Calibration of Etched Fiber Bragg Grating Sensor Arrays for Measurement of Molecular Surface Adsorption

Renilkumar Mudachathi, B. Nanjunda Shivananju, Gurusiddappa R. Prashanth, Sundararajan Asokan, and Manoj M. Varma

***Abstract*—Etched Fiber Bragg Grating (EFBG) sensors are attractive from the point of the inherently high multiplexing ability of fiber based sensors. However, the strong dependence of the sensitivity of EFBG sensors on the fiber diameter requires robust methods for calibration when used for distributed sensing in a large array format. Using experimental data and numerical modelling, we show that knowledge of the wavelength shift during the etch process is necessary for high-fidelity calibration of EFBG arrays. However as this approach requires the monitoring of every element of the sensor array during etching, we also proposed and demonstrated a calibration scheme using data from bulk refractometry measurements conducted post-fabrication without needing any information about the etching process. Although this approach is not as precise as the first one, it may be more practical as there is no requirement to monitor each element of the sensor array. We were able to calibrate the response of the sensors to within 3% with the approach using information acquired during etching and to within 5% using the post-fabrication bulk refractometry approach in spite of the sensitivities of the array element differing by more than a factor of 4. These two approaches present a tradeoff between accuracy and practicality.**

***Index Terms*—Etched fiber Bragg grating, FBG sensor arrays, fiber array based biosensing.**

I. Introduction

SENSOR arrays functionalized with differential receptors which generate unique binding patterns for distinct proteins, small molecules and cells have attracted considerable recent attention due to the possibility of highly specific detection of the target molecule based on their binding pattern [1]–[4]. Most of the prior work has been done with fluorescence or absorbance measurements, sometimes in conjunction with nanoparticle labels. Much work remains to be done in the development of label-free sensor array platforms with high multiplexing capability. In this context, fiber based sensors offer great promise due to the inherently high multiplexing ability of the fiber [5], [6]. Although Fiber Bragg Grating (FBG) sensors have conventionally been used for strain and temperature sensing [7]–[10], recently Etched Fiber Bragg Grating (EFBG) sensors, tilted fiber Bragg grating (TFBG) sensors and Long Period Grating (LPG) sensors have been widely used for refractive index sensing of the surrounding medium [11]–[19]. Normal FBG's are insensitive to the ambient medium due to the thick cladding which inhibits the interaction of the optical field with the surrounding medium. This interaction can be increased by thinning down the cladding. The interaction of EFBG with the surrounding medium was first demonstrated by Dong *et al.* [20]. EFBGs have also been used for measuring molecular surface adsorption, though less widely, compared to their use for bulk refractometry [21], [22]. For example, Saini *et al.* has demonstrated the detection of a monolayer of 3-aminopropyl-monoethoxydimethyl-silane and polymers of 3-aminopropyl-triethoxysilane on the surface of an EFBG [23]. For bulk index sensing, refractive index resolution of 10^{-5} has been reported by Ladicicco *et al.* [24] and a higher sensitivity sensor with an index resolution of 7.2×10^{-6} has been reported by Athanasios *et al.* [25], in which the fiber was etched down to 3.4 μm exposing the core to the surrounding medium.

We had recently done a systematic quantitative study of EFBG sensor performance for measuring surface molecular adsorption in real-time and showed that the limit of detection (LoD) of EFBGs can attain and even surpass prominent optical label-free molecular sensing techniques such as Surface Plasmon Resonance (SPR) when the fiber is etched down below 2 microns diameter [26]. In this paper, we analyze the problem of calibrating a large array of EFBG sensors for high-resolution measurement of analyte binding patterns on the functionalized sensor arrays. As shown in our previous work and by several others [11]–[19], the sensitivity of the EFBG is strongly dependent on the fiber diameter, particularly in the ultra-thin regime where the sensor exhibits high sensitivity. A variation in fiber diameter, even as small as few tens of nanometers, can significantly affect the sensitivity of the individual EFBG sensors forming the array and therefore the biological fidelity of data from large scale EFBG sensor arrays will be fundamentally limited by the method used to calibrate the sensor elements

Manuscript received January 15, 2013; revised May 27, 2013; accepted June 02, 2013. Date of publication June 07, 2013; date of current version June 19, 2013. This work was supported in part by the Robert Bosch Center for Cyber Physical Systems at the Indian Institute of Science.

R. Mudachathi and G. R. Prashanth are with Center for Nano Science and Engineering (CeNSE), Indian Institute of Science, Bangalore 560012, India.

B. N. Shivananju is with the Department of Instrumentation and Applied Physics, Indian Institute of Science, Bangalore 560012, India.

S. Asokan is with the Department of Instrumentation and Applied Physics, Applied Photonics Initiative, and Robert Bosch Center for Cyber Physical System, Indian Institute of Science, Bangalore 560012, India.

M. M. Varma is with Center for Nano Science and Engineering (CeNSE), and Department of Electrical Communication Engineering, Indian Institute of Science, Bangalore 560012, India (e-mail: mvarma@ece.iisc.ernet.in).

Color versions of one or more of the figures in this paper are available online at http://ieeexplore.ieee.org.

Digital Object Identifier 10.1109/JLT.2013.2266658

forming the array. In this paper, using a numerical model along with experimental data, we show that accurate calibration of an EFBG sensor array requires a precise knowledge of the Bragg wavelength shift during the etching process involved in the EFBG fabrication. Unfortunately, this means that every EFBG sensor which is part of the sensor array must be monitored during its etching process, which could prove to be a bottleneck for large scale manufacturing, due to the relatively long usage of FBG interrogators which relatively expensive. A post-fabrication scheme such as exposing the fabricated sensor array to a solution consisting of molecules that do not bind or adsorb on the EFBG surface would be ideal. This approach does not require the extensive use of interrogators to monitor etching of each sensor and it is also advantageous because the sensor surface needs to be in pristine condition for measurement of molecular interactions and bulk refractometry can be done with calibrated solutions that do not affect the sensor surface. Thus, bulk refractometric measurements using samples that do not affect the sensor surface would be an ideal choice for post-fabrication calibration of the sensor array. However, as the bulk refractometry sensitivity is different from surface adsorption sensitivity of an EFBG sensor, the use of a post-fabrication bulk refractometry based calibration procedure must use the correlation between bulk refractometry and surface adsorption sensitivity for proper calibration. Using experimental data, we show that by converting the bulk refractometry performance of EFBG sensors forming an array into an equivalent surface adsorption performance, we can calibrate the sensors to a reasonably good degree although not as perfect as with prior knowledge of wavelength shift during etching. This means that it is not necessary to monitor each EFBG during the etching process and therefore may be a practical strategy for sensor array calibration.

To perform a systematic study of the two EFBG sensor array calibration methods described above, we measured electrostatic layer by layer (LbL) assembly of polyelectrolyte solutions [27]–[30] simultaneously using three different EFBG sensors with widely varying sensitivities produced by etching the sensors to different diameters. In this manner we were able to get a virtual sensor "array" comprising of 3 sensors which had surface adsorption sensitivity varying by more than a factor of 4. In spite of all the elements of this sensor array measuring the same adsorption phenomena, the sensor with higher sensitivity will produce larger signal change as expected. The purpose of the calibration process is to scale the data such that all sensors in spite of their sensitivity differences should yield the same molecular information. We used a finite element (FEM) numerical model of surface adsorption on the EFBG sensor [26] made using commercially available COMSOL multiphysics package to quantitatively analyze the Bragg wavelength shifts from these sensors. Although several simpler and more efficient methods such as multilayer matrix method are available [31], [32] for modelling the adsorption on EFBGs, we used COMSOL as we will be simulating multiphysics phenomena related to molecular sensing using these sensors in future work. We evaluated two calibration methods, one requiring the knowledge of Bragg shift during etching, and another which doesn't require this knowledge, using the model. The rest of the paper presents the details of the evaluation of calibration methods for EFBG sensor arrays using the model and our 3 element sensor array.

Fiber based approaches for sensing are very attractive from the points of view of highly multiplexed detection with low cross-talk, ready availability of a large number of components from the mass produced fiber optic communication industry, compact footprint, and relatively few fabrication steps related to sensor development as the fiber itself acts as the sensor [33]. In the context of fiber sensors, the development of Fiber Bragg Gratings (FBGs) inscribed on photosensitive silica has been advantageous due to the extreme sensitivity of the resonantly reflected Bragg wavelength on the FBG parameters. Mathematically, the Bragg wavelength λ_b is related to the FBG parameters by the well known relation [34].

$$\lambda_b = 2n_{eff}\Lambda \tag{1}$$

Here, n_{eff} is the effective refractive index of the optical mode propagating inside the grating inscribed optical fiber, and Λ is the grating pitch. FBG sensors have been extensively used for strain and temperature sensing where the grating pitch, Λ, is modified by these variables leading to a shift in the Bragg wavelength. Due to the absence of interaction of the guided optical mode with ambient environment, unmodified FBG sensors are not sensitive to surrounding refractive index (SRI). In order to make the FBGs respond to changes in SRI, one can etch the clad region as demonstrated by various groups [11]–[16] or use cladding modes in long-period gratings [17], [18] which interact with surrounding medium or use tilted FBG [19]. Etched FBGs (EFBGs) with single or few propagating modes in the core, provide a simpler output signal for spectral peak detection compared to LPGs which usually have several modes in the operating spectral band. Although several groups have demonstrated measurement of bulk refractive index changes, relatively few have focused on measuring surface adsorption processes using EFBGs. Among them Saini *et al.* have done pioneering work not only in the measurement of surface adsorption phenomena of charged polymers and DNA [22], but also proposed clever designs permitting the simultaneous measurement of refractive index, strain and temperature [15]. However, there has been no report of a carefully modeled system, where experimentally observed signals from EFBG sensors have been compared with an analytical or numerical model to quantitatively analyze the sensor data and to estimate the limits of performance based on such a validated model. We used a model system, namely, the electrostatic layer by layer (LbL) assembly of polyelectrolyte multilayers (PEMs) in conjunction with numerical finite element modeling of the optical propagation in EFBGs to quantitatively study the LbL molecular surface adsorption process using EFBGs. Such a quantitatively rigorous study has so far been lacking in the limited literature dealing with measurement of surface adsorption with EFBGs. We measured LbL of weak polyelectrolyte systems at two different pH values and showed that the assembly process is non-linear and pH dependent as reported in previous literature [29], [30]. Using the numerical model we could rigorously verify that the non-linear growth measured by the EFBG sensors is not an artifact related to the

measurement technique but an accurate reflection of the actual non-linear growth regime of these polymers. The numerical model was then used to determine the theoretical limits of detection with EFBGs with typical experimental noise figures encountered in current FBG interrogation systems. The sensitivity of EFBGs increases with decreasing fiber radius. Based on our theoretical estimates, EFBGs etched below 2 microns in diameter can achieve similar detection limits reported for currently dominant techniques for real-time monitoring of molecular interactions such as Surface Plasmon Resonance (SPR). In addition to this competitive limit of detection, fiber based sensors have an additional advantage of high multiplexing capability as mentioned before. Surpassing the limits of detection reported for SPR using EFBG sensors is limited by the challenges of maintaining mechanical robustness in ultra-thin fibers. In this context, techniques such as simultaneous measurement of strain and surface adsorption as described by Saini *et al.* [15], may enable the compensation of mechanical distortions of ultra-thin fibers enabling robust measurement of molecular signals. Also, cleverly designed packaging and fluid handling systems may enable the use of high fidelity ultra-thin fiber sensors. In the rest of the paper, we describe the details of our experiment and numerical modeling along with the results and discussion.

II. Materials and Methods

A. FEM Model of EFBG

An FBG is a periodic photo-induced permanent modulation of refractive index along the length of the core of a fiber optic cable, which is formed by exposing the fiber to an UV interference pattern. The incident broadband light undergoes Bragg scattering and only a narrow band of the incident light is reflected back due to the constructive interference of Bragg scattered light from each interface. An FBG is characterized by its pitch or periodicity Λ of refractive index modulation and effective refractive index n_{eff} of the waveguide mode and the resultant Bragg resonance is a sensitive function of Λ and n_{eff} as shown in the (1).

In the case of refractive index and bio sensors based on FBG's, the working principle relies on the Bragg wavelength shift induced by the change in n_{eff} upon interaction between the evanescent field of the core mode and the surrounding medium. The sensitivity of such sensors directly depends on the penetration of evanescent field in to the surrounding medium and this interaction can be increased by etching the cladding down to the core causing the mode to expand out of the core and to interact strongly with the surrounding medium, which perturbs the effective index of the propagating mode. To model the effect of bulk refractive index change as well as surface adsorption of molecules on the EFBG surface, we used a finite element (FEM) model, shown in Fig. 1 and described in our previous work [26]. The FEM model was created using commercially available multiphysics package COMSOL. Referring to Fig. 1, n_1, n_2, n_p, and n_{out} are the refractive indices and a, b, c and d are the radii of fiber core, cladding, adsorbent and outer medium respectively. The n_{eff} of the fundamental core mode for surrounding refractive index (SRI) and/or varying thickness of surface adsorbent can be

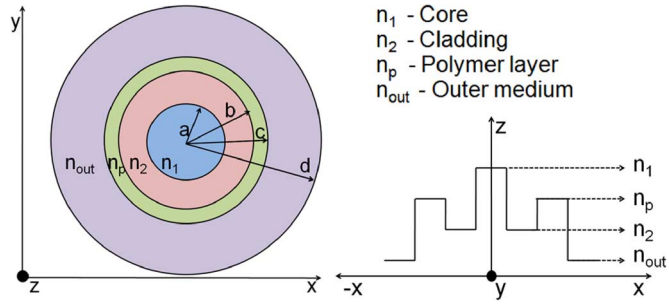

Fig. 1. FEM model used for the numerical analysis.

extracted by a modal analysis using the FEM model and the corresponding Bragg wavelength shift can be estimated using the following equation,

$$\frac{\delta\lambda_B}{\lambda_B} = \frac{\delta n_{eff}}{n_{eff}} \tag{2}$$

The modal analysis is based on the assumption that the n_{eff} change of an etched ordinary fiber will be the same as that of an EFBG since the refractive index perturbation of the latter is only of the order of 10^{-4}. For the modal analysis, the Maxwell's equation is numerically solved by imposing the continuity of the transverse components of electric and magnetic fields across all interior boundaries (interface between core/cladding, cladding/adsorbent and adsorbent/outer medium) and making the tangential component of electric and magnetic field equal to zero at the exterior boundary.

B. Fabrication of EFBG Sensors

The phase mask technique is used for writing the Fiber Bragg Grating [35]. The UV beam from a KrF excimer laser source of 248 nm wavelength passes through the phase mask with approximate pitch of about 1064 nm, over a length of $\mathrm{L} = 3\ \mathrm{mm}$ to form interference pattern, which photo imprints a refractive index modulation (Bragg Grating) in the photosensitive fiber SM1500 from Fibercore Inc., which is placed immediately behind the phase mask. The core diameter of the fiber is about 4.2 $\mu\mathrm{m}$ and the cladding diameter is about 80 $\mu\mathrm{m}$. The refractive index of the core $(\mathrm{n_1})$, obtained from the manufacturer is about 1.4749 where as the cladding refractive index $(\mathrm{n_2})$ is about 1.444. The periodic modulation of the core refractive index is typically of the order of 10^{-4}. The grating periodicity obtained in this manner is about 532 nm.

The sensors were realized by etching the FBG using buffered hydrofluouric (HF) solution to increase the interaction between the core mode evanescent field and the surrounding medium although some groups have used techniques such as plasma etching [36]. As described in our previous work [26], etching process causes a shift in the Bragg wavelength due to change in the effective index as the clad is thinned down. The Bragg shift during etching is a good measure of the fiber diameter and the fiber diameter is a strong determinant of the bulk refractometric as well as surface adsorption sensitivities. The FBG etching process is monitored by the shift in Bragg wavelength and the etching process is stopped when the Bragg wavelength shifts by a pre-set value (say 2 nm, 3 nm and so on).

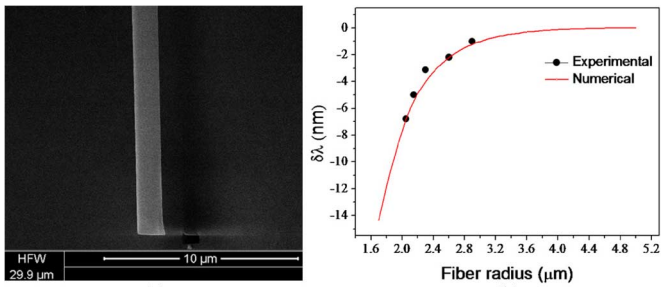

Fig. 2. (a) SEM of a thinned down FBG and (b) Comparison of the experimental and computed Bragg shifts as a function of fiber radius. The good agreement between the experimental values and computed values serves to validate the numerical model.

Using this procedure, we fabricated EFBG sensors of different sensitivities by etching the FBGs to yield Bragg shifts from 1 nm to about 6 nm. We also modelled the etching process using the FEM model by varying the clad diameter. The measured fiber diameters of these EFBGs, obtained using Scanning Electron Microscopy (SEM) were compared with the FEM model results and found to be in good agreement. The SEM of one of the thinned down FBGs is shown in the Fig. 2(a) and the comparison of the experimental data with the FEM numerical model is given in Fig. 2(b).

C. Layer by Layer Assembly of Polyelectrolyte

Polyelectrolytes are polymer groups possessing a charge (positive or negative in solution). Exposing a surface alternatively to cationic and anionic polyelectrolytes causes multilayer buildup on the surface with each layer with thickness around 3–4 nm [27], [28]. This assembly process is called electrostatic layer by layer assembly. Cationic Poly(Allylamine Hydrochloride), PAH with average mol. wt. ∼15 kDa and Anionic Poly(Acrylic acid), PAA with average mol. wt. ∼200 kDa were obtained from Sigma Aldrich. All polyelectrolytes were used as received without any further purification. Polyelectrolyte solutions of 10^{-2} M (based on the repeat unit molecular weight) were made from 18 MΩ DI water. The ionic strength of the polymer solutions was adjusted to 0.01 M NaCl and pH was adjusted to 5.5 with either 0.1 M HCL or 0.1 M NaOH. The EFBG bonded substrate on a custom made holder was first dipped into the 80 ml polycation solution of PAH in a 100 ml beaker for 20 min, followed by dipping the EFBG in DI water for 5 min to remove loosely bound molecules. The EFBG was then immersed into the 80 ml polyanion solution of PAA in a 100 ml beaker for 20 min to adsorb a layer of PAA molecules onto the EFBG followed by the DI water rinse as described before. This process was repeated to obtain the described number of bilayers. An un-etched FBG sensor was used as a reference for any temperature fluctuation during the experiment.

D. Measurement of Polyelectrolyte Assembly Using an EFBG "Sensor Array"

To understand the process of EFBG sensor array calibration, we monitored the electrostatic layer by layer assembly

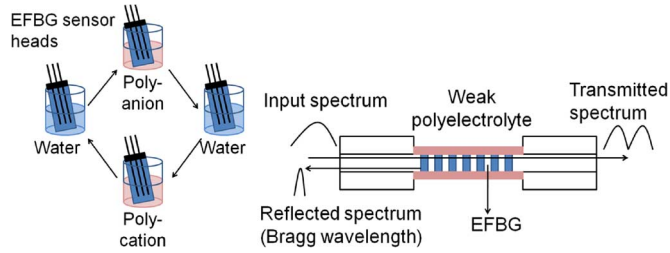

Fig. 3. Schematic of the experimental test setup used for evaluating methods for calibrating sensors arrays using simultaneous measurement of electrostatic layer-by-layer assembly of polyelectrolyte layers using a "sensor array" comprising of 3 sensors with varying sensitivities.

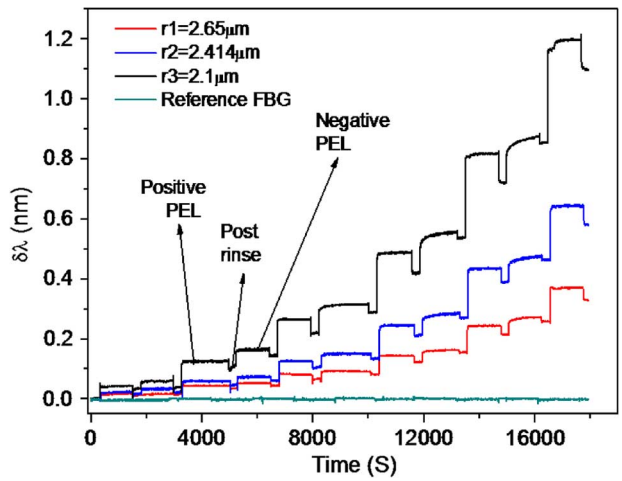

Fig. 4. Experimentally measured Bragg shift as a function of time for the LbL growth of polyelectrolyte on the surface of EFBG measured simultaneously using a 3 element sensor array with their sensitivities differing by more than a factor of 4.

of polyelectrolytes simultaneously using 3 different EFBG sensors with widely varying sensitivities. They were fabricated by etching the FBGs to a Bragg shift of 2 nm, 3 nm and 6 nm respectively with fiber radii of 2.65, 2.41 and 2.05 μm respectively. The difference between the lowest and highest sensitivity was more than a factor of 4. We bonded these 3 EFBGs on a single glass substrate along with a temperature reference and deposited polyelectrolyte multilayers using the layer by layer assembly procedure described in the previous section and depicted in Fig. 3.

As all the sensors are monitoring the same surface adsorption process, they should yield the same molecular information even though the raw data they measure will be different due to their sensitivity differences. The ideal calibration procedure should be able to scale the raw data measured by the sensors to yield minimal deviation between the 3 sensors.

III. Results and Discussion

Fig. 4 shows the raw LbL growth data obtained from the 3 EFBG sensors forming the small-scale sensor array. It can also be seen from this figure that the reference sensor shows no wavelength shift compared to the other etched FBGs. This indicates that there was no temperature fluctuation during the experiment.

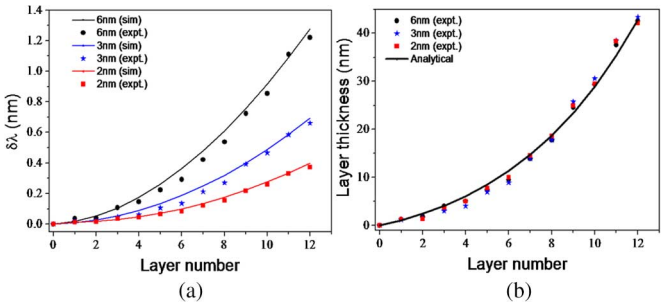

Fig. 5. (a) Comparison between the Bragg shifts observed experimentally (symbols) and those estimated theoretically (lines) using the numerical model for surface adsorption. The model requires the fiber diameter which is estimated using the Bragg shift during etching as shown in Fig. 2(b) and (b) Molecular information extracted using the expression (4) as a function of layer number.

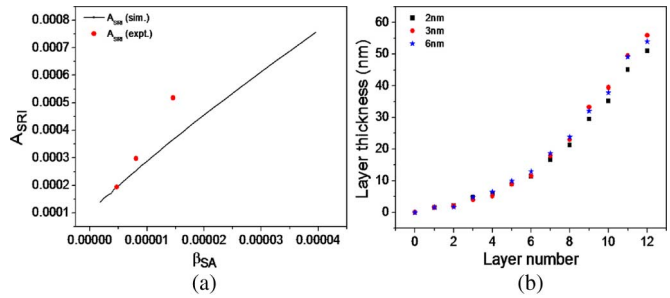

Fig. 6. (a) Correlation between A_{SRI} from (6) and surface adsorption sensitivity from (4) and (b) Layer thickness estimated using the expression (5) with β_{SA} estimated from Fig. 6(a) instead of using Bragg shift during etching.

As we can see even though the sensors are all measuring the same molecular adsorption process, the raw data from the 3 sensors differs by more than a factor of 4 due to a diameter difference of about 1 μm between the thinnest and thickest EFBGs. The calibration method employed, should be able to scale data from different sensors such that they provide the correct molecular information, which in this case implies that the best calibration process will be one that minimizes the variation between the 3 sensor outputs.

As described in our previous work [26], the thickness of the polyelectrolyte multilayers as a function of number of layers can be described by a non-linear (exponential) equation,

$$t(n) = A\left(\exp(Bn) - 1\right) \tag{3}$$

For the polyelectrolyte system considered here, A and B are given by 5.2 and 0.2 respectively [26]; n is the number of layers. Using the thickness calculated using (3); refractive index of the polyelectrolyte layers as 1.52 [37] and EFBG diameters of the 3 sensors as mentioned in Section II-D, we calculated the Bragg wavelength shift using the FEM numerical model. As shown in Fig. 5(a), we noticed an excellent match between the numerical model and the experimental data by varying only the fiber diameter in the numerical model.

We used the numerical model for calibration in the following way. First, we can define a surface adsorption sensitivity β_{SA} as the relative Bragg wavelength shift per unit nm of thickness change in the adsorbed layer.

$$\beta_{SA} = \frac{d\left(\frac{\delta\lambda_b}{\lambda_b}\right)}{dt} \tag{4}$$

Needless to say, β_{SA} is a function of fiber radius, outer medium refractive index and so on. However, this quantity can be obtained from the FEM model. Using the calculated value of β_{SA} one can convert Bragg shift data from EFBGs to a molecular layer thickness t, using the following equation,

$$t = \frac{1}{\beta_{SA}}\left(\frac{\delta n_{eff}}{n_{eff}}\right)_{\exp.} \tag{5}$$

The layer thickness extracted using the above equation as a function of layer number is shown in the Fig. 5(b). It can be seen that all the three sensors give the same molecular information even though the sensors have widely varying sensitivities.

Although this method gives pretty good data normalization, the disadvantage is the requirement to monitor the Bragg shift resulting from the etching for each sensor forming the sensor array. Therefore one would like to explore calibration procedures where data from a post fabrication test, e.g. exposing the entire sensor array to a standard solution consisting of molecules that do not adsorb on the EFBG surface, is used to normalize the raw sensor data. In order to compare this approach with the one described above, we exposed our small-scale sensor network to water $(\mathrm{n} = 1.33)$, ethanol $(\mathrm{n} = 1.36)$ and propanol $(\mathrm{n} = 1.39)$. This data was taken prior to the layer by layer assembly as LbL would modify the EFBG surface whereas the solutions mentioned above do not adsorb on the EFBG surface and leave it pristine for LbL assembly. The bulk refractometric sensitivity scales differently from the surface adsorption sensitivity as a function of fiber radius. Nevertheless they are correlated. We use this correlation to calibrate the sensors responses based on the bulk refractometry data. First we define a quantity A_{SRI}, related to the bulk refractometry sensitivity, as

$$A_{SRI} = \frac{\lambda_b(n_2) - \lambda_b(n_1)}{n_2 - n_1} \tag{6}$$

where $\lambda_b(n_1)$ is the Bragg wavelength observed when bulk index is n_1. We used water-ethanol transition to calculate A_{SRI} with $n_1 = 1.33$ and $n_2 = 1.36$. We then used the model to compute A_{SRI} and β_{SA} as a function of fiber radius to construct the correlation between the two quantities as shown in Fig. 6(a). From the experimentally measured A_{SRI}, we can then estimate the β_{SA} values and use them to calibrate the sensor data according to (5) where the β_{SA} values are now determined from the experimental A_{SRI} values by making use of the correlation of bulk and surface index sensitivity. Calibrating in this manner yields the result shown in Fig. 6(b). Although the performance is not as good as the one shown in Fig. 5(b), we note that in this case we did not require the monitoring of the etching process.

The variation (measured as standard deviation) between the three sensors after the normalization for first, sixth and twelfth layers were 0.96%, 6.8% and 4.6% respectively, for this approach compared to 2.18%, 5.1% and 1.9%. The difference in performance between the two calibration methods is more apparent in the measurement of thicker layers.

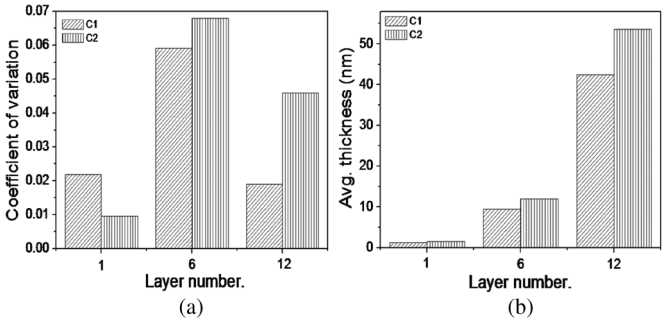

Fig. 7. (a) Coefficient of variation between the three sensors for specific layer numbers estimated using the two calibration methods. and (b) Comparison between the average layer thickness estimated using the two calibration methods.

The Fig. 7(a) and (b) compare the performance of the two calibration approached in terms of the average layer thickness and the coefficient of variation (ratio of standard deviation to the mean) between the three sensors for specific layer numbers measured using the aforementioned calibration approaches. Where C1 represents calibration using Bragg shift information during etching and C2 represents the post-fabrication calibration scheme using correlation of bulk refractometric sensitivity and surface adsorption sensitivity.

Average thickness obtained from C1 is closer to the expectations based on literature [27]–[30]. Similarly the coefficient of variation is also smaller for C1 compared to C2. These results show that knowledge of Bragg shift during etching which gives a good estimate of the fiber radius enables precise calibration of the sensor array. However this requires the monitoring of each sensor element before and after etching.

Alternately one can use a post-fabrication bulk refractometry experiment and use the correlation between bulk and surface index sensitivity for calibration. Although this approach is not as precise as the first one, it may be more practical as there is no requirement to monitor each element of the sensor array. In either case, using the model we were able to calibrate the sensor responses to within 5% even though their sensitivities were different by more than a factor of 4. The average thickness estimated using C1 is closer to the expectation from literature and this method also has lower coefficient of variation compared to C2 which does not use Bragg wavelength shift information during etching.

IV. Conclusions

We analyzed the problem of calibrating an etched fiber Bragg grating sensor array and found that the knowledge of Bragg wavelength shift during etching is necessary for accurate calibration of the sensor array. However as this approach requires the monitoring of every element of the sensor array during etching, we also proposed and demonstrated a calibration scheme using data from bulk refractometry measurements conducted post-fabrication without needing any information about the etching process. The post-fabrication approach eliminates the extensive use of interrogators for array calibration. We were able to calibrate the response of the sensors to within 3% with the approach using information acquired during etching and to within 5% using the post-fabrication bulk refractometry approach in spite of the sensitivities of the array element differing by more than a factor of 4. These two approaches present a tradeoff between accuracy and practicality.

References

[1] M. De, S. Rana, H. Akpinar, O. R. Miranda, R. Arvizo, U. F. Bunz, and V. M. Rotello, "Sensing of proteins in human serum using conjugates of nanoparticles and green fluorescent protein," *Nat. Chem.*, vol. 1, pp. 461–465, Sept. 2009.

[2] O. R. Miranda, B. Creran, and V. M. Rotello, "Array-based sensing with nanoparticles: 'Chemical noses' for sensing biomolecules and cell surfaces," *Curr. Opin. Chem. Biol.*, vol. 14, no. 6, pp. 728–736, Dec. 2010.

[3] S. Rana, A. K. Singla, A. Bajaj, S. G. Elci, O. R. Miranda, R. Mout, B. Yan, F. R. Jirik, and V. M. Rotello, "Array-based sensing of metastatic cells and tissues using nanoparticle fluorescent protein conjugates," *Acs. Nano.*, vol. 6, no. 9, pp. 8233–8240, Aug. 2012.

[4] A. P. Umali and E. V. Anslyn, "A general approach to differential sensing using synthetic molecular receptors," *Curr. Opin. Chem. Biol.*, vol. 4, no. 6, pp. 685–692, Aug. 2010.

[5] B. Lee, "Review of the present status of optical fiber sensors," *Opt. Fiber Technol.*, vol. 9, no. 2, pp. 57–79, Apr. 2003.

[6] B. G. Grossmanny and L. Huang, "Fiber optic sensor array for multi-dimensional strain measurement," *Smart Mater. Struct.*, vol. 7, no. 2, pp. 159–165, Apr. 1998.

[7] A. D. Kersey, M. A. Davis, H. J. Patrick, M. LeBlanc, K. P. Koo, C. G. Askins, M. A. Putnam, and E. Friebele, "Fiber grating sensors," *J. Lightw. Technol.*, vol. 15, no. 8, pp. 1442–1463, Aug. 1997.

[8] A. Othonos, "Fiber Bragg gratings," *Rev. Sci. Instrum.*, vol. 68, no. 12, pp. 4309–4341, Dec. 1997.

[9] S. W. James, M. L. Dockney, and R. P. Tatam, "Simultaneous independent temperature and strain measurement using in-fibre Bragg grating sensors," *Electron. Lett.*, vol. 32, no. 12, pp. 1133–1134, Jun. 1996.

[10] X. Shu, K. Chisholm, I. Felmeri, K. Sugden, A. Gillooly, L. Zhang, and I. Bennion, "Highly sensitive transverse load sensing with reversible sampled fiber Bragg gratings," *Appl. Phys. Lett.*, vol. 83, no. 15, pp. 3003–3005, Oct. 2003.

[11] D. A. Pereira and J. L. Santos, "Fiber Bragg grating sensing system for simultaneous measurement of salinity and temperature," *Opt. Eng.*, vol. 43, no. 2, pp. 299–304, Feb. 2004.

[12] W. Liang, Y. Huang, Y. Xu, R. K. Lee, and A. Yariv, "Highly sensitive fiber Bragg grating refractive index sensors," *Appl. Phys. Lett.*, vol. 86, no. 15, p. 151122, Apr. 2005.

[13] N. Chen, B. Yun, and Y. Cui, "Cladding mode resonances of etch-eroded fiber Bragg grating for ambient refractive index sensing," *Appl. Phys. Lett.*, vol. 88, p. 133902, Mar. 2006.

[14] N. Chen, B. Yun, Y. Wang, and Y. Cui, "Theoretical and experimental study on etched fiber Bragg grating cladding mode resonances for ambient refractive index sensing," *J. Opt. Soc. Am. B.*, vol. 24, no. 3, pp. 439–445, Mar. 2007.

[15] S. M. Lee, S. S. Saini, and M. Y. Jeong, "Simultaneous measurement of refractive index, temperature, strain using etched-core fiber Bragg grating sensors," *IEEE Photonic. Tech. Lett.*, vol. 22, no. 19, pp. 1431–1433, Oct. 2010.

[16] S. M. Lee, M. Y. Jeong, and S. S. Saini, "Etched-core fiber Bragg grating sensors integrated with microfluidic channels," *J. Lightw. Technol.*, vol. 30, no. 8, pp. 1025–1031, Apr. 2012.

[17] S. W. James and R. P. Tatam, "Optical fibre long-period grating sensors: Characteristics and application," *Meas. Sci. Technol.*, vol. 14, pp. 49–61, 2003.

[18] A. Iadicicco, S. Campopiano, M. Giordano, and A. Cusano, "Spectral behavior in thinned long period gratings: Effects of fiber diameter on refractive index sensitivity," *Appl. Opt.*, vol. 46, no. 28, pp. 6945–6952, Oct. 2007.

[19] J. Albert, L. Y. Shao, and C. Caucheteur, "Tilted fiber Bragg grating sensors," *Laser Photonics Rev.*, vol. 7, no. 1, pp. 83–108, 2013.

[20] L. Dong, J. L. Cruz, L. Reekie, and J. L. Archambault, " Tuning and chirping fiber Bragg gratings by deep etching," *IEEE Photonic. Technol. Lett.*, vol. 7, no. 12, pp. 1433–1435, Dec. 1995.

[21] G. Ryu, M. Dagenais, M. T. Hurley, and P. Deshong, "High specificity binding of lectins to carbohydrate-functionalized fiber Bragg gratings: A new model for biosensing applications," *IEEE J. Quantum Electron.*, vol. 16, no. 3, pp. 647–653, 2010.

[22] A. N. Chryssis, S. S. Saini, S. M. Lee, H. Yi, W. E. Bentley, and M. Dagenais, "Detecting hybridization of DNA by highly sensitive evanescent field etched core fiber Bragg grating sensors," *IEEE J. Quantum Electron.*, vol. 11, no. 4, pp. 864–872, 2005.
[23] S. S. Saini, C. Stanford, S. M. Lee, J. Park, P. Deshong, W. E. Bentley, and M. Dagenais, "Monolayer detection of Biochemical agents using etched-core fiber Bragg grating sensors," *IEEE Photon. Technol. Lett.*, vol. 19, no. 18, pp. 1341–1343, Sep. 2007.
[24] A. Iadicicco, A. Cusano, S. Campopiano, A. Cutolo, and M. Giordano, "Thinned fiber Bragg gratings as refractive index sensors," *IEEE Sens. J.*, vol. 5, no. 6, pp. 1288–1295, Dec. 2005.
[25] A. N. Chryssis, S. M. Lee, S. B. Lee, S. S. Saini, and M. Dagenais, "Highly sensitivity evanescent field fiber Bragg grating sensor," *IEEE Photon. Technol. Lett.*, vol. 17, no. 6, pp. 1253–1255, Jun. 2005.
[26] B. N. Shivananju, M. Renilkumar, G. R. Prashanth, S. Asokan, and M. M. Varma, "Detection limit of etched fiber Bragg grating sensors," *J. Lightw. Technol.*, 10.1109/JLT.2013.2262231, (accepted).
[27] J. Choi and M. F. Rubner, "Influence of the degree of ionization on weak polyelectrolyte multilayer assembly," *Macromolecules*, vol. 38, no. 1, pp. 116–124, Dec. 2005.
[28] T. Yamada and S. Shiratori, "In situ observation of the initial absorption process of layer-by-layer sequential adsorbed polyelectrolyte film using an AFM," *Electr. Eng. Jpn.*, vol. 141, no. 2, pp. 1–7, 2002.
[29] P. Bieker and M. Schonhoff, "Linear and exponential growth regimes of multilayers of weak polyelectrolytes in dependence on pH," *Macromolecules*, vol. 43, no. 11, pp. 5052–5059, May 2010.
[30] S. S. Shiratori and M. F. Rubner, "pH dependent thickness behavior of sequentially adsorbed layers of weak polyelecytolytes," *Mactomolecules*, vol. 33, no. 11, pp. 4213–4219, Apr. 2000.
[31] M. Monerie, "Propagation in doubly clad single-mode fibers," *IEEE J. Quantum Electron.*, vol. 18, no. 4, pp. 535–542, Apr. 1982.
[32] T. G. Giallorenzi, J. A. Bucaro, A. Dandridge, G. H. Sigel, JR., J. H. Cole, S. C. Rkshleigh, and R. G. Priest, "Optical fiber sensor technology," *IEEE J. Quantum Electron.*, vol. qe-18, no. 4, pp. 625–645, Apr. 1982.
[33] F. Baldini, M. Brenci, F. Chiavaio li, A. Giannetti, and C. Trono, "Optical fibre gratings as tools for chemical and biochemical sensing," *Anal. Bioanal. Chem.*, vol. 402, no. 1, pp. 109–116, Jan. 2012.
[34] K. O. Hill and G. Meltz, "Fiber Bragg grating technology fundamentals and overview," *J. Lightw. Technol.*, vol. 15, no. 8, pp. 1263–1276, Aug. 1997.
[35] K. O. Hill, B. Malo, F. Bilodeau, D. C. Johnson, and J. Albert, "Bragg grating fabricated in monomode photosensitive optical fiber by UV exposure through a phase mask," *Appl. Phys. Lett.*, vol. 62, no. 10, pp. 1035–1037, Mar. 1993.
[36] H. Kumazaki, Y. Yamada, H. Nakamura, S. Inaba, and K. Hane, "Tunable wavelength filter using a Bragg grating fiber thinned by plasma etching," *IEEE Photon. Technol. Lett.*, vol. 13, no. 11, pp. 1206–1208, Nov. 2001.
[37] C. Picart, "Polyelectrolyte multilayer films: From physico-chemical properties to the control of cellular processes," *Curr. Med. Chem.*, vol. 15, no. 7, pp. 685–697, 2008.

Renilkumar Mudachathi received the B.Sc. degree from Kannur University, Kerala, India, in 2002, and the M.Sc. degree from Bharathidasan University, Tiruchirappalli, India, in 2005, both in physics. He obtained a Ph.D. in Physics from Anna University, Chennai, India, in 2012. He is currently a Postdoctoral fellow at Center for Nano Science and Engineering, Indian Institute of Science, Bangalore, India. His research interests cover integrated silicon photonics, MEMS, and development of micro/nanodevices based on photonic band gap for fiber optics and biomedical sensing applications.

B. Nanjunda Shivananju was born India, in 1984. He received the B. E. degree from Bangalore Institute of Technology, Bangalore, India. He is currently pursuing Ph.D. degree in Instrumentation and Applied Physics, Indian Institute of Science, Bangalore, India. His thesis research was focused on Bio-Chemical sensing by using Fiber Bragg Grating sensor.

Gurusiddappa R. Prashanth received the B.E. degree from Bangalore University in 2000 and M.Tech from Visvesvaraya Technological University, Belgaum in 2004. He is currently a doctoral student in the Department of Electrical Communication Engineering at the Indian Institute of Science, Bangalore. He is working on developing optical applications using layer by layer assembly of polyelectrolytes.

Sundararajan Asokan obtained his Ph.D. from Indian Institute of Science, Bangalore in the Division of Physical and Mathematical Sciences. He is currently a Professor at the Department of Instrumentation and Applied Physics, and Applied Photonics Initiative, and Chairman for Robert Bosch Center for Cyber Physical Systems, Indian Institute of Science, Bangalore. His interests span a variety of topics related to Fiber Bragg Grating Sensors and Phase Change Memory using Chalcogenide glasses.

Manoj M. Varma obtained his Ph.D. from Purdue University in 2005. He is currently an Assistant Professor at the Center for Nano Science and Engineering (CeNSE), Indian Institute of Science, Bangalore. His interests span a variety of topics related to label-free bio-molecular sensing.