dip. This writing method using focused CO_2 laser is highly controllable and is applicable to ordinary telecommunication fiber.

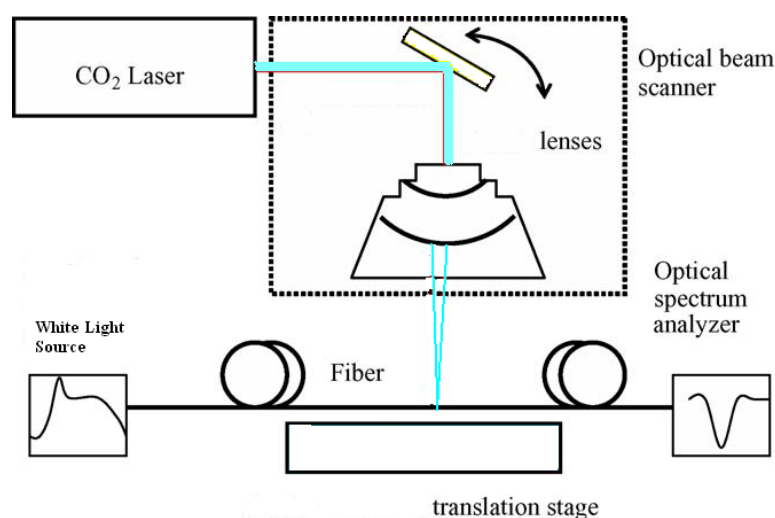


Fig. 2. Schematic diagram of fabrication of LPG.

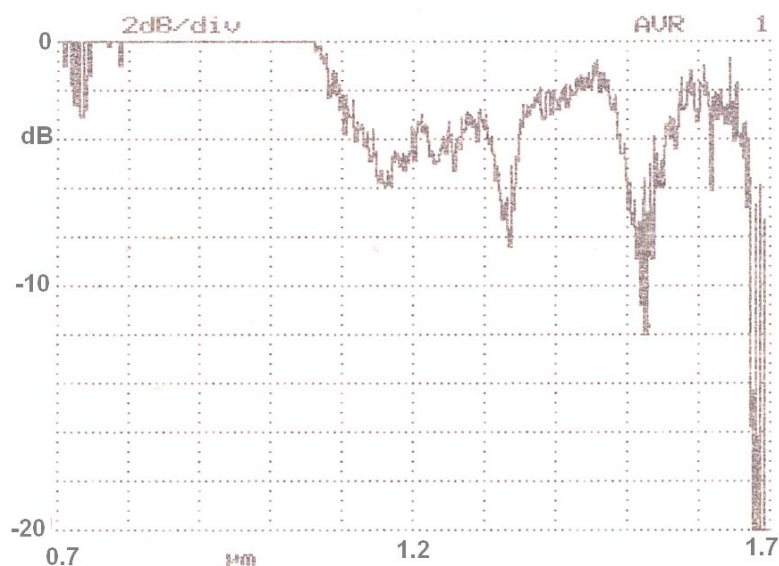


Fig. 3. LPG spectrum in air.

4. Design of Chemical Sensor and Experimental Setup

A schematic diagram of the proposed LPG based concentration sensor is shown in Fig. 4. The fiber containing the LPG element was connected to the white light source (Anritsu MG922) of wavelength 400-1600 nm on one side and to the optical spectrum analyzer (Anritsu MS9001A) 0.6 to 1.75 μm on the other side to record the transmission spectrum. Different solutions of cadmium in the range 0.01 ppm – 0.04 ppm are prepared by dissolving CdSO₄ (Aldrich) in distilled water (Aldrich). 4 ml solutions are taken into different test tubes. To each Cd concentration solution, reagents N,N-dimethylaformamide and thiourea are added in appropriate volumes and let it stand for 5 minutes to complete reaction to take place. The spectra were recorded on the OSA. The LPG sensor head was fixed in a glass cell designed in the laboratory with provision for filling the sample and draining it out when desired. Drastic changes in performance of the LPG had been noted when there were fluctuations in external characteristics like temperature, bending and strain. To avoid the effect of strain and bending a special glass cell holder was designed and the fiber was placed stretched and bonded both the end points of the cell such that the grating section was kept at the centre of the cell. For precise measurement, the experimental setup and sample solution temperature were maintained constant.

The transmission spectra were recorded for sample mixtures of various concentrations as shown in Fig. 5. Sensor responded to concentration changes as soon as new samples were introduced to the glass cell. At the end of each sample measurement, the grating was cleaned with acetone repeatedly, followed by drying properly, so that the original transmission spectrum of LPG was obtained. The most sensitive dips from the spectra (Fig. 5) are taken for the graph wavelength versus concentration shown in Fig. 6, which is a calibration graph.

Ground water samples (test samples) were collected from different places in the local geographic region. Grating spectra measurements for wavelength identification have been carried out by recording the transmitted spectrum for each water sample by adding proper reagents and hence Cd concentration in each water sample could be determined from the calibrated graph Fig. 6.

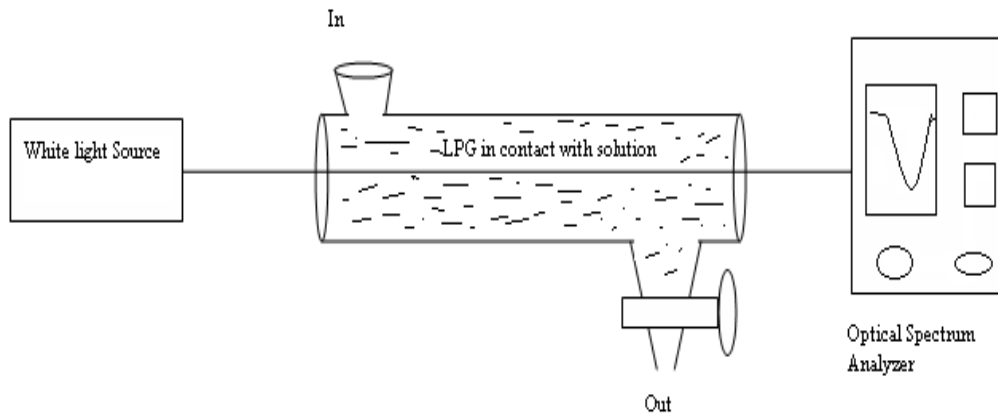


Fig. 4. Experimental setup.

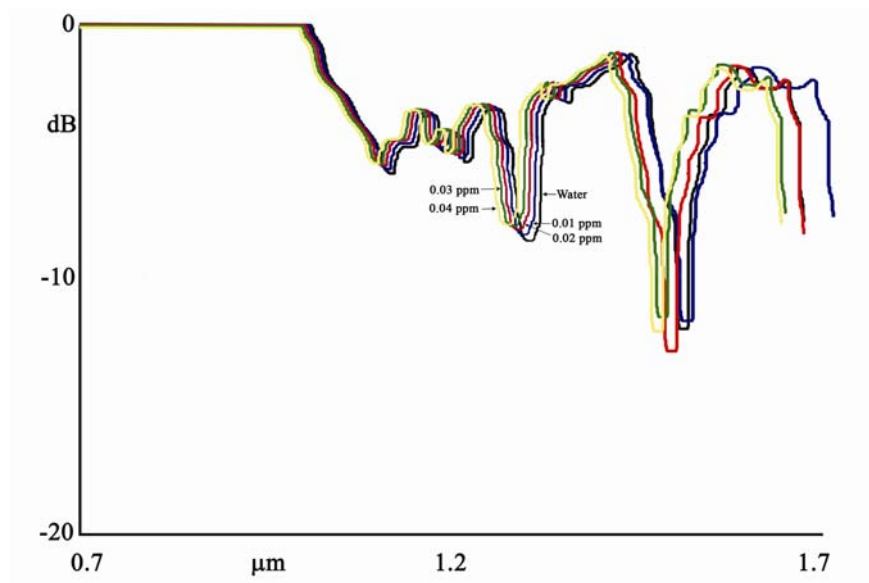


Fig. 5. LPG spectra for 0.01, 0.02, 0.03, 0.04 ppm concentration.

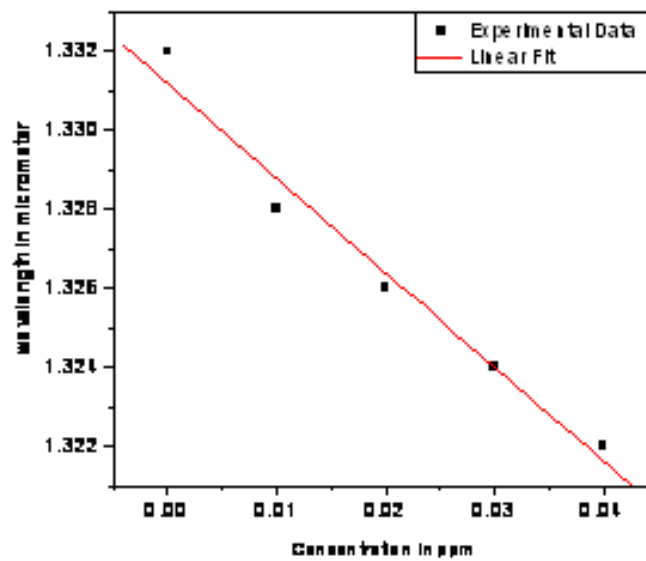


Fig. 6. Wavelength vs. Concentration.

5. Results and Discussion

The spectral response of change in wavelength with concentration is shown in Fig. 5. In the experiment, the LPG resonance wavelengths were monitored, while samples with different concentrations of cadmium were in contact with the grating. For the grating used in our studies the sensitive attenuation peak, in air, are located at 1.336 and 1.526 micrometer and attenuation dips at -8.42 and -11.65 dB respectively as in Fig. 3. If the refractive index of the surrounding medium is lower than that of the cladding, the fiber supports bounded cladding modes that are maintained by total internal reflection at the surrounding cladding interface. In this case, the surrounding refractive index sensitivity arises due to the evanescent wave from cladding interacted with the external medium. From the transmission spectra it is found that with increasing concentrations of cadmium, the sensitive attenuation bands shows blue shift with respect air surrounding medium. The LPG exhibited a total blue shift of approximately 10 nm when the surrounding medium was gradually changed from water to 0.04 ppm of cadmium.

We can determine the concentration of Cd in these samples using graph of transmitted wavelength versus concentration by interpolating the graph Fig. 6. The shift in wavelength is linear with correlation factor of -0.93918. The interpolated graph data are given in the Table 1. The concentration of Cd in collected water sample is found to be 0.002 ppm. A comparative study has been done using atomic absorption spectrometer (AAS) and inductively coupled plasma spectrometer (ICP) and the results are found to be in agreement. The results are given in Table 2. Table 3 provides information on detection limits for Cd element by flame atomic absorption and ICP atomic emission.

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

Parameter	Values
Slope	1.3312 μ m
Intercept	-0.93918
Correlation Coefficient	-0.24

Table 2. Concentration of Cd in test sample as measured using different methods.

Chemical Species	LPG Sensor (ppm)	Atomic absorption Spectrometer (ppm)	Inductive coupled plasma (ppm)	WHO Standard (ppm)
Cadmium	0.002	0 *	<0.03 **	0.005

*measurements were made in USIC (University Scientific Instrument Center, Karnatak University Dharwad, India.)

**Measurements were made at Met – Chem Laboratories, Bangalore. (India Pvt. Ltd.)

Table 3. Detection limits for Cd.

AAS (ng/mL)	ICP (ng/mL)
1 (0.001 ppm)	2 (0.002 ppm)

6. Conclusion

We have fabricated and designed a highly sensitive LPG sensor using B-Ge co-doped fiber to detect the traces of Cd in drinking water. We have also observed the enhancement in sensitivity of chemical sensor by depositing Au nanoparticle layers on LPG. Long Period grating sensors can be used for measuring the cadmium compounds in water with good sensitivity in the lower concentration range. The advantage of this type of sensor is its simplicity in construction and ease of use. An important characteristic of the LPG sensor is the detection on the spectral domain, not influenced by intensities changes of the source. We believe a better sensitivity of our designed sensor, can be achieved by optimization of some key parameters such as grating length, RI profile of the core and optical design.

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