24th IMEKO TC4 International Symposium
22nd International Workshop on ADC and DAC Modelling and Testing
IMEKO TC-4 2020
Palermo, Italy, September 14-16, 2020

Development and characterization of a fiber Bragg grating ethanol sensor for liquids

Antonino Quattrocchi[1], Roberto Montanini[1], Mariangela Latino[2], Nicola Donato[1]

[1] *Department of Engineering, University of Messina, Messina, Italy, aquattrocchi@unime.it, rmontanini@unime.it, ndonato@unime.it*
[2] *Department of MIFT, University of Messina, Messina, Italy, mlatino@unime.it*

***Abstract* – This paper reports some very first results about the development and the metrological evaluation of a fiber Bragg grating (FBG) ethanol sensor, able to work into liquid solutions. The sensing device is established by dip-coating a single-mode silica FBG with a poly(methyl methacrylate) (PMMA) coating film. The thickness of the sensing coating was set by means of a deposition system able to control the rising/dipping rate of the FBG in the PMMA solution. Tests were performed in a range spanning from 3% to 40% in v/v of ethanol in water in order to investigate the device response. In particular, dynamic performance in terms of response and recovery time values were also estimated.**

I. INTRODUCTION

Fiber Bragg grating (FBG) are widely preferred over other types of optical sensors thanks to their unique advantages. They present small sizes, immunity from electromagnetic disturbances, good resistance in terms of temperature and humidity and, above all, are chemically inert. For these reasons, they are commonly employed to develop sensing systems for critical applications, e.g. in an explosive environment. [1]

The sensitivity of FBG to a specific variation can be improved by choosing a suited coating material. This will allow the FBG to be sensitive to other physical quantity in addition to temperature and strain. The basic principle relies in the swallowing of the coating material which eventually can induce a change in the grating period and in the refractive index along the fiber length. Different types of materials and deposition techniques can be used to create a fiber coating for specific applications: Montanini et al. [2] compared results obtained on two polymer-coated FBG humidity sensors, using poly(methyl methacrylate) (PMMA) or poly(vinyl acetate) (PVAc) as sensing materials. The calibration of the two sensors has been carried out in the 20 - 70 %RH range at three different temperatures (15 °C, 30 °C and 45 °C) by a two-pressure humidity generator. The PMMA-coated sensor prototype displayed overall much better performance when compared to the PVAc-coated one, although it manifested a somewhat higher temperature sensitivity. Yeo et al. [3] manufactured a humidity sensor by dip coating in a polyamide solution, while Lupi et al. [4] measured cryogenic temperatures through FBGs, coated with metals (copper, zinc, tin, and lead) by electrodeposition and casting. Another specific application was developed by Campopiano et al [5]. The authors produced several types of hydrophones, using plastic-coated FBGs. They evaluated how the use of polymers, which exhibit great variability in terms of obtainable geometries and acousto-mechanical properties, guarantees the achievement of customized performances.

An important role in industry field is that of FBG sensors adopted in the chemical sector. For example, Cong et al. [6] proposed an optical salinity sensor consisting of a FBG coated with hydrogel. The detection mechanism in this device is based on the mechanical stress induced in the coating of chemically sensitive polymers when water comes out of it. The main limitation of these sensors lies in the slow response due to the balance between the forces acting at the intermolecular level of the coating material.

Furthermore, FBGs have been adopted to realize a concentration sensor of ethanol solution. Latino et al. [7] developed an FBG, coated with a thin layer of poly(methyl methacrylate) (PMMA), to measure low concentration of ethanol in aqueous vapor. PMMA is distributed on the FBG surface with a simple dip coating technique at different concentrations to guarantee a defined coating thickness [8]. Coradin et al. [9] evaluated the performance and the metrological characteristics of four FBGs, assembled in different configuration, for the analysis of water-ethanol mixtures. Raikar et al. [10] employed a Ge-B doped FBG to highlight as the wavelength shift decreases with the increase in liquid solution concentrations, moving from 0% to 50%. Arasu et al. [11] developed an FBG, coated with different thicknesses of thin gold films via sputtering deposition method. Their results displayed as the concentration of ethanol in water is better from 0% to 100% assessed measuring the change in absorbance with the thickest gold layer. Recently, Kumar et al. [12] analysed a graphene oxide-coated FBG for ethanol detection in petrol. This sensor was manufactured covering a partially chemical etched FBG with a layer of an oxidize graphite powder by means of a drop casting technique. The detection of ethanol

proportion in petrol, from 0 to 100%, was investigated and a calibration curve was defined. The main result shows as the graphene oxide-coated FBG allows a significant enhancement (by a factor of ten) in the detection of ethanol in comparison with un-coated FBG.

The aim of this paper is to develop a FBG ethanol sensor for liquid solution using an organic coating. A PMMA coating film has been employed to cover a single-mode silica FBG and to create a sensor able to assess ethanol concentration in water. The performance and the metrological properties of the sensing system have been investigated putting into evidence the calibration procedure and the repeatability of the measurement.

II. EXPERIMENTS

The sensor was developed by coating a single-mode silica FBG with a sensing layer of PMMA. Figure 1 reports a magnification of the used sample, focusing the attention on the sensing layer thickness of 0.44 mm ± 0.03 mm. The active length of the grating was about 14 mm.

Fig. 1. FBG device with PMMA sensing layer.

The deposition procedure was performed as follows: after removing the buffer of the optical fiber, the FBG device was dipped under controlled conditions (constant dipping/rising velocity) in a solution of 560 mg PMMA and 3 ml of acetone. The system, employed in the prototype development, is depicted in Figure 2. The core of the deposition setup is composed of an Agilent E3632A power supply with a 22 mm precision DC servo system motor brushed. In this way, by modulating the power delivered to the motor, it is possible to control the rising/dipping rate of the FBG in the solution ethanol-water.

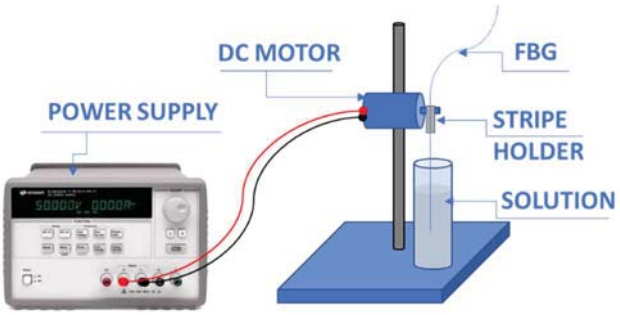

Fig. 2. Block diagram of the deposition setup.

Figure 3 reports the calibration curve of the deposition system (DC motor), in order to achieve a controlled rising/dipping rate to set the thickness of the sensing film on the FBG sensor. The regression shows a linear trend useful to guarantee a uniform coating deposition. By means of this technique it is possible to develop several prototypes equipped with different thickness values of the sensing PMMA film.

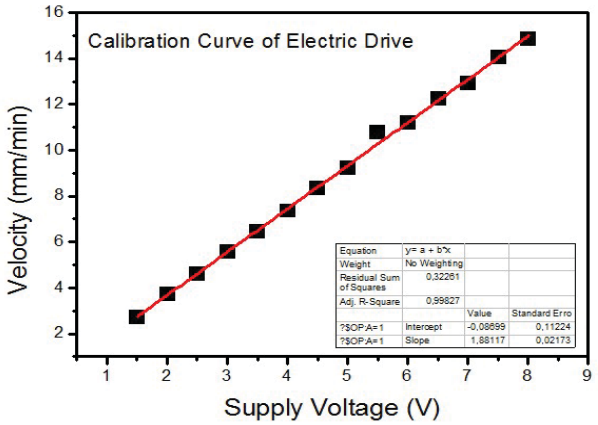

Fig. 3. Calibration curve of the deposition system.

The measurement setup consisted of an optical spectrum analyzer (OSA) (Micron Optics mod. SI 720) with ± 3 pm accuracy, equipped with a National Instruments GPIB-USB-HS IEEE-488.2 communication protocol. A graphical user interface, developed in Labview™ Environment, allowed to display in real time and store the measurement data for further analysis. A block diagram of the measurement setup is reported in Figure 4. The operating temperature value is controlled by means of a thermostatic bath, were finds place a "becker" (a cylindrical container) with the target solution of distilled water and ethanol. This container can be considered as a test chamber were the FBG sensor is put in order to evaluate its sensing performance toward ethanol in water. The thermostatic bath is necessary to avoid the temperature dependence of the device during the entire measurement procedure.

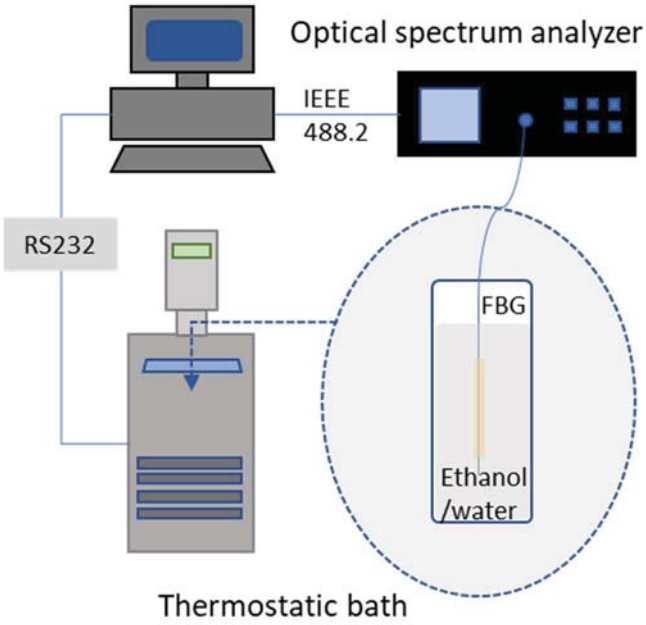

Fig. 4. Block diagram of the measurement setup.

III. RESULTS

Figure 5 shows the power spectra behavior towards ethanol concentration values. It can be clearly seen that the peak shifts with the increasing of ethanol, while the magnitude can be considered sufficiently constant.

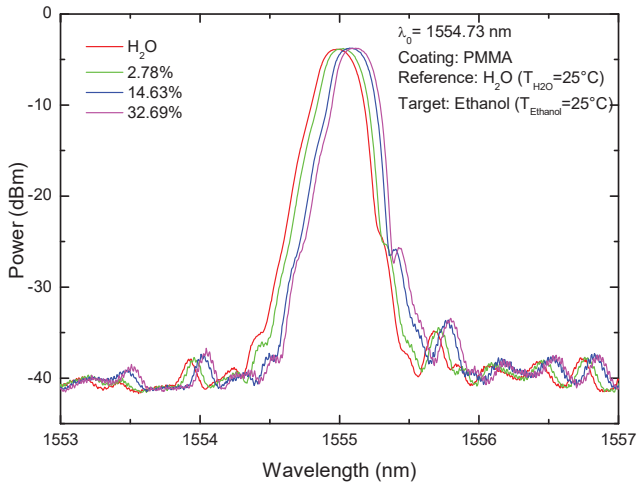

Fig. 5. Power spectra towards ethanol concentration.

The sensor response is defined as the relative wavelength shift:

$$\Delta\lambda = \lambda_R - \lambda_0 \quad (1)$$

where λ_0 is the baseline wavelength in distilled water (0% of ethanol, reference temperature of T_0=25 °C) and λ_R is the wavelength value exposed by the sensor at several ethanol concentration values in distilled water.

Figure 6 displays an example of the data recorded in real-time by increasing the ethanol concentration and by triggering the OSA on the maximum marker. It can be seen how a greater λ_R value is correlated to an increasing of the ethanol concentration. This procedure is useful in order to evaluate the sensor dynamic performance.

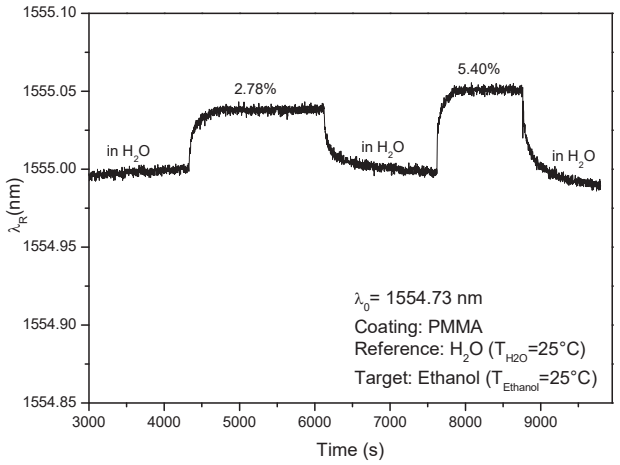

Fig. 6. Sensor λ_R value towards ethanol concentration.

The repeatability features, when coated FBG is at the same concentration value, was also evaluated. In Figure 7 it is reported the λ_R by exposing the sample to three pulses of 7.89 % of ethanol and, then, to distilled water. Both the specific λ_R and the baseline λ_0 values are easily compatible and show good in terms of repeatability and reversibility.

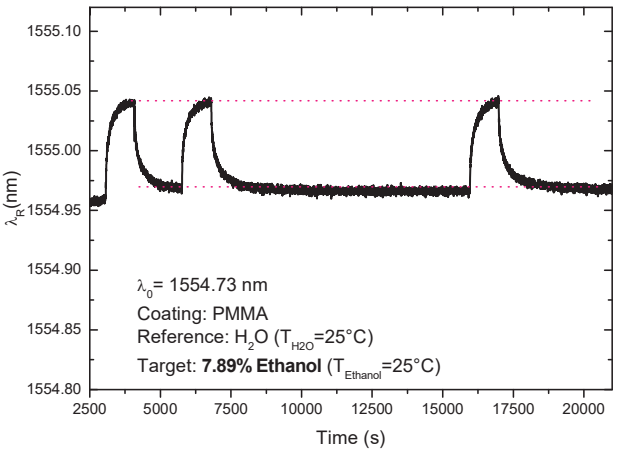

Fig. 7. Sensor repeatability towards a set ethanol concentration value.

Figure 8 highlights the calibration curve. Although a linear behavior for low concentration values can be seen, a polynomial curve is able to fit the data in the whole ethanol range. This trend is similar to those described by [11, 12].

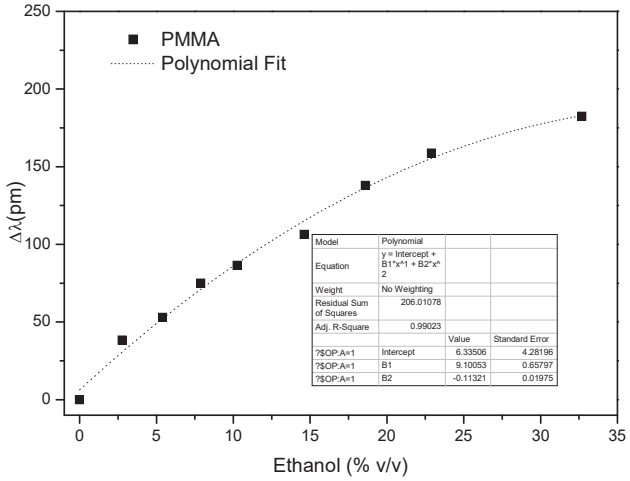

Fig. 8. Calibration curve.

Figure 9 displays the λ_R when the sample is exposed to a transition air-distilled water-ethanol. The considered ethanol concentration value is 7.89 %. The obtained data show how the λ_R reaches a constant value for the set concentration one, while the FBG guarantees the baseline value when exposed again to distilled water.

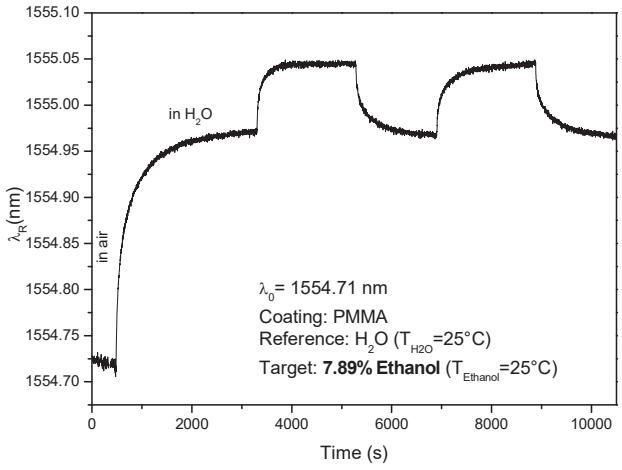

Fig. 9. Transition air-distilled water-ethanol.

Finally, in Figure 10 the response and the recovery time values are plotted. These parameters are respectively calculated as the time to reach the 90 % of the final value when exposed to ethanol and the time to reach the 90 % of the baseline value when exposed to distilled water, respectively. The response time value is of 290 seconds while the recovery one is of 660 seconds. By considering these results, it can be seen how the sensing of ethanol (i.e. the absorbing process) is faster than the desorbing one (the process to reach again the baseline in presence of distilled water, 0 % of ethanol).

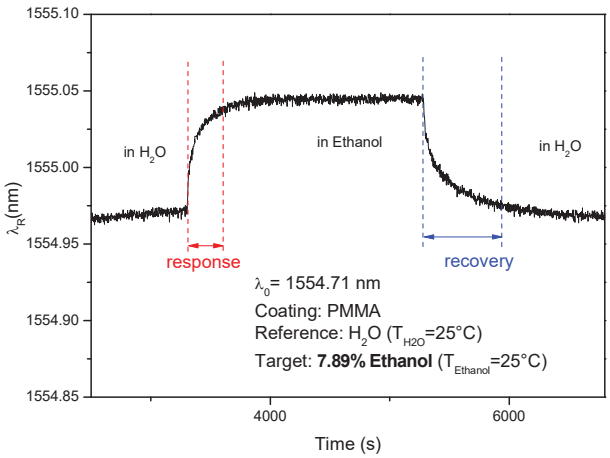

Fig. 10. Response/recovery time values.

IV. CONCLUSIONS

In this paper, we have been reported about the development and characterization of a FBG based sensing device equipped with a PMMA sensing layer. The sensor was specifically manufactured using a dedicated deposition system, able to apply the sensing coating thickness by controlling the rising/dipping of the FBG in the PMMA solution. The measurement data show a clear shift of the wavelength values, strictly related to the increasing of the ethanol concentration in distilled water. The sensing system presents a good repeatability and the calibration curve highlights a polynomial trend that easily and adequately describes the whole range of considered concentration (from 3 % to 40 % in v/v of ethanol in water). The response and recovery times for a fixed concentration of ethanol (7.89 %) were also evaluated, estimating their values.

REFERENCES

[1] R. Montanini, S. Pirrotta, "A temperature-compensated rotational position sensor based on fibre Bragg gratings", Sensors and Actuators A: Physical, vol. 132, is. 2, 2006, pp. 533-540.

[2] R. Montanini, M. Latino, N. Donato, G. Neri, "Comparison between PMMA and PVAC coated fiber Bragg grating sensors for relative humidity measurements", Proc. of 22nd International Conference on Optical Fiber Sensors OFS2012, International Society for Optics and Photonics, 2012, vol. 8421, pp. 842145.

[3] T.L. Yeo, T. Sun, K.T.V. Grattan, D. Parry, R. Lade, B.D. Powell, "Characterization of a polymer-coated fibre Bragg grating sensor for relative humidity sensing", Sensors and Actuators B: Chemical, vol. 110, is. 1, 2005, pp. 148-156.

[4] C. Lupi, F. Felli, A. Brotzu, M.A. Caponero, A. Paolozzi, “Improving FBG sensor sensitivity at cryogenic temperature by metal coating”, IEEE Sensors Journal, vol. 8, is. 7, 2008, pp. 1299-1304.
[5] S. Campopiano, A. Cutolo, A. Cusano, M. Giordano, G. Parente, G. Lanza, A. Laudati, “Underwater acoustic sensors based on fiber Bragg gratings”, Sensors, vol. 9, is. 6, 2009, pp. 4446-4454.
[6] J. Cong, X. Zhang, K. Chen, J. Xu, “Fiber optic Bragg grating sensor based on hydrogels for measuring salinity”, Sensors and Actuators B: Chemical, vol. 87, is. 3, 2002, pp. 487-490.
[7] M. Latino, R. Montanini, N. Donato, G. Neri, “Ethanol sensing properties of PMMA-coated fiber Bragg grating”, Procedia Engineering, vol. 47, 2012, pp. 1263-1266.
[8] D. Sengupta, M.S. Shankar, P.S. Reddy, R.S. Prasad, K.S. Narayana, P. Kishore, “An improved low temperature sensing using PMMA coated FBG”, Prof. of Asia Communications and Photonics Conference and Exhibition, Optical Society of America, 2011, vol. 1, pp. 831103.
[9] F.K. Coradin, G.R. Possetti, R.C. Kamikawachi, M. Muller, J.L. Fabris, “Etched fiber Bragg gratings sensors for water-ethanol mixtures: A comparative study”, Journal of Microwaves, Optoelectronics and Electromagnetic Applications, vol. 9, is. 2, 2010, pp. 131-143.
[10] U.S. Raikar, V.K. Kulkarni, A.S. Lalasangi, K. Madhav, S. Asokan, “Etched fiber Bragg grating as ethanol solution concentration sensor”, Optoelectronics and Advanced Material, vol. 1, 2007, pp. 149-151.
[11] P. Arasu, A.S.M. Noor, A.A. Shabaneh, S.H. Girei, M.A. Mahdi, H.N. Lim, H.A. Abdul Rashid, M.H. Yaacob, “Absorbance properties of gold coated fiber Bragg grating sensor for aqueous ethanol”, Journal of the European Optical Society-Rapid publications, vol. 9, 2014, pp. 14018-1-5.
[12] P. Kumar, S. Kumar, J. Kumar, G. S. Purbia, O. Prakash and S. K. Dixit, “Graphene-oxide-coated fiber Bragg grating sensor for ethanol detection in petrol”, Measurement Science and Technology, vol. 31, 2019, number 2.