TiO$_2$ ALD Nanolayer as Evanescent Waveguide for Biomedical Sensor Applications

A. Purniawan$^a$, P.J. French$^a$, G. Pandraud$^b$, P.M. Sarro$^b$

$^a$Electronic Instrumentation Laboratory (EI)-DIMES, TU Delft, Mekelweg 4, 2628 CD Delft, The Netherlands
$^b$Electronic Components, Technology and Materials (ECTM)-DIMES, TU Delft, Fieldmannweg 17, 2628 CT Delft, The Netherlands

Abstract

In this paper, a low surface roughness and highly homogenous TiO$_2$ layer was deposited by Atomic Layer Deposition (ALD) and used as an evanescent waveguide for biomedical sensing applications. Anastomosis is an operation to continue an organ like colon, bowel, pancreas etc. If there is leakage in during an anastomosis surgery (often indicated by a high bacteria concentration), it probably will cause some problems to the patients. The diagnostic of the leakage must be done soon to avoid multi complications and mortality. The research aims at developing optical sensor to detect the leakage by bacteria concentration detection in the drain fluid. In this present paper, we will report preliminary results of our research such as the simulation, fabrication and optical measurement of TiO$_2$ optical waveguide. Freestanding structure is introduced to enhance sensitivity of such a sensor. Low stress SiN is deposited using LPCVD as interlayer material and etching stop-layer in between the Si substrate and the TiO$_2$ waveguide. To estimate the optimum SiN thickness especially in the freestanding region, simulation was done by effective index method (EIM). Propagation and coupling losses measurement were conducted using the cut-back technique.

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1. Introduction

Many techniques are employed to deposit TiO$_2$ thin film such as sol-gel [1], sputtering [2], spray pyrolysis [3], atomic layer deposition [4-6], etc. Atomic layer deposition has advantages in conformality, homogeneity, and stress because this method deposits layer by layer at atomic scale and at low temperature. TiO$_2$ will be used in this work as evanescent waveguide. In an optical waveguide, the evanescent tales of the electric field propagates outside the waveguide and can then reacts with the surrounding. Therefore evanescent waveguide has high potential as a sensor to detect changing in the vicinity of the waveguides surface by detecting the change of transmitted power. The sensor can be used to detect the changes such as concentration of a fluid, a bacteria concentration or a refractive index by investigating the transmission through the waveguide. The anastomosis leakage can be detected by a 20% bacteria concentration in drain fluid [7]. Nowadays, the patient mortality due to the leakage is quite high, about
18.6% against 2.6% without leakage. The leakage can also generate secondary complications, and mortality in this case is about 62.7% against 19.9% for without leakage [8]. To avoid these problems, the leakage must be detected soon. However the conventional methods such as clinical, physical or manual [7] can be used to detect the leakage but the disadvantage of the methods is that they take a lot of time so it is often too late to take action accordingly to the patient’s condition. In order to solve the problem, in this work, we develop an optical waveguides sensor by assuming that it will reduce the diagnostic time and also we design the structure in an freestanding waveguide structure to guaranty the maximum sensitivity to the sensor (two times larger than slab waveguide). However the limitation to fabricate the structure is one of the challenges in this research.

2. Experimental

2.1. Material and fabrication

Since we aim at an optical device, the criteria of the materials to be used as waveguide must be transparency and low impurities that might causes scattering [9]. In this research, TiO$_2$ is used as waveguide material and deposited by ALD technique. Titanium tetrachloride (TiCl$_4$) and water vapor (H$_2$O) are used as the precursors and N$_2$ as gas carrier. By taking cycle time 7 s/cycle (by ratio per cycle 1:2:1:3 for H$_2$O: N$_2$: TiCl$_4$: N$_2$) and deposition temperature 300°C was found that deposition rate is 0.5Å/cycle. The material has a high refractive index 2.4 and surface roughness of 0.4 nm [10]. A Si wafer is used as substrate. As Si has high refractive index of 3.4 we must put an interlayer in between to avoid the light to leak the substrate. Hence prior the deposition of TiO$_2$, in order to separate the Si wafer and the TiO$_2$ film a thick low stress SiN (1 μm) was deposited by low pressure chemical vapor deposition (LPCVD) as interlayer. However, in the freestanding region, to obtain evanescent tale in both sides, the thickness of SiN must be reduced. The optimum thickness is simulated using Effective Index Method (EIM) [11] and found to be 300 nm for the SiN layer and 250 nm for the TiO$_2$ layer. Fig.1 shows the schematic of device and freestanding TiO$_2$ waveguide.

![Cross Section of TiO2 evanescent waveguide sensor](image)

The fabrication process was conducted in the cleanroom class 100 using photolithographic fabrication methods. The process used three masks consisting of one mask to make windows in the backside and two masks to pattern the rib and the ridge on the front side. Etching processes use Alcatel RIE plasma etching with CF$_4$ (70 sccm), SF$_6$ (10 sccm) and O$_2$ (10 sccm) as gases to etch SiN using a power of 60 watt (etching rate 78 nm/minute). During plasma etching, ions bombard the surface then cause high surface roughness. Consequently the power has to be reduced to eliminate the effect of the bombardment. By doing so the etching rate is also lowered. In our experiments we found that by using power 20 watt the etching rate decrease to 23 nm/minute and, interestingly, the roughness increased.
from 0.4 nm to 4 nm. In addition to etch TiO$_2$ we used CH$_4$ (50 sccm), CHF$_3$ (25 sccm) and H$_2$ (40 sccm). We found that etching rate of 60 watt and 20 watt are 10 nm/minute and 2.3 nm/minute, respectively.

Silicon back side etching using KOH within 6 hours was carried out to make membrane until stop layer. Besides as an interlayer between Si and TiO$_2$, SiN is also used as a stopping layer during backside etching through Si wafer. Meanwhile in this etching, vacuum holder protection was employed to protect front side wafer. Finally, reactive ion etching (RIE) Alcatel is used to remove SiN membrane so that become freestanding structure. Fig. 2 shows the cross section profile and top view of freestanding waveguide. The results show that thickness of rib is 51 nm and the ridge is 490 nm. These are quite close with expected results.

![Fig. 2. Profile of TiO$_2$ freestanding waveguide (a) cross section profile investigated using DEKTAK profilometer and (b) top view SEM image of waveguide length and width of freestanding structure of 354 $\mu$m and 49 $\mu$m, respectively.](image)

2.2. Optical measurement

To measure the propagation and coupling losses of the fabricated TiO$_2$ waveguides we used the optical measurement system schematically shown is Fig. 2. The system consists of a source of light (LED), photo diode, oscilloscope and optical fiber. Light with wavelength 1.3 $\mu$m is used. Light propagates in an optical fiber to the waveguide and then through the waveguide. The light will be converted to voltage or current flow by photo diode at the end of the waveguide. Then from the photo diode the intensity will be display in oscilloscope.

![Fig. 3. Schematic of the optical measurement system](image)

3. Results and Discussion
The optimum thickness was estimated by Effective Index Methods (EIM) [11]. Based on our simulation, for 1 micron SiN, we did not find evanescent tale in backside of waveguide. By reducing the thickness of SiN we found that by using 300 nm of SiN, we have evanescent tales on both front side and backside (Fig. 3).

Propagation and coupling losses were investigated using optical measurement. In this measurement we used a LED as light source and a photodiode to convert intensity to voltage. Losses can be obtained by recording the output signal and using formula as follow [12]:

$$dB = 10 \log \frac{P_{in}}{P_{out}}$$

By using “cut back method” which divides the waveguide into several different lengths, then the losses are plotted in the figure. Fig. 4 shows results of waveguide losses. By using linear regression, the propagation loss and coupling losses can be estimated. Propagation loss is the slope of the line and coupling losses are losses when the length of waveguide in zero length.

Based on the experiments were found that the propagation loss of the waveguide is 6.7dB/cm and a coupling loss of 19 dB (Fig. 5). The high propagation loss of the waveguides make them not suitable for telecommunication but it can be applied for sensing because in this application only a readable output signal is required.
4. Conclusion

TiO2 ALD film has acceptable losses and can be considered as waveguide materials to be applied as sensor. Freestanding waveguide structure has been successfully fabricated by combining high power and low power RIE. To enhance the strength of the freestanding structure, SiN is used as support layer. However the thick SiN will affect on the evanescent tale (i.e the sensitivity) and consequently must be reduced. A bi layer of 250 nm TiO2 on 300 nm SiN is an optimum to have an evanescent tale in both sides. In this configuration the expected sensitivity of the device is two times slab waveguide.

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