An imprinted polymer-based guided mode resonance grating sensor

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ABSTRACT

In this paper, different guided mode resonance (GMR) grating sensors are studied with the focus on investigating the possibility of high-performance configurations with simplified and potentially low-cost fabrication methods. The gratings are fabricated in polymer using nano-imprint lithography (NIL). We have chosen Ormocer® materials, as they allow fabrication at room temperature, are UV patternable and have good optical and dielectric properties. The GMR gratings have a pitch around 550 nm, which corresponds to a resonance response around 850 nm with a Q factor of 2200 in simulations. In experiment, gratings imprinted on glass and on foil are characterized, showing a successful imprinting process of both devices. The measured optical response of the GMR grating for a change of the refractive index unit (RIU) of the cover ($\Delta\lambda/\Delta n_c$) is 100 nm/RIU. The measured temperature sensitivity is -0.07 nm/K.

Keywords: Guided mode resonance grating sensor, nano-imprint lithography, polymer materials, flexible sensor foil

1. INTRODUCTION

Optical sensors based on guided mode resonance realized in polymers are promising candidates for sensitive and cost-effective sensors. Optical sensors, in general, are increasingly being used in several applications for their immunity to electromagnetic interference and possible high sensitivity. Various sensors based on different resonance structures such as Fabry-Perot etalons, Bragg gratings and micro-ring resonators have already been demonstrated in literature. In [1], a multi-axial strain Bragg grating sensor is described. These sensors are realized in very thin foils to make them suitable for embedding in composite materials. Another example is the study of polymer microring resonators for biosensing [2] or ultrasonic detection [3]. Most compact optical sensors based on resonances employ waveguides. Therefore, precise alignment of a fiber is required for in- and out-coupling to the light, which is impractical for many applications. GMR gratings, on the contrary, allow easy read out as they are simply illuminated with a collimated beam from a distance and then a resonance in the transmission or reflection spectrum can be recorded with a detector.

GMR sensors have already been studied as biosensors [4], pressure sensors [5] and narrow-band filters [6]. In this paper, we present an approach for refractive index (RI), strain and temperature sensing which utilizes the high sensitivity of GMR gratings in conjunction with the advantages of polymer materials, which allow elegant and cost-effective fabrication.

1.1 GMR biosensors

GMR gratings can be used as label-free biosensors. Optical biosensors have undisputed advantages compared to standard lab-scale techniques as they are non-invasive, non-destructive and allow simple, rapid and continuous in situ detection for clinical diagnostics, biomedical research and pathogen or toxic detection. The cover of the designed GMR grating is air. By depositing a liquid on top of the grating the cover RI increases, resulting in a shift of the resonance. Measuring this shift gives information about the liquid to be investigated. This can either be bulk sensing where the RI variation occurs homogeneously above the sensor surface or surface sensing where a thin layer of bioreceptors is immobilized on the sensor surface and interacts selectively with the corresponding analyte, which results in a RI change as well [7].

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1.2 GMR strain sensors

The grating structures can be realized in polymer materials on a very thin foil, which can be integrated in thin membranes [1]. By applying a strain to the sensor, the GMR grating is stretched out, leading to an increase in the pitch of the sensor and therefore a resonance shift. The application of a GMR grating as a strain sensor has not been intensively studied yet as most GMR gratings are fabricated on rigid substrates so far.

1.3 GMR temperature sensors

GMR gratings can also be used as temperature sensors. Two effects occur when the temperature changes: the thermo-optic effect and thermal expansion. These are, respectively, the change of the RI and the grating pitch due to a temperature variation. The influence of these two effects is investigated.

2. DESIGN AND SIMULATION

A guided mode resonance structure typically consists of a sub-wavelength periodic grating and a slab waveguide as shown in figure 1. The grating layer consists of a high index material n_H and a low index material n_L and is placed on top of a waveguide layer consisting of a core layer with high index n_H and a cladding layer with a lower index n_s. The grating has a pitch Λ and a fill factor $FF = \frac{w}{\Lambda}$.

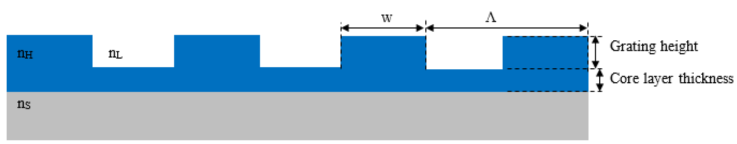

Figure 1: A schematic of a guided mode resonance structure: a grating on top of a slab waveguide.

Illuminating this structure results in a resonance in the reflection or transmission spectrum. At resonance part of the incident light is coupled into a guided mode. Due to periodic modulation, the structure becomes leaky and re-radiates the waves to the cover and substrate. These reradiated waves interfere with the incident waves leading to a dip in the transmission or correspondingly, a peak in the reflection spectrum.

The sensor is designed for operation around 850 nm to make it compatible with cost-effective CMOS-based detectors. The line shape of the resonance, which should be as small as possible, is tailored by choosing the most interesting polymers and by tuning different grating parameters, e.g. the grating height, the pitch, the fill factor and the core layer thickness, indicated on figure 1. To this end, the grating is simulated with FDTD solutions from Lumerical.

Ormocer® materials [8] are chosen, as they allow fabrication at room temperature, have a tunable thickness and are UV patternable. Further, these materials have good optical and dielectric properties and are flexible. The initial design is a grating in OrmoCore on top of a glass substrate. The pitch of the grating is 547 nm with a fill factor of 0.44. The depth of the grating is 100 nm and the core layer is 80 nm thick. A high index coating of silicon (Si) is added to increase the in- and outcoupling efficiency. The resonance for this grating is at 855 nm with a full width half maximum (FWHM) of 15 nm, corresponding to a quality factor (Q) of 57.

A lower contrast of the slab waveguide results in a narrower peak and improves the quality factor. Therefore, an extra cladding layer (OrmoClad) is added between the substrate and the core layer. The grating parameters are further optimized and a quality factor of more than 2200 for a resonance at 850 nm is achieved in simulations. Figure 2 shows the transmission spectrum of this GMR grating.

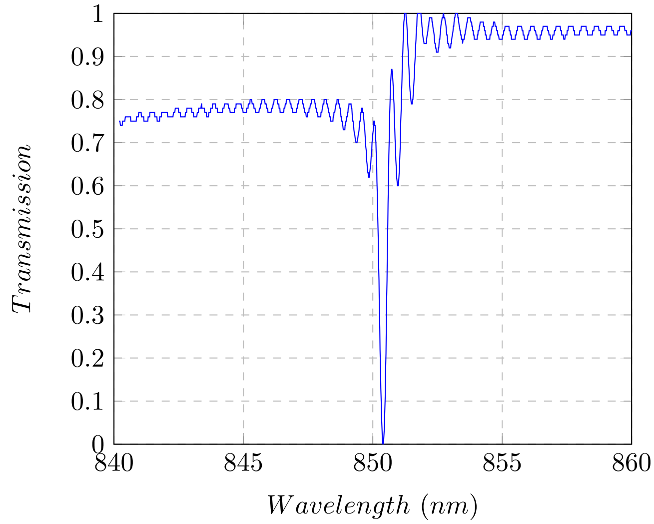

Figure 2: Simulated transmission spectrum.

To act as a sensor, the relative shift of the resonance due to an environmental change is important. The resonance wavelength can shift due to temperature variations, a change of the RI of the cover or by stretching the sensor. To investigate a particular sensitivity, a sweep of the corresponding parameter is done and the resonance wavelength is plotted as a function of this parameter. This curve is then fitted to calculate the simulated sensitivity.

2.1 Refractive index sensitivity

The design without cladding layer is optimized for aqueous solutions. By changing the RI of the cover from air to water a red shift can be detected. Slightly increasing the RI of the cover results in a simulated RI sensitivity of more than 150 nm/RIU as shown in figure 3.

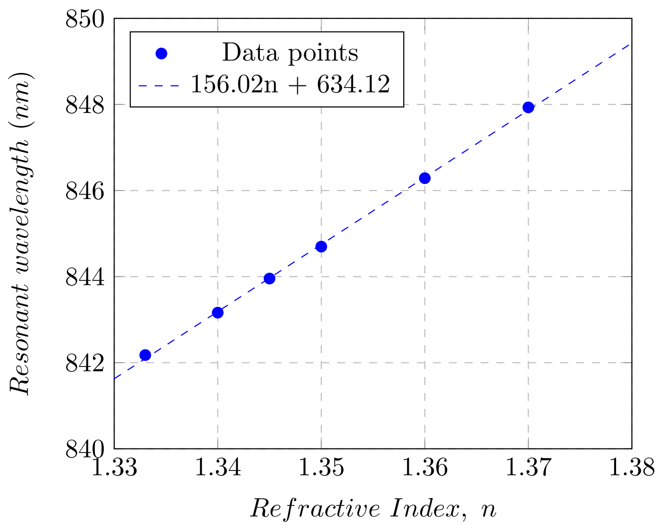

Figure 3: Simulated resonance wavelength as a function of RI of the cover of the GMR sensor.

2.2 Strain Sensitivity

The GMR sensor is also sensitive to strain as the pitch increases when strain is applied. We designed one dimensional gratings, so the sensors are only able to measure strain in one direction. Either thin foils are used directly as substrates for the strain sensors, or the flexible sensors are fabricated on a temporary carrier and afterwards released to achieve even thinner sensors. Simulating the strain sensitivity gives us a value of 0.9 pm/$\mu\epsilon$ as can be seen from figure 4. This is comparable to the sensitivity of commercial Bragg grating sensors [9].

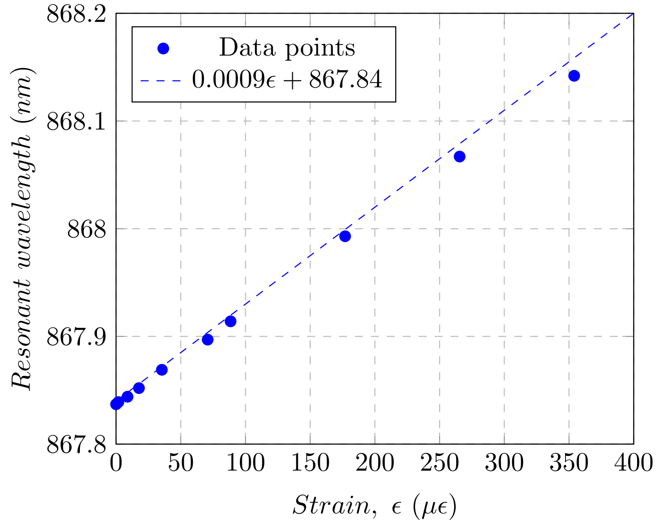

Figure 4: Simulated resonance wavelength as a function of strain.

2.3 Temperature Sensitivity

As already mentioned, two effects play a role in the temperature sensitivity. These are described by the thermo-optic coefficient and the thermal expansion coefficient. The thermo-optic coefficients of OrmoCore, OrmoClad and Si are -250 ppm/K, -270 ppm/K and 180 ppm/K, respectively [10]. For glass the thermo-optic coefficient is two orders of magnitude smaller, i.e. 3.4 ppm/K. The coefficient of thermal expansion (CTE) for OrmoCore and OrmoClad are comparable, i.e. 100 ppm/K and 130 ppm/K, respectively. The Si en glass CTE's, however, are low compared to the values from polymers, i.e. 2.6 ppm/K and 8.5 ppm/K, respectively [10]. Figure 5 shows the simulated data which reveals a sensitivity of -0.08 nm/K.

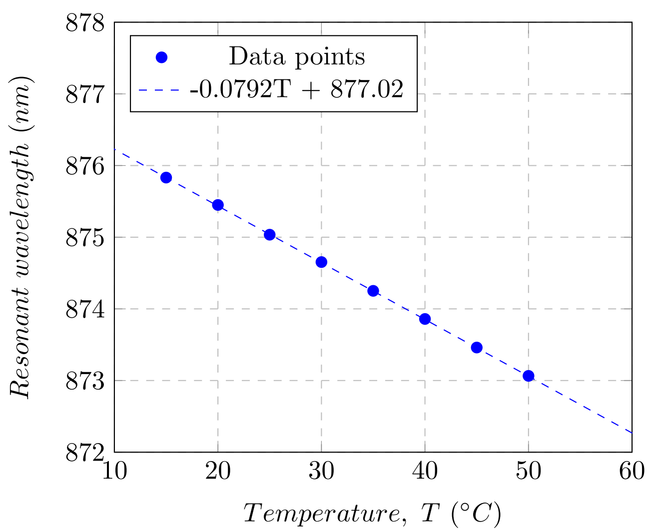

Figure 5: Simulated resonance wavelength vs temperature.

3. DEVICE FABRICATION

Using nano-imprint lithography (NIL) gratings can be realized in polymer materials at a low cost and with a high throughput. The different steps in the process are illustrated in figure 6. The master mold is fabricated using electron beam lithography. This can either be done with a negative resist, where the resist is used as a master stamp, or with a positive e-beam resist, where the grating is transfered to Si to yield a fully Si master. A first NIL step is used to fabricate the soft stamp. Soft stamp material is spin coated over the master and a flexible foil is brought in contact with a NIL roller. After a UV-curing step, the foil is released from the master and as a result the grating structures are reverse copied in the soft stamp. To imprint the gratings in the polymer OrmoCore, the same principle is used: the soft stamp is rolled over a substrate with a spin coated polymer layer and after a UV curing and baking step the grating structures are imprinted in the polymer.

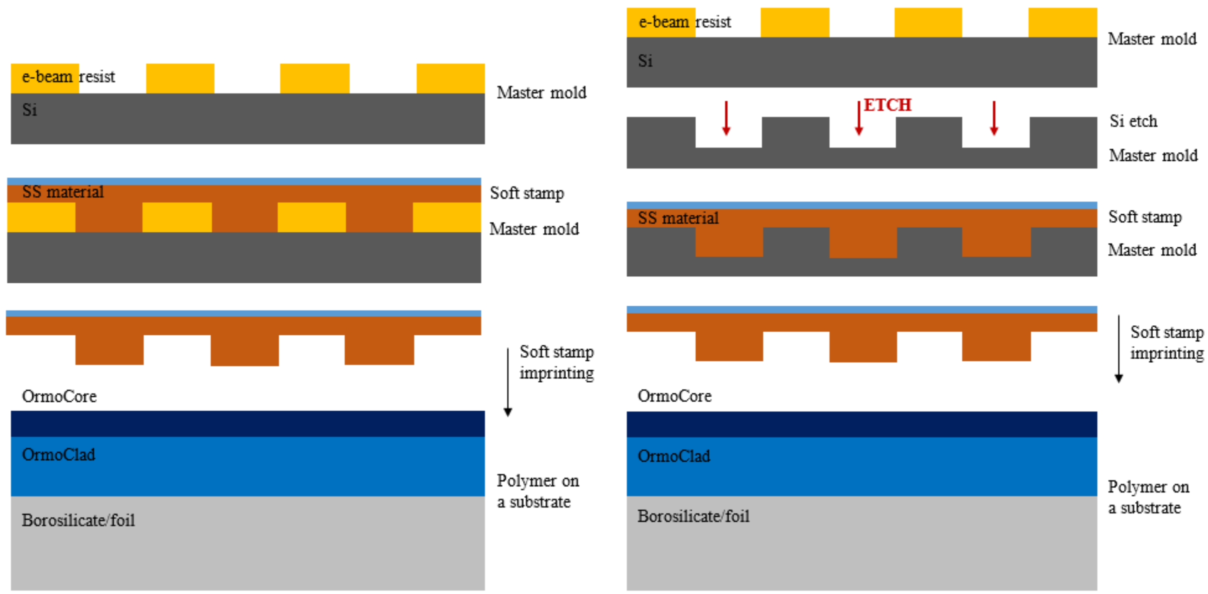

Figure 6: Illustration of the nano-imprint lithography process. Left: negative e-beam resist. Right: positive e-beam resist + Si etch.

3.1 Negative e-beam resist

The negative e-beam resist maN 2403 [11] is used to fabricate the master stamp for the GMR grating. A conductive coating is spin coated on top of the resist to realize fully developed grating lines without a residual layer in between during e-beam lithography. The depth of the grating can be controlled by the spin coating parameters. This negative resist is hard enough to be used directly as a master stamp. A gold coating is sputtered to make sure no material gets stuck between the grating lines during soft stamp fabrication. Figure 7 shows a maN 2403 master with an imprinted grating in OrmoCore.

3.2 Positive e-beam resist

The positive e-beam resists ARP 6200.09 and ARP 6200.13 [12] can also be used to realize gratings in the e-beam machine. Also for these resists a conductive coating is spin coated on top to improve the exposure conditions. However, these resist are not ideal as a master for stamp fabrication. When imprinting this grating directly, most material gets stuck in the grating lines and the gratings are destroyed. Therefore, the gratings are etched in Si to realize a Si master. The depth of the grating can be controlled by the etch parameters. With an anti-sticking-layer on top, a Si master is ideal for making soft stamps.

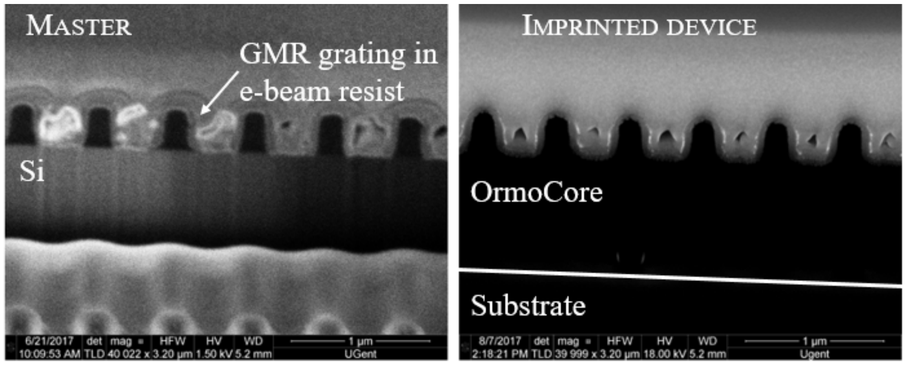

Figure 7: A cross-section of a master mold in the negative e-beam resist maN and the corresponding imprinted grating in OrmoCore.

3.3 Final devices in polymer

Next, a soft stamp is fabricated from the master stamp. This soft stamp is used to imprint the gratings in the polymer materials. Several soft stamps can be fabricated from one master mold and one soft stamp can be used many times to imprint the grating structures in the polymer.

The substrate can be either glass, favored for RI sensing or a flexible foil for strain sensing. For the first design, a 120-nm-thick layer of OrmoCore is spin coated on a glass substrate. The soft stamp is rolled in the spin coated layer and UV cured. Afterwards, the soft stamp is peeled off and the device is hardbaked. For the second design, first a 15-μm-thick layer of OrmoClad is spin coated on a 175-μm-thick foil and UV-cured. The gratings are then imprinted in a 250-nm-thick OrmoCore layer and UV cured. Finally, a thin layer of Si is evaporated on the gratings of all the samples to enhance the in- and outcoupling of the incident light. Figure 8 shows a final device with an evaporated layer of Si.

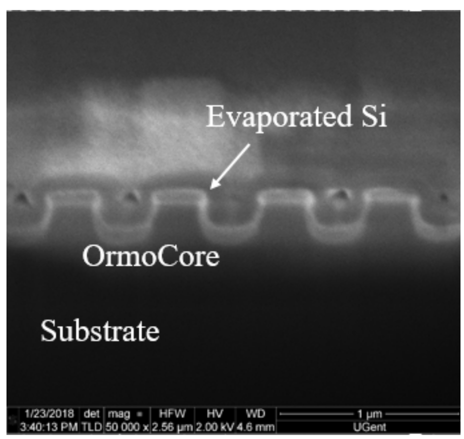

Figure 8: A cross-section of an imprinted grating in OrmoCore with an evaporated layer of Si on top.

4. DEVICE CHARACTERIZATION

The devices can either be measured in transmission or reflection. For a transmission measurement the grating is illuminated with a light source around 850 nm and the signal is captured with either an integrating sphere or a multimode fiber, connected to a spectrometer. In reflection, a 2x2 coupler is used between the source and spectrometer at one side and the GMR at the other side. Figure 9 shows a schematic of both measurement methods and a transmission and reflection spectrum of the same grating is shown in figure 10.

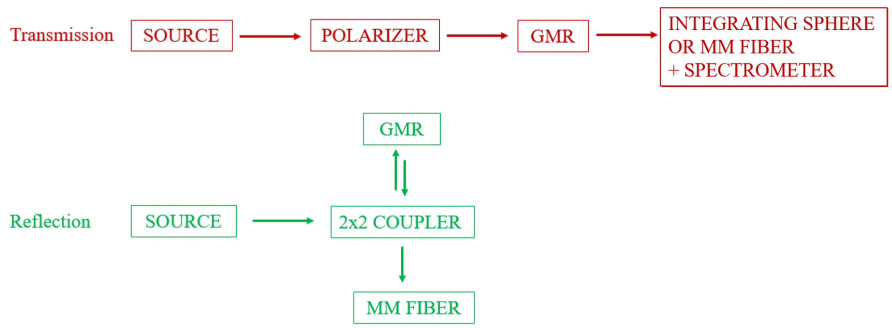

Figure 9: A schematic of a transmission and reflection measurement set-up.

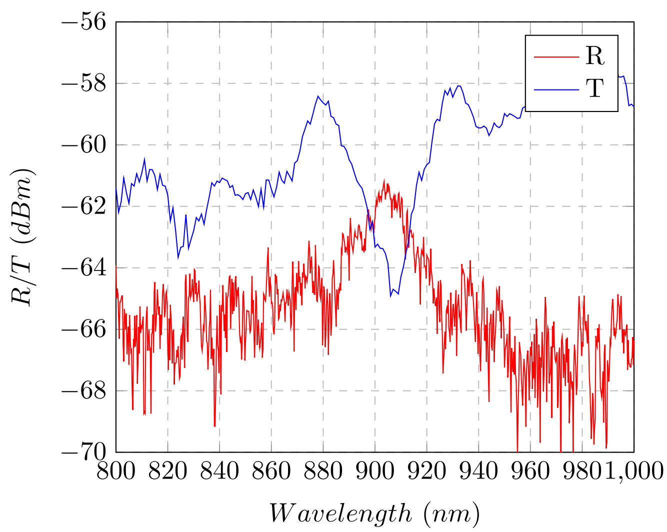

Figure 10: Measured reflection and transmission spectrum.

Different gratings are fabricated on glass and foil following both the first and the second design. The signal from a grating on glass and on foil are compared in figure 12. The quality factor of both resonances is comparable which indicates a successful imprinting process both on glass and on foil. Taking into account fabrication tolerances and to verify the influence of the grating pitch on the resonance wavelength, gratings with different pitches are written with the e-beam. The resonances of these gratings are measured and plotted on a graph shown in figure 11. The peak shifts 1.35 nm per nm pitch variation. This value is in good agreement with the strain simulations where a sensitivity of 1.52 nm resonance wavelength shift per nm pitch is calculated. This gives already an indication for the sensitivity to strain of the GMR sensor.

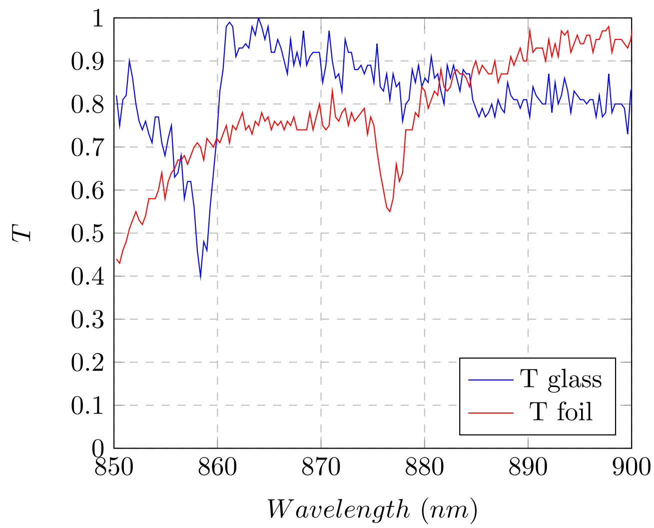

Figure 11: Measured transmission spectrum of a GMR on glass and foil.

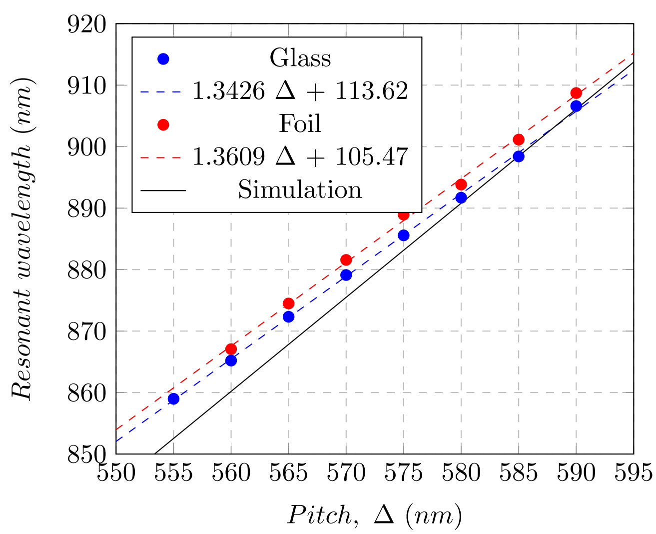

Figure 12: Measured resonance wavelength corresponding to different pitches of GMR's on glass and foil.

4.1 Refractive index

In a vertical set-up the sensor is illuminated from below and the signal is captured with an integrating sphere. Liquids with different refractive indices are deposited on the sensor. Figure 13 shows the measured resonance wavelength corresponding to the RI of the sensor's cover. A sensitivity of around 100 nm/RIU is measured, which is less than the simulated value of 150 nm/RIU.

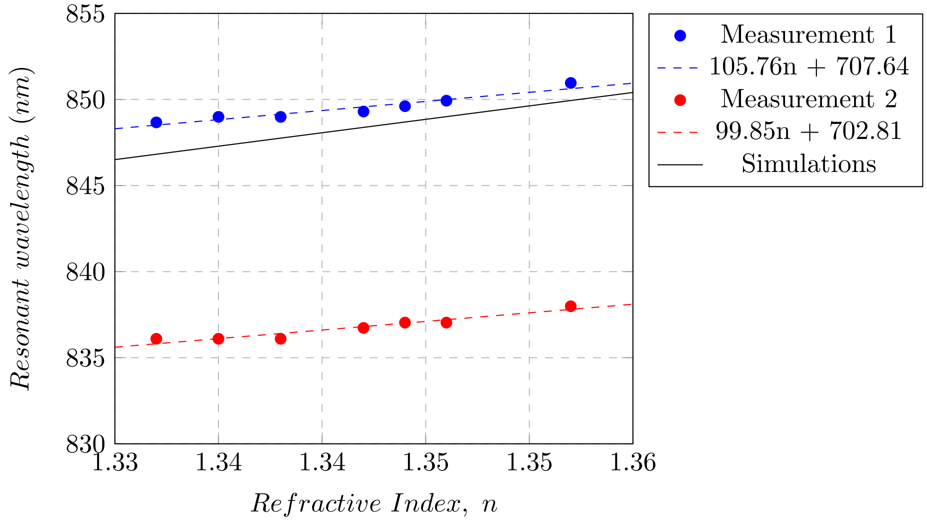

Figure 13: Measured resonance wavelength vs RI of the cover of the GMR sensor.

4.2 Temperature

For a temperature experiment, the sensor is fixed on a temperature-controlled holder and the resonance wavelength is measured at different temperatures and compared with simulations. Figure 14 shows the results. The measured sensitivity of -0.072 nm/K is in good agreement with the simulated value of -0.079 nm/K.

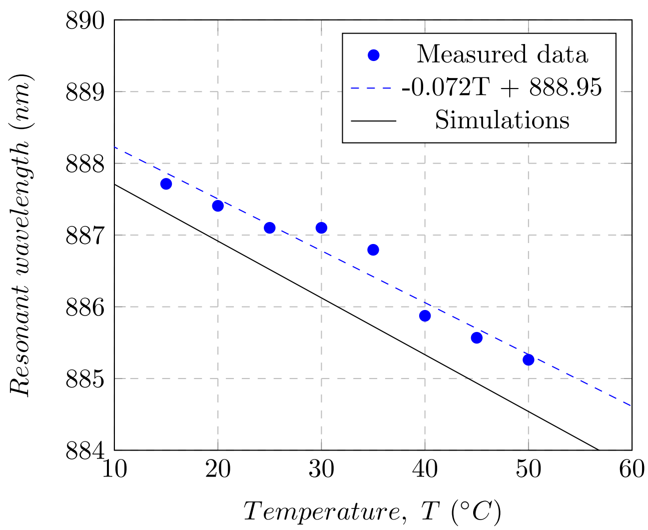

Figure 14: Measured resonance wavelength vs temperature.

5. CONCLUSION

We have shown the potential of GMR grating sensors imprinted in polymer. In this work, Ormocer® materials were chosen, owing to their relatively low loss from visible to telecom wavelengths and the fact that they are UV patternable. This latter allows us to use the cost-effective fabrication method of roller nano-imprint lithography, which is a convenient and fast way to replicate the GMR gratings.

We have studied different GMR grating sensor configurations for biosensing, strain sensing and temperature sensing. Different devices are demonstrated in theory, simulation and experiment. The devices, both on glass and on foil have shown a comparable response indicating the successful imprinting process on foil. As there is a large flexibility in design and fabrication, the sensor design can be tuned to specific needs without extensive re-optimization. Furthermore, the GMR principle allows a non-contact and easy readout, avoiding costly alignment procedures.

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