

# Optical biochemical sensors based on long-period fibre gratings UV-inscribed in D-fibre with enhanced sensitivity by HF etching process

Xianfeng Chen, Kaiming Zhou, Lin Zhang, Ian Bennion  
Photonics Research Group, Aston University, Birmingham, B4 7ET, UK

## ABSTRACT

We report a new concept of biochemical sensor device based on long-period grating structures UV-inscribed in D-fibre. The surrounding-medium refractive index sensitivity of the devices has been enhanced significantly by a hydrofluoric acid etching process. The devices have been used to measure the sugar concentrations showing clearly an encoding relation between the chemical concentration and the grating spectral response, demonstrating their capability for potential biochemical sensing applications.

## 1. INTRODUCTION

Fibre Bragg gratings (FBGs) and long-period gratings (LPGs) have been studied extensively [1,2] and used for many applications such as gain-flattening and band-rejection filters [3] and optical sensors for monitoring strain, bend, temperature and pressure [4-7]. However, LPGs offer an additional capability of surrounding-medium refractive index (SRI) sensing that has been utilised to implement chemical and biochemical sensors [8,9]. The SRI sensitivity of LPGs originates from the mode coupling taking place between the core and cladding modes, in contrast to that occurs only between the core modes in an FBG structure. It has also been reported that the SRI sensitivity of LPGs can be enhanced by chemical etching [10].

In this paper, for the first time to our knowledge, we present a new type of optical biochemical sensor based on LPGs UV-inscribed in D-fibre. The sensors exhibit much higher SRI sensitivity than that made in standard circular fibre. More importantly, we demonstrated that the SRI sensitivity of D-fibre LPGs can be further enhanced by a hydrofluoric acid (HF) etching process and the devices have been used to measure the concentrations of sugar solution.

## 2. FABRICATION AND SPECTRAL RESPONSES OF LPGS

The fibres used in our work were single mode standard fibre (SMF) and D-fibre. Fig. 1 shows the cross-section profile of the D-fibre. The radius of the circular cladding is  $\sim 62.5 \mu\text{m}$ , the distance between the core and the flat surface of the cladding is  $\sim 13.0 \mu\text{m}$ . The core is elliptical with a short axis of  $2.5 \mu\text{m}$  and a long axis of  $4.5 \mu\text{m}$  and surrounded by a Fluorine-doped thin elliptical inner layer cladding.

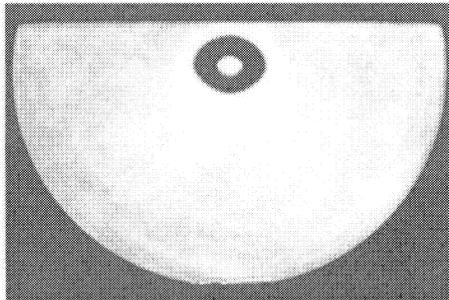


Fig. 1 The cross-section image of the D-fibre used in investigation.

In order to enhance the photosensitivity, the SMF and D-fibre samples were first loaded with hydrogen at a pressure of 180 bar for 48hrs at  $80^\circ\text{C}$ . The LPG structures with periods of  $488 \mu\text{m}$  and  $381 \mu\text{m}$  were then written into the hydrogenated

SMF and D-fibre samples using a 244nm UV laser and the point-by-point method. After the exposure, the LPGs were annealed at 80 °C for 24hrs to stabilise their optical properties. The spectral responses of the LPGs were measured using a system incorporating a broadband light source, a polariser and a polarisation controller, and an optical spectrum analyser. The typical transmission spectra of LPGs in SMF and D-fibre are illustrated in Fig. 2a & b, respectively, exhibiting several broad attenuation resonances in the wavelength range from 1300nm to 1700nm.

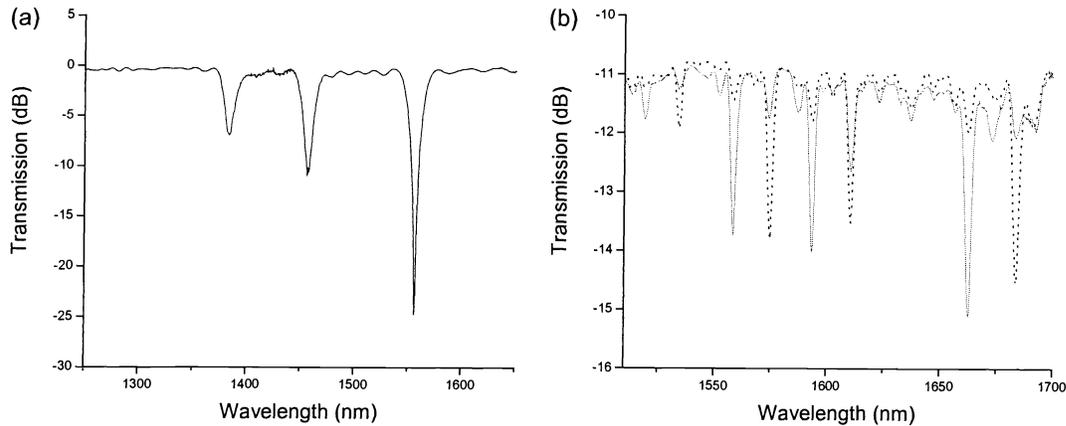


Fig. 2 Typical transmission spectra of LPGs in (a) SMF and (b) D-fibre. Note: there are two sets of modes (represented by solid and dotted lines respectively) corresponding to two polarisation states.

The transmission loss peaks of LPGs were generated by the light coupling from the core mode to the cladding modes in the forward propagating direction at the phase match wavelengths. In the case of the D-fibre LPG, two sets of loss peaks can be observed as shown in Fig. 2b, corresponding to the two polarisation states resulted from the birefringence effect from the D-shaped cladding and the elliptical core.

### 3. HF ETCHING EXPERIMENT

In order to effectively control the LPG etching process, we first investigated the etching rate using 10% HF dilute solution using non-grating-containing fibres. Twenty samples of each SMF and D-fibre were immersed in the HF solution and were taken out in turn in every 10min. The samples with different etched claddings were then examined and measured using a microscope with high magnification. Fig. 3a displays the cross-section profile of a D-fibre sample etched for 40min and Fig. 3b plots the cladding radii of the SMF and D-fibre and the distance between the core and the flat surface of D-fibre against etching time, showing an etching rate of ~0.068µm/min. The cladding on the flat side of the D-fibre was almost etched off after ~173min, leaving the core exposing to the air on this side.

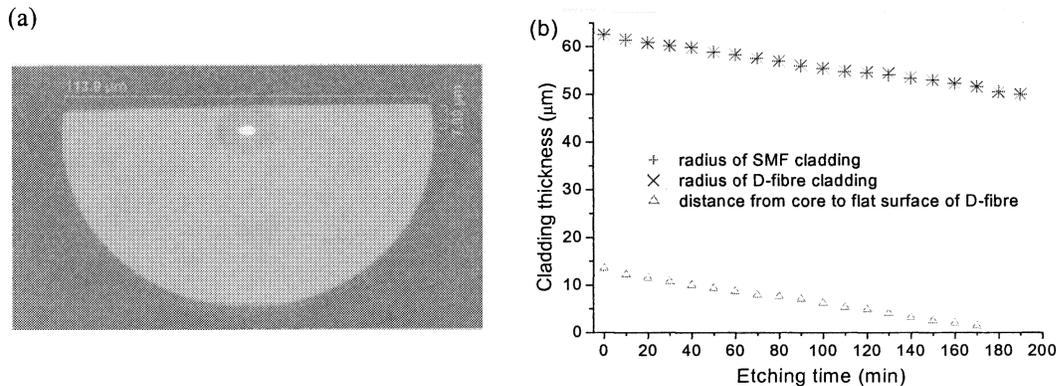


Fig. 3 (a) The cross-section profile of a D-fibre etched for 40min. (b) The cladding radii of SMF and D-fibre and the distance between core and flat surface of D-fibre against etching time.

The spectral evolution of D-fibre LPG under HF-etching was then investigated. Fig. 4 depicts the spectral evolution of the D-fibre LPG during the etching process, clearly showing that the four coupled cladding modes were red-shifting with decreasing cladding thickness.

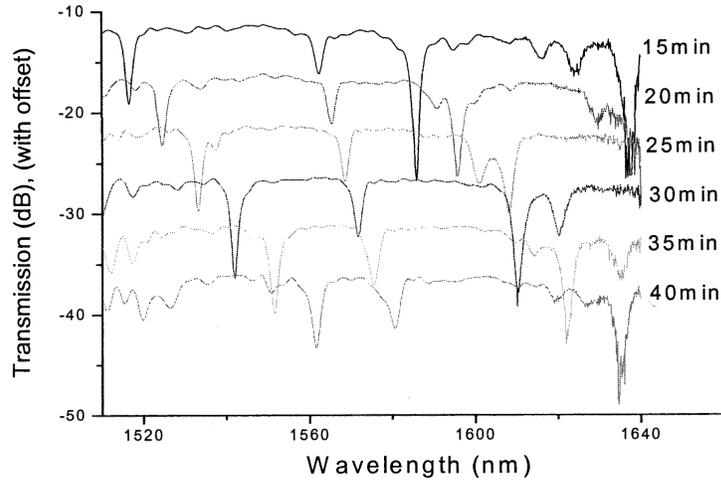


Fig. 4 The spectral evolution of an LPG in D-fibre against the etching time.

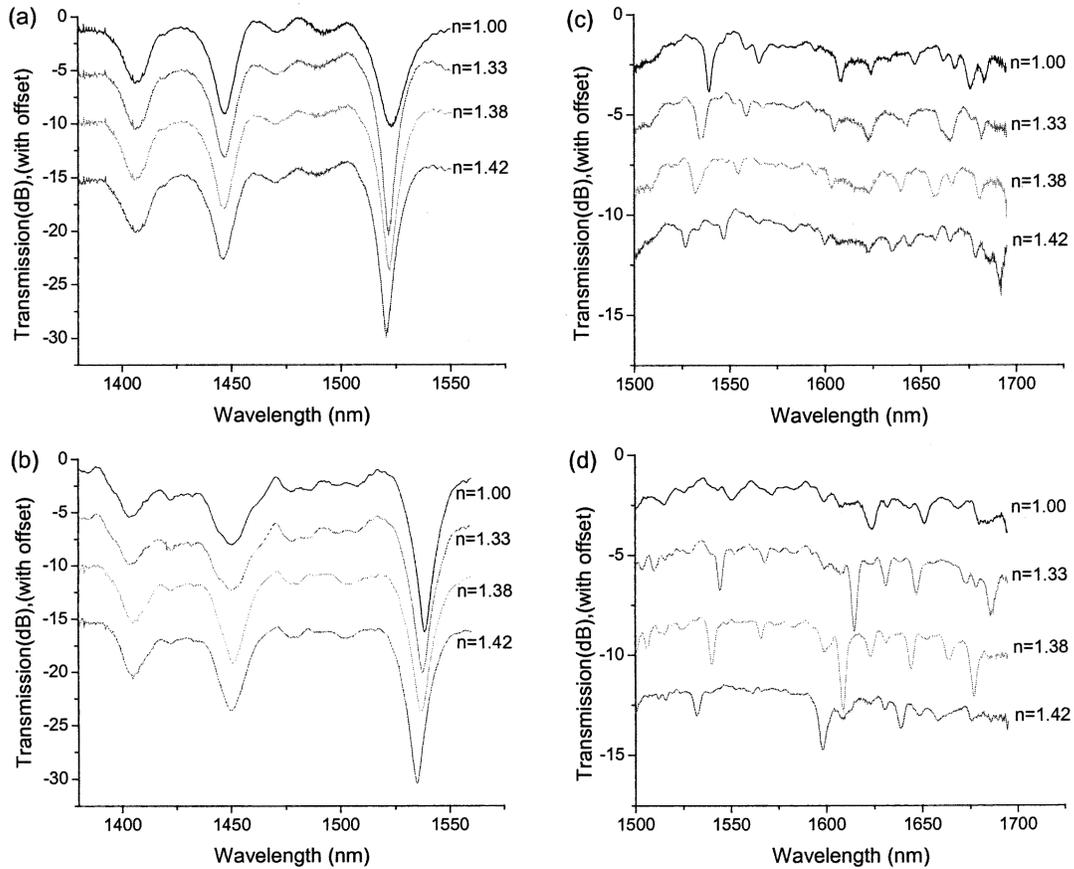


Fig. 5 Spectral evolutions of LPGs in SMF (a, b) and D-fibre (c, d) before etching (a, c) and after being etched for 40min (b, d).

#### 4. LPGS AS SENSORS FOR SUGAR CONCENTRATION MEASUREMENT

Two LPGs UV-inscribed in SMF and D-shape fibre were used to measure the concentrations of sugar solution before and after they were subjected to the etching treatment. The LPG was placed in a V-grooved metal plate filled in with the sugar solution and its spectral response was measured using the system described in section 2. After each measurement, the grating and the plate were carefully cleaned before applying next solution. The sugar solution with concentrations ranging from 0% to 60% were measured using both SMF and D-fibre LPGs. In the D-fibre case, because the light can couple from the core mode to cladding modes with either polarisation state, the polariser and polarisation controller were used to maintain one polarisation state throughout the measurement. It was also ensured that there was no measurable changes caused by the fibre positioning and thermal fluctuation in the experiment.

Fig. 5 shows the spectral evolutions induced by sugar concentration change for the LPGs in SMF ( a: without etching; b: etched for 40min) and in D-fibre (c: without etching; d: etched for 40min), respectively. It is clear that the transmission loss bands of the LPGs are blue-shifting with increasing sugar concentration and the shift-rate greatly depends on the fibre type and the etching state.

Because the sensing mechanism of the LPGs is based on the grating response to the change of SRI, we have calibrated the correlation between the refractive index and the concentration of sugar solution using the data from reference [11].

Table 1. Refractive Index of Sugar Solution ( $C_{12}H_{22}O_{11}$ )

Mass% of sugar solution	10	20	30	40	50	60	70	80
Refraction Index	1.348	1.364	1.381	1.400	1.420	1.442	1.465	1.491

The resonance shifts against the refractive index of the sugar solution for three cladding modes of the LPG in SMF and two in D-fibre before and after the etching treatment are plotted in Fig.5. Two trends can be clearly recognised from the figure: (1) the three modes of the LPG in SMF exhibit much smaller SRI sensitivities than the two modes in D-fibre before and after the etching; (2) the etching process has significantly sensitised the SRI response of the D-fibre LPGs. Making a quantitative comparison between Fig. 5a and 5b, the SRI sensitivity ( $\Delta\lambda/\Delta n$ ) of the high order mode of the LPG in D-fibre has been increased from 30nm/0.11 to 46nm/0.11 after 40min etching.

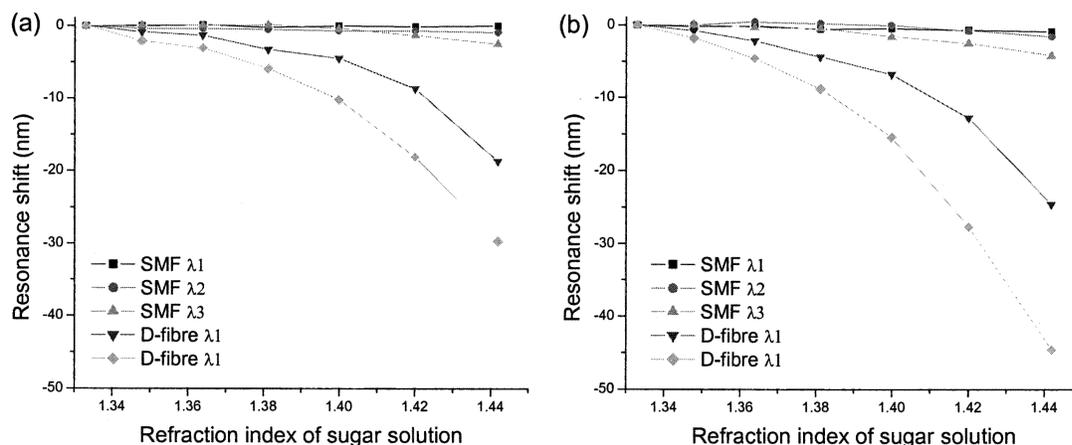


Fig. 6 Wavelength shifts against refractive index of the sugar solution for three cladding modes in SMF and two modes in D-fibre before etching (a) and after being etched for 40min (b).

#### 5. CONCLUSIONS

The detailed and comparative investigation has been conducted to implement SRI sensing employing the LPGs UV-inscribed in SMF and D-fibre and sensitised by HF etching process. The results from the study clearly reveal that the

SRI sensitivity of LPGs in D-fibre is intrinsically high and can be further enhanced significantly by the HF etching process. We have used these devices to measure sugar concentrations, demonstrating their suitability to be used as sensors for potential biochemical, medical and environmental sensing applications.

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