Fabrication and characterization of pH sensor based on side polished single mode optical fiber

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Abstract

Fabrication and characterization of side-polished single mode optical fiber pH sensor utilizing evanescent field absorption spectroscopy and dye doped sol-gel immobilization technology are presented. The sensor is prepared by fixing the fiber in a groove made on one of the surfaces of a fused silica block. To make the surface sensitive to pH it is polished up to the core of the fiber and a film containing a mixture of three pH sensitive dyes is coated on it using sol-gel technology. The influence of the radius of curvature of the groove on the sensitivity of the sensor is studied. The decrease in the radius of curvature of the groove increases the sensitivity. This occurs due to the increase in the evanescent field and hence its interaction with the pH sensitive dyes entrapped in the film. The advantage of the side-polished single mode optical fiber sensor is that only 0.1 ml of sample is sufficient to measure the pH of the fluid. This is important when the procurement of the sample in large quantity is not possible.

Keywords: Optical fiber; Sensor; pH; Side polished half block; Sol-gel; Sensitivity

1. Introduction

The measurement and control of pH is required in practically all kinds of sciences including chemical, biomedical and environmental. In recent years, numerous efforts have been directed toward the development of fiber optic pH sensors. This is because fiber optic sensors offer many advantages over other types of sensors. Some of the advantages are their small size, immunity to electromagnetic and radio frequency interference, explosion proof, remote sensing, possibility of multiplexing the information from a large number of sensors in a single fiber and in some cases the low cost.

Optical fiber pH sensors are based on pH-induced reversible changes in optical or spectroscopic properties such as absorbance, reflectance, fluorescence, energy transfer, etc. The major component in an optical fiber sensor is a pH sensitive layer/film that is prepared by immobilizing a pH sensitive dye onto the tip or sides of an optical fiber. To immobilize, many research groups have used sol-gel technology [1-7]. In this technique, a thin film of glass with dye entrapped in it is prepared from the hydrolysis and condensation polymerization of a metal alkoxide solution followed by the densification process. The sol-gel technology has several advantages over other methods of film deposition. Apart from simplicity, the film produced is tough, inert and more resistant than polymer films in aggressive environments. Most of the fiber optic pH sensors developed and reported in the literature are based on evanescent wave absorption spectroscopy. The probe is prepared by immobilizing a pH-sensitive dye on the surface of the unclad core of the fiber using sol-gel technology. To increase the range of the sensor amixture of three suitably selected pH-sensitive dyes were used [6,7].

Many fiber optic components such as couplers, polarizers, modulators, filters, amplifiers, etc. have been developed using side-polished single mode optical fibers [8-11]. In side polished fiber, cladding is partially removed from one side of the fiber. If this side is brought in contact of some medium the mode field of the fiber is modified. This principle is used to sense physical and chemical parameters of a fluid. In the present paper we report the fabrication and characterization of a fiber optic pH sensor based on side polished single mode optical fiber. The sensor is prepared by fixing the fiber in a groove made on one of the surfaces of a fused silica block. To make the surface sensitive to pH it is polished up to the core of the fiber and a film containing a mixture of three pH sensitive dyes is coated on it using sol-gel technology. When light is launched in the fiber the evanescent field present in the film interacts with the dyes in the presence of H^+ ions. The strength of interaction depends on the pH of the fluid and accordingly the transmitted power at the other end of the fiber is changed. The main advantage of the side-polished single mode fiber is that a very small amount of sample is needed to measure the pH of the fluid. This is important when the procurement of the sample in large quantity is not possible.

2. Experiment

2.1. Fabrication of the side-polished fiber half block

To fabricate the side polished single mode fiber sensor, a groove was made on a fused silica/quartz block of size 3.0 cm x 1.5 cm x 1.0 cm using the machine shown in Fig. 1. The fused silica block was mounted on a XYZ movement in order to provide proper alignment and appropriate pressure on the block during the process of groove making. The assembly was fixed below the diamond coated wire which moves to and fro over the block with the help of a DC motor. The block was placed at the center of the wire with the help of XYZ movement. The top surface of the block and the wire were made exactly parallel and then the block was raised to provide appropriate pressure on the block. A uniform pressure should be applied over the entire length of the wire as the radius of curvature of the groove depends on this pressure. Diamond paste and 1-2 drops of glycerin oil that acts as lubricant were used to reduce the frictional heat generated due to the movement of the wire over the surface of the block. When the depth at the center of groove was found to be sufficiently enough to house the fiber (without jacket) comfortably, the deepening of the groove was stopped. The block was then cleaned in an ultrasonic bath with acetone to ensure that no glass particle is left inside the groove. The radius of curvature of the groove is a very important parameter. It is used to estimate the required depth of polishing of the surface of the block. Grooves of different radii of curvature can be made on the surface of the block by applying different pressure over the block. The radius of curvature of the

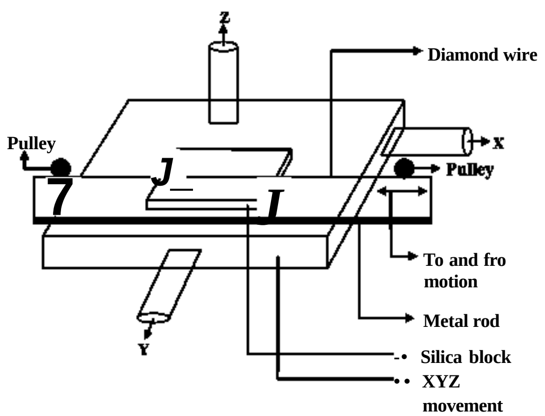

Fig. 1. The schematic diagram for the machine used to make the groove of desired radius of curvature on a silica/quartz block.

groove was measured using a traveling microscope. To measure, the microscope was focused on the top flat surface of the block and then at the points inside the groove at small intervals from one end of the block to the other. This is then fitted to the equation of a circle using the least square fit technique to obtain the radius of curvature. Radius of curvature R of groove can be calculated from the following expression:

$$R^2 = \{LIT)^1 + (R-h + d)\backslash$$

where L is the length of the block, h is the depth of groove at the end of the block, and d is the depth of the groove at the center of the block.

Single mode fiber (SM 600 YORK fiber, wavelength 563 nm, core diameter 4.9 lm and cladding diameter 103.6 lm) of 2 m length was used to fabricate the sensor. The plastic jacket from approximately 2.8 cm length (slightly smaller than the length of block) of fiber around the center was removed. This part of fiber was dipped into a petridish containing acetone for few seconds to remove any trace of the remaining jacket. The groove was filled from one side with an epoxy prepared by mixing one drop of Epotec 330A (Hardner) and seven drops of 330B (Fixer). The capillary action was used to fill the groove to ensure that there are no air bubbles in the groove. The fiber was placed gently inside the groove and the weights were placed on both sides of the block on the fiber. The block was then heated around 80 °C until the color of the epoxy in the groove changed to brown. This generally takes approximately an hour. The block was then allowed to cool. Finally, the polishing of the block was carried out using Logitech machine. It involves the polishing with different abrasives. First polishing was done by a mixture of abrasive (Al_2O_3) of particle size 3 lm with de-ionized water. This process is called lapping. The process of lapping was stopped when 4-5 lm inside cladding portion is reached. To ensure it, the polished region is viewed from the top with the help of a traveling microscope. The presence of an ellipse in the polished region confirms the beginning of polishing. The length of the major axis of the ellipse is measured by means of traveling microscope. This is used to estimate the depth of the polishing already achieved and balance to be done in order to approach the core. The depth of polishing is equal to $l^2 = 2R$, where l is the length of the semi-major axis of the ellipse. Polishing was carried out with 3 lm particle size until 18 lm of remaining cladding was left. Next a second polishing was done by a mixture of abrasive of particle size of 1 lm and deionized water till approximately 8 lm of cladding was left. The final polishing was done by utilizing opaline (cerium oxide) with the particle size of 0.5 lm in the form of a paste in de-ionized water. Using the above procedure two side-polished single mode fiber blocks of radii of curvature 22 and 25 cm were fabricated as shown in Fig. 2.

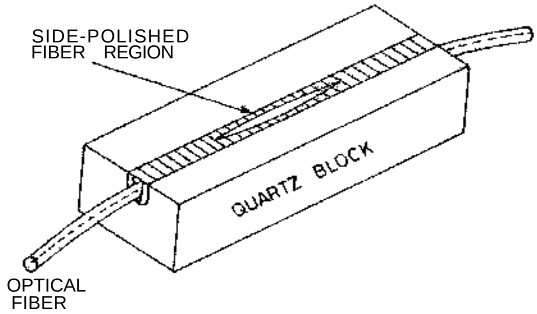

Fig. 2. The schematic diagram for the side-polished single mode optical fiber half block.

2.2. Preparation of film

The method of preparing a thin film of glass on the fiber core using sol-gel technology was similar to our previous study [7]. Tetraethyl orthosilicate (TEOS) was used as the precursor liquid. To prepare the coating solution, 50 ml of TEOS, 50 ml of anhydrous ethanol, 4 ml of de-ionized distilled water, 5 ml of 2-methoxy ethanol and 2 ml of HNO3 along with 46 mg cresol red, 82 mg bromophenol blue and 42 mg chlorophenol red were mixed at room temperature. These dyes were selected because the mixture of these dyes gives a long pH range [6]. After mixing, the solution was stirred for 5 h at 60 °C. Two side-polished single mode fiber blocks of different radii of curvature were dip coated at a pulling speed of 10 cm/min. The coated blocks were then dried at 100 °C for 8h. The thickness of the coating was about 0.15 lm. These

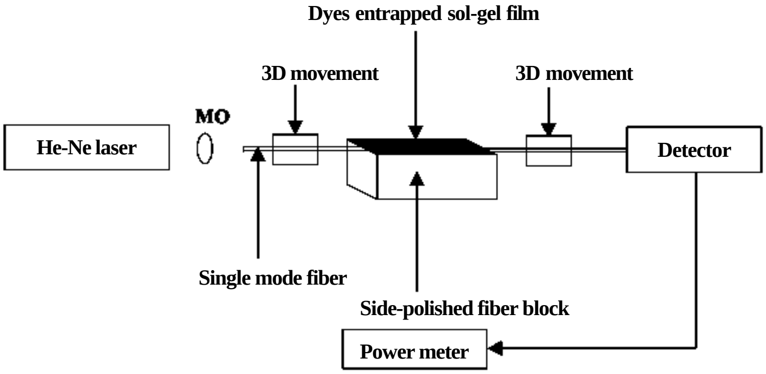

Fig. 3. Experimental set up of the fiber optic pH sensor based on side-polished single mode optical fiber half block.

blocks were then immersed in water to allow the excess and unbound dyes to be removed from the film.

2.3. Experimental set up

The experimental arrangement used to measure the output power from the side-polished single mode fiber half block as a function of the pH of the fluid is shown in Fig. 3. Light from a He-Ne laser operating at wavelength 632.8 nm was launched into one of the ends of the side polished fiber with the help of a 20 x microscope objective of numerical aperture 0.40. It may be noted that in our previous study [4,6,7] we have used tungsten halogen lamp with 600 nm interference filter to launch light in the fiber. This arrangement could not be used in the present study because its power is much less than that required to couple light in a single mode fiber. The disadvantage of using He-Ne laser is that the absorbance of dyes is small at its wavelength. This can be tolerated because the power of the laser is larger than that of the tungsten halogen lamp and interference filter combination. The power coming out from the other end of the side polished single mode fiber was measured by using a silicon detector and a power meter.

3. Results and discussion

To characterize the side polished single mode fiber pH sensor, samples of de-ionized water of different pH ranging from pH 2 to 13 were prepared using concentrated HCl and NaOH solutions. A few drops of one of the water samples were placed at the center of the polished surface of the single mode fiber half block using a syringe and the corresponding output power was measured. Fig. 4 shows the variation of normalized power recorded as a function of pH of the water placed over the surface of the coated half block. The results have been plotted for two half blocks of 22 and 25 cm radii of curvature of the groove. These measurements were first carried out in the ascending order of the pH of the water sample and then in the descending order of the pH in the range 3-13 pH. In both the cases almost same response curve was obtained. The experiments were repeated on each block 3-4 times up to 2 weeks from

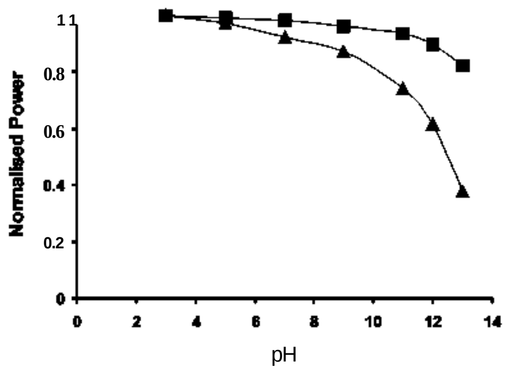

Fig. 4. Calibration curve of the side polished single mode optical fiber pH sensor for half blocks with radii of curvature of the grooves 22 cm (N) and 25 cm (j).

first measurements. Less than 2% deviation in the response curve was observed in these measurements. It may be noted from Fig. 4 that the half block with large radius of curvature of the groove has small variation in output power in the pH range 3-14 while in the case of block with 22 cm radius of curvature of the groove the variation of output power is large and it covers almost full range of the normalized output power (0-1) in the pH range 3-14. Thus the block with 22 cm radius of curvature of the groove is more sensitive than the block with radius of curvature of 25 cm. This is because as the radius of curvature of the groove and hence the bending radius of the side polished single mode fiber decreases the evanescent field in the film increases. This results in the increase in the absorption of light in the film and hence the sensitivity of the side polished single mode fiber block. The advantage of side polished single mode optical fiber pH sensor is that a very small amount of sample (few drops) is needed to measure its pH. This is very important when the procurement of the sample in large quantity is not possible.

In summary, the fabrication and characterization of a side polished single mode optical fiber pH sensor based on evanescent wave absorption and sol-gel immobilization technique have been carried out. To make the probe less fragile the single mode fiber is fixed in a groove fabricated on the surface of the quartz block. The influence of the radius of curvature of the groove on the sensitivity of the pH sensor has been studied. The sensitivity of the side polished single mode optical fiber pH sensor increases with the decrease in the radius of curvature of the groove.

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