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Using double resonance long period gratings to measure refractive index of milk of varying fat content

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We have studied the possibility to measure the refractive index of milk using double resonance (DR) long period fiber grating (LPG) characterized by a higher refractometric sensitivity around water refractive index values. DR LPGs are characterized by a turning point beyond which the resonance minimum splits into two minima which shift in opposite directions for surrounding refractive index (SRI) changes to which they can be highly sensitive. Due to this DR LPGs are a promising platform for bacteria sensing that allows the implementation of lab-on-the-fiber concept. Similar application for milk analysis requires the measurement of milk refractive index for different fat content levels. We have applied direct center wavelength (CW) shift method and the differential signal method to track RI changes and compared the values obtained with a standard Abbe refractometer. CW shift as high as 5 nm were obtained for a fat content varying from 0.1% to 3.5%.

Key words: double resonance (DR) long period fiber grating (LPG), refractive index, sensors

INTRODUCTION

Fiber optic (FO) sensor technology has advanced dramatically in the area of biochemical sensing [1]. Among the variety of sensing transducer types are long period gratings (LPG) [2] because, similarly to core-cladding intermodal sensors [3, 4], they exhibit a very high sensitivity to surrounding refractive index (SRI) changes. Sensing the presence of bacteria such as *Escherichia Coli* and Staphilococcus then naturally becomes feasible and desirable with suitable FO sensors. In particular, the importance of sensing *E. Coli* bacteria in milk has been of interest worldwide for decades [5–7]. So far the method mostly employed for FO E-Coli sensors is based on fluorescence [8, 9]. However, the recently demonstrated sensitivity of about 2322 nm/r.i.u. with double resonance (DR) LPGs has enabled their successful application to bacteria in water [10] and it has been possible to measure bacteria concentration rather than just the detection of *E. Coli* presence [10]. With the far higher theoretically possible sensitivity to SRI, DR LPGs become a promising transducer for bacteria sensing in milk and dairy products. The principle for bacteria sensing is immobilization of phages on the surface of a double resonance LPG and when immersed in water infected with *E. Coli*, the latter are selectively captured by the phage. The accumulation of bacteria on the LPG surface changes the refractive index which in turn causes center wavelength down shifts of the LPG. The wavelength shifts are measured by an OSA and a spectrometer, and are a measure of the bacteria concentration. To achieve maximum sensitivity of the LPG it is made in a way that its turning point is around the refractive index of water in which the bacteria is to be detected. This is done by deliberate controlled etching of the LPG. However, if *E. Coli* is to be measured in milk, then it matters what the refractive index of milk is and how it varies with fat content.

The purpose of the present work is to measure refractive index changes of milk with varying fat content using the same type of DR LPGs as the ones used to sense the concentration of *E. Coli* [10].

EXPERIMENTAL SET-UP

The experimental set-up to take the measurements consists of a 16-channel LPG interrogation unit which uses an InGaAs CCD unit [11]. The two LPGs (LPG1 and LPG2) are connected to port 1 and 10 respectively, while Port 16 is used for taking a reference measurement with respect to the spectrum of the internal C+L (1522-1622 nm) band ASE broadband source.

The LPG spectrum is observed in a reflection mode using a fiber mirror deposited at one end of a tunable air-gap attenuator. In this way if the signal from the source is too strong it can be attenuated to

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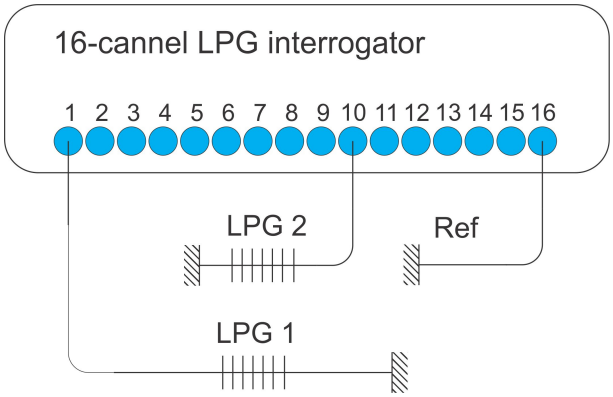

Fig. 1. Experimental set-up.

a desirable level. Before taking measurements, first a reference signal was taken through channel 16 using a fiber jumper. Next spectra of the double resonance LPGs were taken in air with an RI = 1. It is characteristic of this type of gratings to be observable after immersed in water or in a liquid with a higher RI.

The first thing we must do is to calibrate the LPG. To calibrate the LPG we measure its spectrum response to different surrounding refracting index (SRI) as it is shown in Fig. 2. In the spectral range only the left side of the double resonance is seen. The right side is beyond 1622 nm and is out of the range of the interrogator. The calibration is made using glycerin-water solutions with different concentration and correspondingly different refracting indices. What we can see in Fig. 2 is that the spectrum response of the LPG is shifting to smaller wavelengths when SRI increases. Then the spectral responses with increasing values of the RI were taken for each grating. To measure the spectral shifts we take the wavelengths at a given loss level for example -15 dB on either side of the minimum.

Having the spectral responses of the LPGs over a range of SRIs, we can determine the wavelength shifts at the level of -15 dB for the left and the right sides of the minimum and the average. As we can see in Fig. 3 when SRI is increasing the negative spectral shifts increase as well. The changes of the spectral shifts vs. SRI are linear for the both sides and their average (Fig. 3). It is evident that the spectral shifts are not symmetrical and the right side shifts more than the left side. This changes the shape and width of the DR LPG. For LPG1 the sensitivity to SRI of the right side is $S_n = -1530.7$ nm/r.i.u. and of the left side $S_n = -698.87$ nm/r.i.u. The average sensitivity is $S_{\text{avg}} = -1114.8$ nm/r.i.u. Since the LPG has two resonances, the minimum-to-minimum shifts is double i.e. -2229.6 nm/r.i.u. However, if the internal slopes' shifts of the double resonances are measured the sensitivity would be -3061.4 nm/r.i.u. With a resolution of 0.1 nm that is easily attainable by the LPG multichannel interrogator a sensitivity of around 3.3×10^{-5} r.i.u can be obtained for double resonance tracking or 6.6×10^{-5} r.i.u for single resonance tracking.

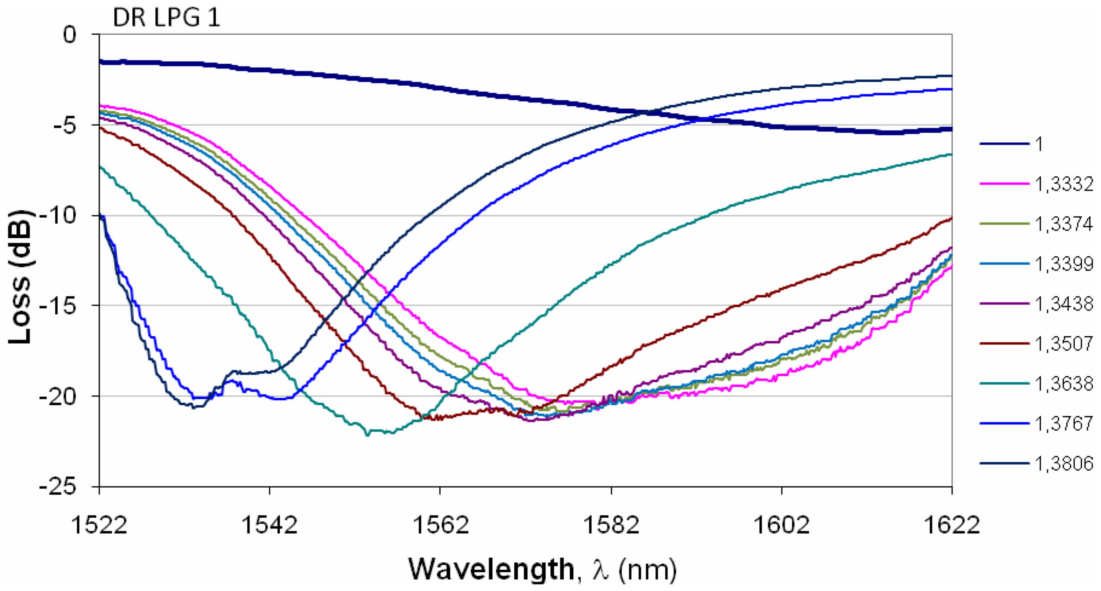

Fig. 2. Spectral response of DR LPG 1 over different SRI.

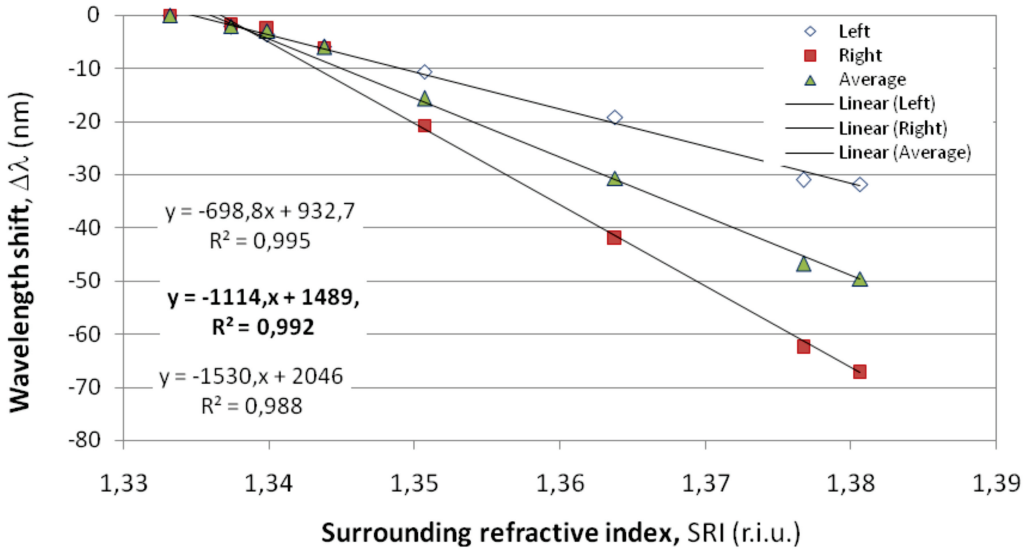

Fig. 3. Wavelength changes at -15 dB for the left and right sides of the minimum and their average for DR LPG 1.

Having determined the sensitivities of the LPG, we measure the spectral changes caused by water and four different fat contents namely 0.1%, 1%, 2% and 3.25%). All measurements were taken at room temperature.

We perform the same spectral measurements for milk with a different fat concentration levels. When the concentration of fat in milk increases the refractive index of the milk increases and the higher concentration, the higher refractive index. That can be seen in Fig. 3. Thus, when the concentration of fat in milk increases, the minimum of the spectral response shifts to smaller wavelengths. Also, the widths of the grating's spectrum become narrower, which means that the right side of the spectrum shifts at a higher rate.

In this case again we measure the wavelength shifts to the right and to the left of the minimum. Then we determine the corresponding refractive indices by placing the points on the Wavelength vs. SRI plots from Fig. 2. This is shown in Fig. 5. for the left and right side shifts. For greater accuracy the curve fitting is polynomial.

Fig. 6 shows the final dependence of the milk refractive index vs. the percentage of the fat contents for both LPGs.

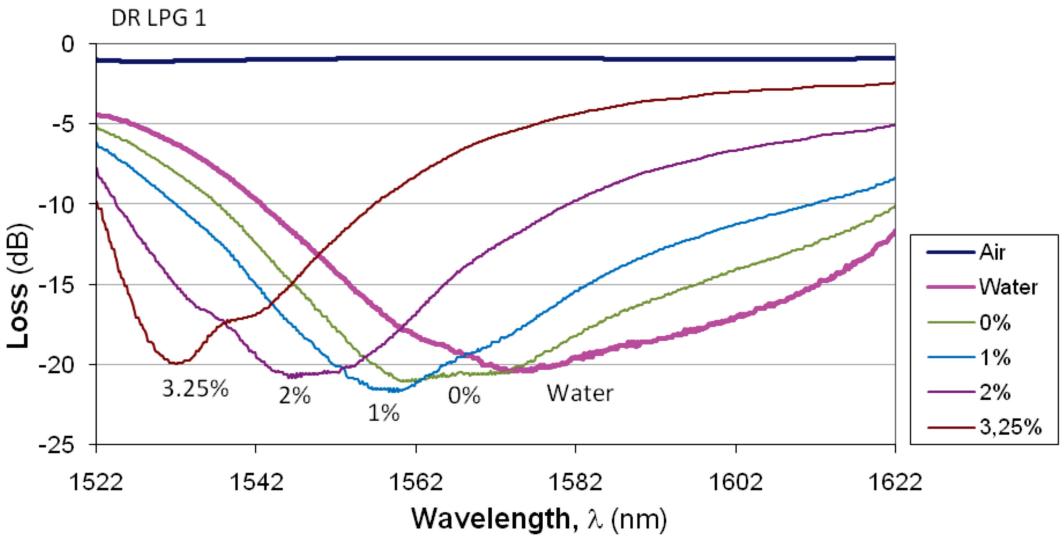

Fig. 4. Spectrum response of DR LPG 1 to milk with different fat concentrations.

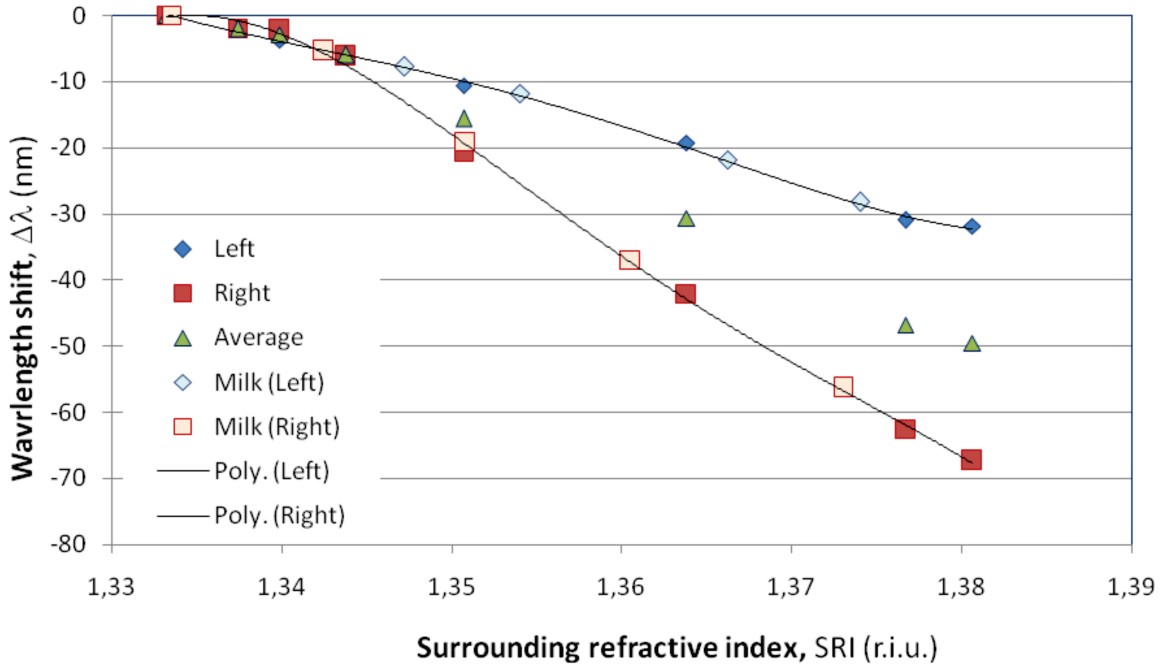

Fig. 5. Wavelength changes at -15 dB for the left and right sides of the minimum and their average for DR LPG 1 dipped in milk with different fat concentration.

For both gratings the dependence of the milk refractive index on the percentage of fat is linear and both gratings provide practically the same results.

These results are particularly important in view of the fact that milk is a turbid media and LPGs can be multiplexed spectrally and spatially as in this case which permits to track changes during different technological processes.

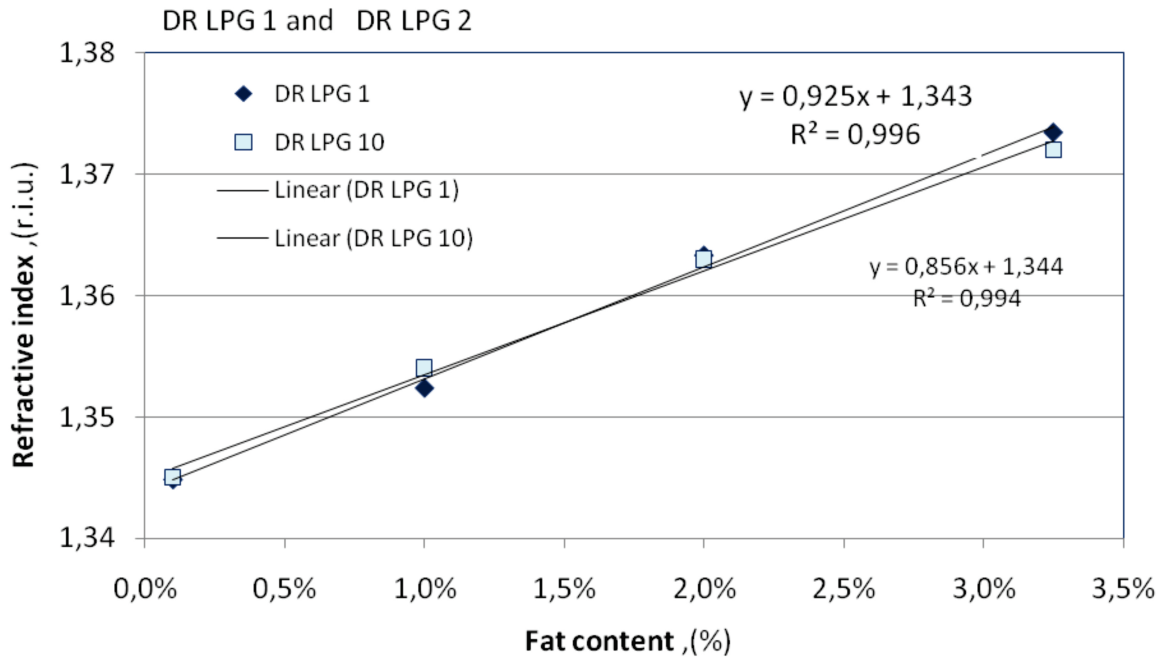

Fig. 6. Dependence of milk refractive index vs. fat content.

CONCLUSION

Our experiments allow the formulation of the following conclusions:

1. The refractive index of raw cow milk can successfully be measured making use of high SRI sensitivity double resonance LPGs
2. The increase of the fat content of raw milk causes a linear increase of the refractive index
3. To measure bacteria concentration in raw milk the DR LPGs must be manufactured with a maximum sensitivity around different refractive indices depending on the fat content.

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ИЗПОЛЗВАНЕ НА ДЪЛГО-ПЕРИОДИЧНА РЕШЕТКА С ДВОЕН РЕЗОНАНС ЗА ИЗМЕРВАНЕ ПОКАЗАТЕЛЯ НА ПРЕЧУПВАНЕ НА МЛЕКА С РАЗЛИЧНО СЪДЪРЖАНИЕ НА МАЗНИНИ

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(Резюме)

В настоящата статия сме проучили възможността за измерване на коефициента на пречупване (RI) на мляко, като за целта сме използвали дълго-периодична решетка (LPG) с двоен резонанс (DR), която се характеризира с по-висока рефрактометрична чувствителност около коефициента на пречупване на вода. Дълго-периодичните решетки с двоен резонанс се характеризират с това, че при определена стойност на показателя на пречупване на околната среда резонансният минимум се разделя на два минимума, които се отместват в противоположни посоки с повишаване на показателя на пречупване, към който те могат да бъдат много чувствителни. Поради тази причина DR LPGs са подходящи за сензори за детектиране на бактерии. Подобни приложения за анализ на мляко изискват измерване на показателя на пречупване на млека с различно съдържание на мазнини. Използвали сме метода за директно следене на дължината на вълната при изместване на централния максимум (CW) и метода за диференциално следене на сигнал за проследяване на промени на RI. Получените резултати сме сравнили със стойностите, получени със стандартен *Abbe* рефрактометър. Установихме, че при промяна на съдържанието на мазнини от 0.1% до 3.5%, централният максимум се измества с около 5 nm.