

## ARTICLE

**Cladding Modified Fiber Bragg Grating for Copper Ions Detection****Husam Abduldaem Mohammed<sup>1\*</sup> Aqiel Almamori<sup>1</sup> Ali A. Alwahib<sup>2</sup>**

1. Electronic and Communication Engineering Department, College of Engineering, University of Baghdad, Baghdad, 47202, Iraq

2. Department of Laser and Optoelectronics Engineering, University of Technology, Baghdad, 47202, Iraq

## ARTICLE INFO

*Article history*

Received: 29 September 2021

Accepted: 29 October 2021

Published Online: 31 October 2021

*Keywords:*

Fiber Bragg grating

Optical fiber sensors

Etched fiber

C-band

D-heavy metals

## ABSTRACT

This paper reports a fiber Bragg grating (FBG) as a biosensor. The FBGs were etched using a chemical agent, namely, hydrofluoric acid (HF). This implies the removal of some part of the cladding layer. Consequently, the evanescent field propagating out of the core will be closer to the environment and become more sensitive to the change in the surrounding. The proposed FBG sensor was utilized to detect toxic heavy metal ions aqueous medium namely, copper ions ( $\text{Cu}^{2+}$ ). Two FBG sensors were etched with 20 and 40  $\mu\text{m}$  diameters and fabricated. The sensors were studied towards  $\text{Cu}^{2+}$  with different concentrations using wavelength shift as a result of the interaction between the evanescent field and copper ions. The FBG sensors showed a good response in terms of significant wavelength shift in corresponding to varying  $\text{Cu}^{2+}$  concentrations when immersed in aqueous mediums. The sensors exhibited excellent repeatability towards Cu ions. The results demonstrate that the smaller FBG etching diameter, the better optical response in terms of wavelength and linearity.

**1. Introduction**

Regular exposure of people to heavy metals is critical and causes dangerous effects. Many countries are studying this issue nowadays<sup>[1]</sup>. As well, new sensors manufacturing for sensing heavy metals pollutants have economic worth for people. Furthermore, water sources are quite exposed to heavy metals due to contact with heavy metals. This water is considerably used in daily life. Rivers can be affected by heavy metals. Rivers banks are popular place for heavy metals due to the nature of rivers. Hence, detecting heavy metals in a liquid medium is important research which saves lives and avoids increase in poisoning ration which should not exceed 15 ppb<sup>[2]</sup>. Detection of

heavy metals has been studied in the literature and several methods have been industrialized like atomic absorption spectroscopy (AAS)<sup>[3]</sup>, inductive coupled plasma mass spectrometry (ICPMS)<sup>[4]</sup>, X-ray, anodic stripping voltammetry instrument (ASV)<sup>[5]</sup>, fluorescence spectrometry (XRF)<sup>[6]</sup>, instrumental neutron activation analysis (INAA)<sup>[7]</sup>.

The mentioned analytical techniques are outdated. The main disadvantages of those methods are in general have low selectivity, interference problems high cost and fully-trained operators, high self-absorption of emitted radiations, time-consuming, large samples<sup>[8]</sup>. Hence, a need for a rapid process, low cost and accurate results from the perspective of safety ratio to develop heavy metals sensors such as an optical fiber sensor<sup>[9]</sup>. A fiber Bragg

*\*Corresponding Author:*

Husam Abduldaem Mohammed,

Electronic and Communication Engineering Department, College of Engineering, University of Baghdad, Baghdad, 47202, Iraq;

Email: [husam.a@coeng.uobaghdad.edu.iq](mailto:husam.a@coeng.uobaghdad.edu.iq)

grating (FBG) coated sensor was developed for sensing small concentrations of heavy metals using a range of wavelengths. The cost of the coating surface with gold and sensing materials will not hinder its industrial application. An evanescent wave absorption in the fiber optic sensor was used and developed for sensing heavy metals since 1999 in surface plasmon resonance (SPR) [10].

Recently, many optical multimode fibers tip and tapered were developed. They showed a dynamic response toward heavy metals because of the surface modification on the sensing surface of the fiber [11]. These modifications include sensing materials that have a high surface area, good chemical stability, very low noise and finally a broad range of detecting heavy metals.

Essentially an FBG is an optical fiber (mostly single mode). The refractive index of the FBG has periodic modulation. Successive grating planes reflect the light which is guided along the core of FBG [12]. The portions of the reflected light from these grating planes add constructively for a specific wavelength ( $\lambda_B$ ), when the following condition is satisfied [12,13].

$$\Delta\lambda = 2n_{\text{eff}}\Lambda \quad (1)$$

Where  $n_{\text{eff}}$  is the effective refractive index of the core and  $\Lambda$  is the grating period [13]. The use of FBG-based platforms takes the advantages of optical fiber advantages such as compactness, lightweight, highly compatible with optoelectronic devices (both sources and detectors), multiplexing and remote measurement capability as the signal is spectrally modulated [14,15]. FBGs sensors have been used for measuring load, strain, light and temperature [12,13,16].

On the contrary, standard FBGs confine the light within the fiber core. Therefore, the refractive index is not affected by the surrounding. Modified configurations that allow interaction between evanescent field of the core mode to interact with and the external medium are proposed for FBG sensitivity improvement to the surrounding. A solution is accomplished by in part or entirely removing the fiber cladding through etching, polishing, or by writing FBGs directly in microfibers with a diameter of a few micrometres. In the above cases, the evanescent wave outspreads outside the fiber, and accordingly, the resonance wavelength of the reflected signal depends on the surrounding refractive index that affects the core in value [13]. The Bragg wavelength shift of the FBG sensor ( $\lambda_B$ ) was calculated using the following equation.

$$\lambda_B = \lambda - \lambda_0 \quad (2)$$

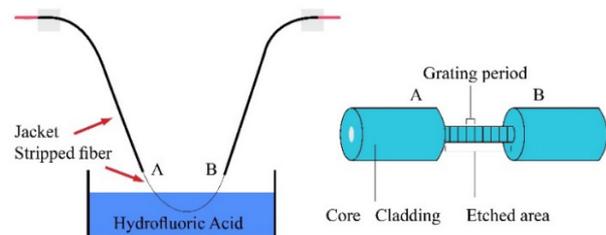
Where  $\lambda_B$  is Bragg wavelength shift,  $\lambda$  is the final Bragg wavelength of sensor after aqueous solution immersion,  $\lambda_0$  is initial Bragg wavelength of sensor before

immersion. In these sensors, the optical properties (i.e. intensity and wavelength) at the fiber and surrounding medium boundary by the electric field of the evanescent wave as a result of the RI variation due to the interaction with the analyte under investigation [13].

Herein, an Etched Fiber Bragg Grating (FBG) based heavy metals sensors are developed and investigated. The developed sensors are etched chemically and inspected towards copper ions ( $\text{Cu}^{2+}$ ) in aqueous medium. FBG core has been exposed via etching was immersed into four concentrations of copper ions solutions (1, 5, 10 and 15 ppm). The FBG sensors showed a significant response in terms of wavelength shift against  $\text{Cu}^{2+}$  concentrations when immersed in aqueous mediums. The sensors exhibited good selectivity towards Cu ions. The results demonstrate that the smaller FBG etching diameter, the better optical response in terms of wavelength shift and linearity selectivity.

## 2. FBG Heavy Metals Sensor Fabrication and Sensing Setup

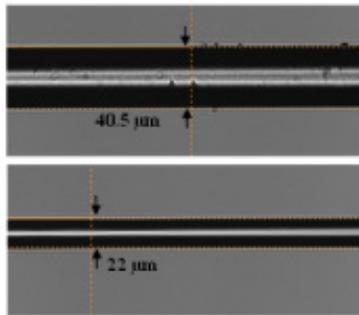
Etched FBG based for heavy metals sensors were fabricated by stripping the FBG area mechanically using stripper and cleaning the stripped area. Some of the cladding layers were removed using a special chemical agent to allow the evanescent waves propagate through it and interact with the sensing layer. The FBG area was immersed in hydrofluoric acid (Aldrich 48%) to reduce cladding thickness as shown in Figure 1. Two FBGs were prepared with 40 and 20  $\mu\text{m}$  diameters by keeping the FBG in HF solution for 64 and 76 s, respectively. The thickness of the etched FBGs was verified using CCD of Vytran workstation machine (GPX-3000, USA) as shown in Figure 2. Etching process increases the surface roughness of the FBGs. Consequently, more sites are available to interact with heavy metal particles in the solution which is under investigation.



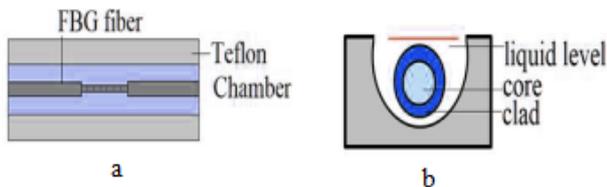
**Figure 1.** Etching FBG using hydrofluoric acid

The modified FBGs were investigated towards  $\text{Cu}^{2+}$  ions with different concentrations ranged ( 0-15 ppm). The deployment of an FBG sensor in the C-band range enables its integration with established optical fiber communication networks such as fiber to the home (FTTH).

FBG based heavy metals sensor was placed in a Teflon chamber, as shown in Figure 3. Test chamber consists of a narrow slit opening from both sides and can hold the optical fiber as well as the analytic solution.

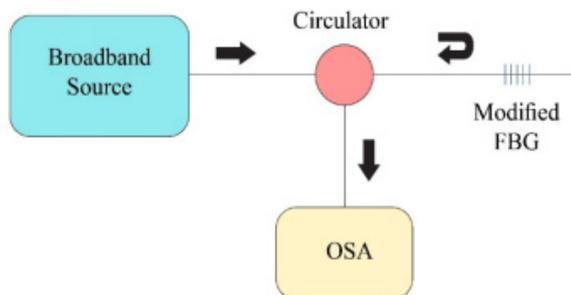


**Figure 2.** Etched FBGs diameter before and after etching process by CCD of Vytran workstation machine



**Figure 3.** FBG fiber attached to the Teflon chamber top view (a) and side view (b)

Figure 4 demonstrates the sensing setup used to investigate the etched FBG sensors towards copper ions. The FBG sensor is connected to a broadband light source (Amonics ALS 18-B-FA, Hong Kong) via a 3-port circulator. The Amonic source has wavelengths ranging from 1520 nm to 1620 nm. The reflected light from the FBG sensor is directed to an optical spectrum analyser (OSA) (AQ6331 Yokogawa, Japan) via the other end of the circulator. Performance of the modified FBG sensors has been studied in terms of FBG center wavelength shift due to the interaction between the heavy metals' ions contained in the solution and evanescent field propagating out of the core area of the FBG.



**Figure 4.** Experimental setup for sensing investigation of FBG based heavy metal detection

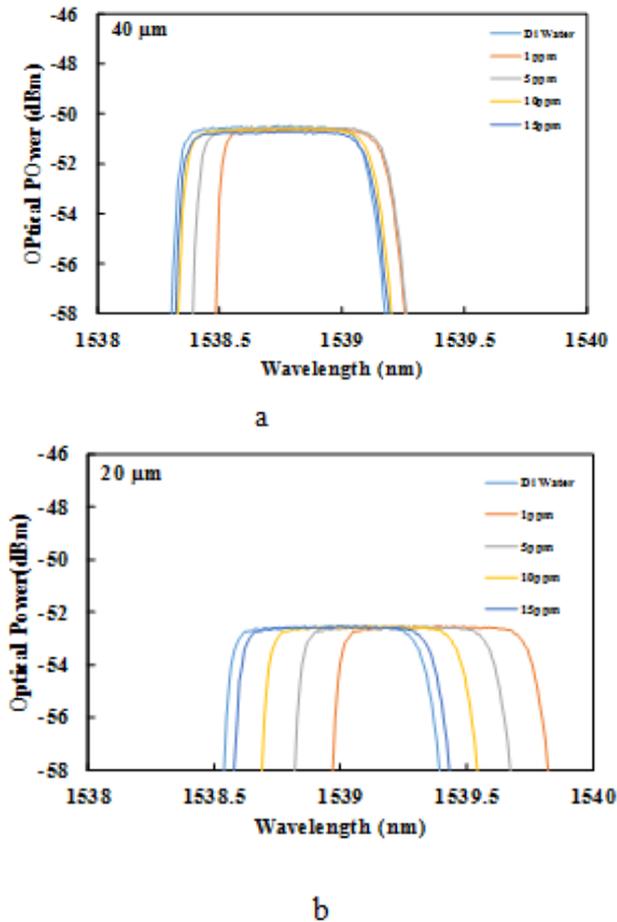
### 3. Results and Discussion

In this section, the response characteristics of the FBGs towards Cu ions were investigated by monitoring the reflected signal from the FBGs. Using optical signal analyzer (OSA). A decent correlation was noticed between the concentrations of copper ions in the water and the reflected optical power during immersing the etched FBG in an aqueous medium. A wavelength shift in reflected optical power from the FBG is found and calculated according to Equation 2. This wavelength shift is because of the interaction between ions and evanescent field extended out of the core dimensions. Figure 5 shows spectral response of the FBGs against different concentrations of Cu ions (1, 5, 10, 15 ppm). Figure 5 (a and b) shows the optical power response when the diameter of etching optical fiber varied between 40  $\mu\text{m}$  and 20  $\mu\text{m}$ , respectively. As can be seen, the wavelength shift increased as the FBG' diameter becomes smaller. For example, a wavelength shift in the response spectrum is found to be 0.13 and 0.43 nm for FBGs with diameters of 40  $\mu\text{m}$  and 20  $\mu\text{m}$ , respectively against 1 ppm ions concentrations. The sensitivity of the refractive index of these sensors strongly depends on the diameter of the fiber within the region that contains the grating. The sensitivity is inversely proportional with fiber diameter. However, this process causes higher fragility and higher difficulties in fiber handling<sup>[12]</sup>. Moreover, the wavelength shift in the response spectrum is found to be increased as the ions concentration is decreased for copper ions and vice versa for lead ions. For example, the measured wavelength shift is observed to be 0.28 and 0.15 nm when the 20  $\mu\text{m}$  etched FBG is immersed in the solution with Cu ions concentration of 5 and 10 ppm, respectively.

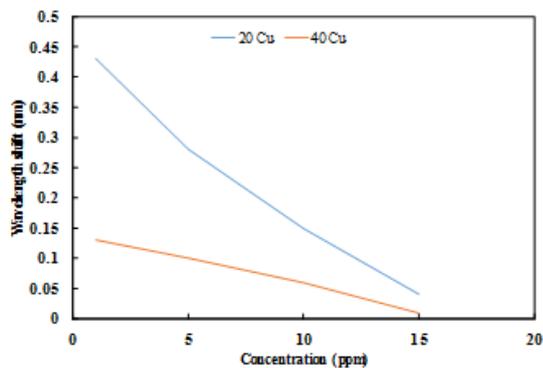
The changes in a wavelength shift of etched FBG against different concentrations of copper ions is shown in this Figure 6. As shown in the figure, the etched FBGs exhibits a linear wavelength shift with ions concentrations at 40  $\mu\text{m}$ . The etched FBG for both diameters demonstrated a decrease in wavelength shift as immersed in Cu ions solutions with higher concentrations. A distinct diameter of etched FBG optical fiber plays a vital role in detections as can be noted from Figure 6.

At low concentrations, the sensor surface area has a dominating role; it allows more ions to interact with the sensing layer. However, at high concentrations, the degradation wavelength shift value towards longer wavelengths could be due to change in refractive index<sup>[17]</sup>. At higher concentrations the dominating effect is refractive index which is faster than ions bonding (at the same time) so the shift is almost identical, i.e., due to impact of surface area. At 40  $\mu\text{m}$  the surface area was 0.001256  $\text{mm}^2$  and

at 20  $\mu\text{m}$  was 0.000314  $\text{mm}^2$ . This explanation cannot be same as copper ion because of the result shows increasing with increasing concentrations. The lead ions have density 11.34  $\text{g}/\text{cm}^3$  compared with 8.96  $\text{g}/\text{cm}^3$ .



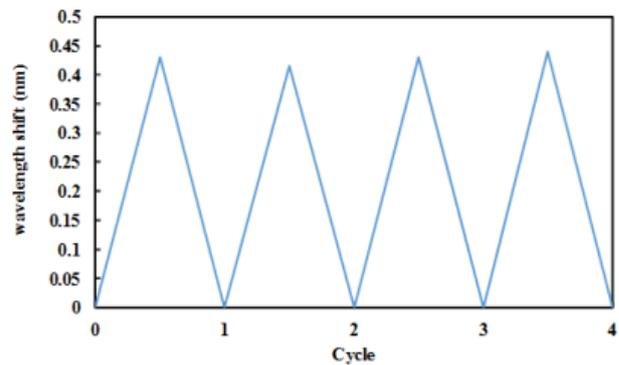
**Figure 5.** Measured optical power as a function of wavelength and etched optical fiber diameter a) 40  $\mu\text{m}$ , b) 20  $\mu\text{m}$  for  $\text{Cu}^{2+}$  sensors



**Figure 6.** wavelength shift at different concentrations of  $\text{Cu}^{2+}$

As shown in Figure 7, recyclability test was performed on etched FBG sensors with 20  $\mu\text{m}$  diameter in 1 ppm of

$\text{Cu}^{2+}$  solution followed by removal of copper ions using distilled water and dried before re-using the FBG as a single complete cycle. As can be noted from Figure 7, the etched FBG sensor exhibited a similar response for the four periods. The wavelength shift for the FBG sensor for the four cycles when immersed in  $\text{Cu}^{2+}$  ion aqueous solution was 0.43, 0.42, 0.43 and 0.44 nm respectively. The FBG sensor may be considered to exhibit good repeatability or reproducibility.



**Figure 7.** Repeatability of the etched FBG in  $\text{Cu}^{2+}$  aqueous solution

## 4. Conclusions

An etched fiber Bragg grating-based sensor was developed for sensing  $\text{Cu}^{2+}$  ions in aqueous solutions with different concentrations. The developed FBG sensors were etched using HF acid with 20 and 40  $\mu\text{m}$ . The FBG-based sensors showed a significant shift in FBG wavelength against  $\text{Cu}^{2+}$  ion concentrations. The smaller the etched FBG diameter, the stronger the optical response in terms of wavelength shift. The sensing of the present sensor is due to a change in refractive index when the sensor was immersed in  $\text{Cu}^{2+}$  ions in aqueous solutions. The developed etched FBG is a significant platform that is sensitive to  $\text{Cu}^{2+}$  heavy metal at a test range with decent repeatability. The potential of development of FBG sensors operating in the C-band is to be integrative easily with the optical fiber communication networks. The compact size of the etched FBG sensor permitted it to be a significant candidate for heavy metal ions detection.

## References

- [1] R. Verma and B. D. Gupta, "Detection of heavy metal ions in contaminated water by surface plasmon resonance based optical fibre sensor using conducting polymer and chitosan," *Food Chem.*, vol. 166, no. 6, pp. 568-575, 2015.
- [2] Y. Zhang, M. Xu, Y. Wang, F. Toledo, and F. Zhou,

- “Studies of metal ion binding by apo-metallothioneins attached onto preformed self-assembled monolayers using a highly sensitive surface plasmon resonance spectrometer,” *Sensors Actuators, B Chem.*, vol. 123, pp. 784-792, 2007.
- [3] P. Niu, C. Fernández-Sánchez, M. Gich, C. Ayora, and A. Roig, “Electroanalytical Assessment of Heavy Metals in Waters with Bismuth Nanoparticle-Porous Carbon Paste Electrodes,” *Electrochim. Acta*, vol. 165, pp. 155-161, 2015.
- [4] Y. W. Fen, W. M. M. Yunus, Z. A. Talib, and N. A. Yusof, “Development of surface plasmon resonance sensor for determining zinc ion using novel active nanolayers as probe,” *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.*, vol. 134, pp. 48-52, 2015.
- [5] K. C. Armstrong, C. E. Tatum, R. N. Dansby-Sparks, J. Q. Chambers, and Z. L. Xue, “Individual and simultaneous determination of lead, cadmium, and zinc by anodic stripping voltammetry at a bismuth bulk electrode,” *Talanta*, vol. 82, pp. 675-680, 2010.
- [6] Y. W. Fen and W. M. M. Yunus, “Surface plasmon resonance spectroscopy as an alternative for sensing heavy metal ions: a review,” *Sens. Rev.*, vol. 33, pp. 305-314, 2013.
- [7] R. K. Dutta, S. Sarkar, S. S. Ram, M. Sudarshan, R. Acharya, and a. V. R. Reddy, “Applications of EDXRF and INAA techniques for studying impact of industries to the environment,” *J. Radioanal. Nucl. Chem.*, vol. 302, pp. 1519-1523, 2014.
- [8] G.-H. Yao, R.-P. Liang, C.-F. Huang, Y. Wang, and J.-D. Qiu, “Surface plasmon resonance sensor based on magnetic molecularly imprinted polymers amplification for pesticide recognition,” *Anal. Chem.*, vol. 85, no. 24, pp. 11944-11951, 2013.
- [9] A. M. Shrivastav, S. P. Usha, and B. D. Gupta, “Fiber optic profenofos sensor based on surface plasmon resonance technique and molecular imprinting,” *Biosens. Bioelectron.*, vol. 79, pp. 150-157, 2016.
- [10] S. S. Yee, “Surface plasmon resonance sensors: review,” *Sensors Actuators B*, vol. 54, pp. 3-15, 1999.
- [11] C. Caucheteur, M. Loyez, Á. González-Vila, and R. Wattiez, “Evaluation of gold layer configuration for plasmonic fiber grating biosensors,” *Opt. Express*, vol. 26, no. 18, p. 24154, 2018.
- [12] F. Chiavaioli, F. Baldini, S. Tombelli, C. Trono and A. Giannetti, “Biosensing with optical fiber gratings,” in *Nanophotonics* vol. 6, ed, 2017, p. 663.
- [13] B. N. Shivananju, P. Pal, S. Yamdagni, M. M. Varma and S. Asokan, “Nanomaterial-coated etched fiber Bragg grating sensors,” in *Workshop on Recent Advances in Photonics (WRAP)*, 2013, pp. 1-2.
- [14] S. N. Abdullah, H. A. Jewad and HA Mohammed, Design Considerations of Laser Source in a Ring Network Based on Fiber distributed Data Interface (FDDI), *Iraqi Journal of Laser*, University of Baghdad, Iraq, part A, Vol. 1, No. 1, 2002, pp.39-46.
- [15] H. A. Mohammed, N. A. Rahman, M. Z. Ahmad, M. H. A. Bakar, S. B. A. Anas, M. A. Mahdi and M. H. Yaacob, “Sensing performance of Modified Single Mode Optical Fiber Coated with Nanomaterials Based Ammonia Sensors Operated in the C-Band,” *IEEE Access*, 2018.
- [16] I. Z. M. Ahad, S. W. Harun, S. N. Gan and S. W. Phang, “Effect of Polymerization Temperatures on Polyaniline Coated Fiber Bragg Grating Sensor for Chloroform Detection,” vol. 382, no. 1, p. 1800088, 2018.
- [17] D. Yang, *Nanocomposite Films for Gas Sensing, Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications*. 2011.