

Distributed Feedback Laser Biosensor Incorporating a Titanium Dioxide Nanorod Surface

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Abstract: A 6.6× enhancement in surface sensitivity of DFB laser biosensors is demonstrated by a device incorporating a porous titanium dioxide nanorod layer on the sensor surface, while a Q-factor of over 25, 000 is maintained.

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Recently, optical biosensors based upon DFB lasers fabricated using plastic materials over large surface areas have been demonstrated to operate with single mode and obtain high Q-factor through the process of stimulated emission [1, 2]. Therefore, DFB laser biosensors (DFBLB) are simultaneously capable of high sensitivity and a high degree of resolution. Previously, we have demonstrated DFBLB sensors that incorporate a 40-80 nm solid TiO₂ layer as the uppermost surface to bias the resonant mode to reside more fully within the liquid media, thus providing a strong dependency of the emission wavelength on the density of adsorbed biomolecules [2]. Here, we demonstrate that a porous TiO₂ nanorod layer may be used in place of the solid film of TiO₂ to enable an ever greater 3-dimensional volume overlap between the lasing mode and biomolecular adsorption. This modification in device design results in a ~6.6x increase in detection sensitivity, while maintaining a narrow spectral output that corresponds to a cavity quality factor Q = 25, 600.

As shown in Fig.1, The DFBLB structure is comprised of a UV-curable polymer grating (RI ~ 1.39) that is overcoated with a laser dye (Rhodamine 590, Exciton) doped SU-8 layer (5.0 wt %; Microchem, RI=1.58, t=300nm), which provides optical gain, vertical light confinement and horizontal feedback. The nanorod structure was deposited on top of the gain layer by e-beam evaporation using the Glancing Angle Deposition (GLAD) method in which the incident material flux is provided at an oblique angle to the device surface[3, 4]. All GLAD depositions were performed at a base pressure of 3.0×10⁻⁶ Torr and deposition rate of 7.2 Å/s. A scanning electron microscope image of a GLAD TiO₂ film is shown in Fig. 3.

As shown in Fig.2, the DFBLB was optically pumped with 10 ns Q-switched Nd:YAG laser pulses at λ=532 nm. The pump light first goes through a spatial filter, and then is expanded and focused onto the sensor through a 20x microscope objective (f = 9 mm) placed beneath the sensor. The laser emission is collected with an optical fiber (diameter = 0.5 mm) and delivered to a CCD-based spectrometer with 0.0125 nm resolution (Horiba Jobin Yvon iHR550). The measured spectrum is fitted with a Lorentzian distribution model using MATLAB to precisely determine the peak wavelength value (PWV) of laser emission. As shown in Fig.4, the observed lasing wavelength was at λ= 602.4513 nm with sensor surface coated with 90nm TiO₂ nanorod film and immersed in water. The emission linewidth (full-width half-maximum) Δλ = 0.0235 nm was obtained, which corresponds to a Q factor of 25,600. Inset shows the red shifts of PWV due to increased TiO₂ nanorod thicknesses.

The DFBLB sensor sheet is attached to a bottomless 96-well microplate that is held in a fixture that is attached to an x-y motion stage, which enables computer-programmed excitation of a precise location on the DFB surface. Within a single microplate well, the excitation laser separately probes 75 independent locations in an x-y rectangular grid with 400 μm spacing. The 75 measurements are averaged to compute the PWV of a single well.

To demonstrate the sensitivity enhancement, sensitivity to surface mass adsorption was characterized. Sensors with either a 30 nm solid TiO₂ film or nanorod TiO₂ films of 22nm, 30 nm, 67 nm, 90 nm were prepared. All the sensors were coated with a single monolayer of protein using the following protocol. First, the sensors were exposed to a phosphate buffered saline (PBS) solution for 15 min to take a baseline PWV measurement. Next, the PBS solution was replaced with 1 mg/ml solution of the protein polymer poly (Lys, Phe) (PPL) (Sigma-Aldrich), which has been demonstrated to form a self-limiting single monolayer coating whose thickness is ~15 nm and refractive index n~ 1.45 upon dielectric surfaces [2]. The PPL solution was incubated with the sensor surfaces for 48 minutes, after which the PPL solution was removed and the sensor surface was rinsed with PBS solution 3 times to remove any PPL that was not firmly attached to the sensor surface. Then, the stable PWV shifts were measured. Our

dynamic measurement results (Fig.5) show that thicker TiO₂ nanorod films result in the greatest PWV shift for adsorption of PPL, and the thickest nanorod layer (90nm) results in a 6.6× enhancement in surface sensitivity.

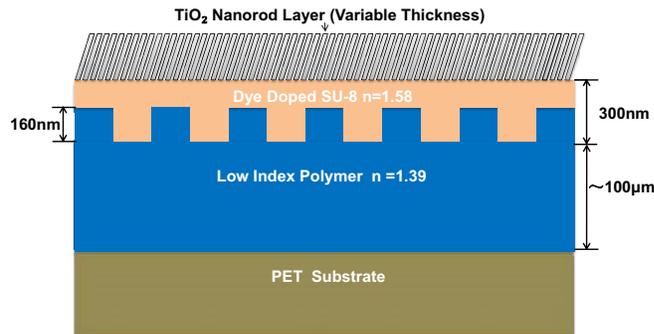


Fig.1. Cross-sectional diagram of the DFBLB structure (not to scale). The period and depth of the grating structure is 550 nm and 160 nm, respectively. The sensor surface was coated with TiO₂ nanorod films with different thickness of 22 nm, 30 nm, 67 nm, or 90 nm.

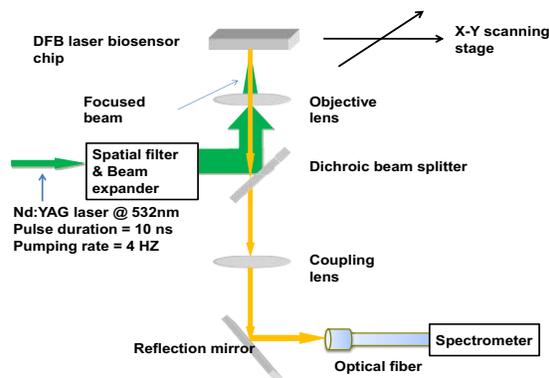


Fig.2 Schematic of the Biosensor detection instrument setup

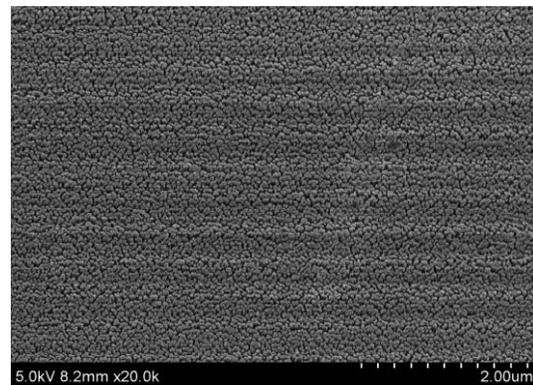


Fig.3 SEM image of the nanorod-coated sensor

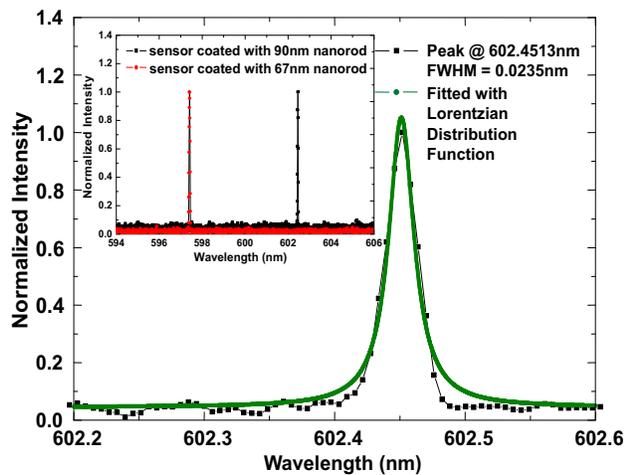


Fig.4. DFB laser emission profile. The sensor was coated with 90nm TiO₂ nanorod film and immersed in DI water. Inset shows the red shifts of PWV due to increased nanorod TiO₂ thicknesses.

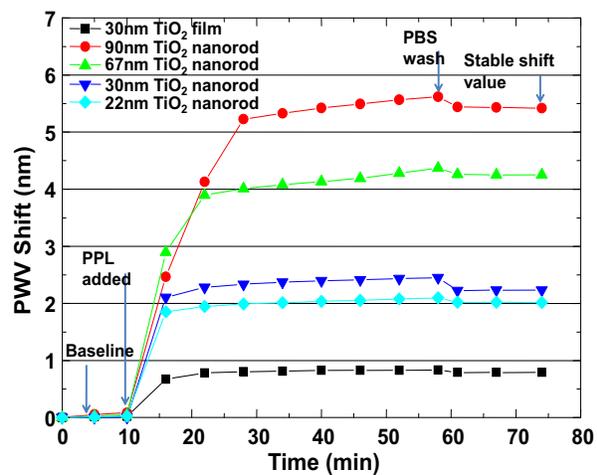


Fig.5. Dynamic detection of adsorption of a polymer protein self-limiting monolayer for nonporous TiO₂ film coated and porous TiO₂ nanorod coated sensors.

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