3080

Integrated Bragg Grating Sensor Applied to Detection of Phase Transitions in Liquid Crystal and Water

I.J.G.Sparrow[a], G.D.Emmerson[b], C.B.E.Gawith[a], P.G.R.Smith[a], M.Kaczmarek[c] and A. Dyadyusha[c]
[a]Optoelectronics Research Centre, University of Southampton, SO17 1BJ, UK
[b]Stratophase Ltd, Romsey, Hampshire, SO51 9AQ, UK
[c]School of Physics and Astronomy, University of Southampton, SO17 1BJ, UK

ABSTRACT

A planar Bragg grating in silica is used to form an integrated optical refractive index sensor. The device, inherently suited to remote sensing using single mode transmission fibre, is shown to clearly detect phase transitions in a nematic liquid crystal and in water. Transitions from ordered to isotropic, gas to liquid and liquid to solid as well as the reverse transitions can all be clearly identified. The sensor also allows supercooled liquid to be easily identified, a task previously found challenging by other sensor technologies.

Keywords: Bragg grating, Planar waveguide, Integrated Optical Sensors, Water, Liquid Crystal

INTRODUCTION

Numerous applications exist for a device capable of identifying, and detecting changes in, the physical state of a chemical. Ranging from agriculture to air travel such a device would allow increased automation and higher levels of safety through the detection of atmospheric conditions. The planar component described here is suited to many such applications and is demonstrated to detect phase transitions in a liquid crystal and in water. Additionally, the device can be used to identify and monitor the condensation, evaporation, supercooling, freezing and melting of water.

The use of Bragg gratings as sensor components is not new. More commonly used as stress or temperature sensors[1], they can also be used as refractometers by stripping away the outer cladding and allowing the evanescent field to penetrate and interact with a chosen analyte[2]. Small changes in the refractive index are observed as a shift in the reflected Bragg wavelength of the grating according to

$$\lambda_b = 2\Lambda n_{eff}$$

Where λ_b is the Bragg wavelength, Λ is the period of the Bragg grating and n_{eff} is the effective index that is modified by changes in the analyte. This method, initially carried out in D shaped, uniformly etched and side polished fibres[2,3,4] is known to be able to detect refractive index changes of the order of 10^{-6}. It is advantageous however, to fabricate such sensors in planar waveguide format. A key advantage of planar geometries is the ease with which arrays of sensors and other optical functionalities can be integrated into a single device. In this way, multiple elements, operating at different wavelengths can be combined along with temperature reference gratings using integrated couplers for a highly flexible device. The inherently robust nature of planar devices is also advantageous in some applications. Commonly such devices are deposited onto a silicon wafer and as such are easy to orient and mount in the desired location with little or no additional modification. All previous planar sensing technology utilising Bragg gratings has been realised using relief gratings[5]. Here we present a silica on silicon waveguiding device featuring UV written waveguides and Bragg gratings. This is the first example of such a device with a smooth planar exposed surface, therefore having less potential for damage by thermal expansion or contraction than a relief grating. An additional advantage offered by this arrangement is the intrinsic compatibility with silica optical fibre. We demonstrate the sensitivity and potential for such a device by detecting changes in the state of liquids and gases over the device.

Previous investigations into the possibility of making distinctions between supercooled and solid water have relied on relatively complex sensing methods or human intervention[6]. The results presented here provide a means of identifying the state of water purely by interrogating the wavelength response of the sensor.

DEVICE FABRICATION

The planar sample used for the sensor device was fabricated using a combination of flame hydrolysis deposition and direct UV writing. Three silica layers were deposited onto a thermally oxidised silicon wafer such that the central core layer was co-doped with germanium to provide photosensitivity to 244nm radiation. To provide additional photosensitivity the sample was placed in a pressurised hydrogen cell at 150 bar for over three days to allow hydrogen to penetrate the silica matrix. To define a waveguide and Bragg grating, the technique of direct grating writing technique[7] was used. This simultaneously defines the waveguiding and grating structures and provides a highly versatile, one step route to single mode waveguiding structures with the use of a CW laser operating at 244nm. After UV exposure the waveguide cladding directly above the Bragg grating was removed using a timed hydrofluoric acid etch process. This step leaves the waveguiding core undamaged but allows the evanescent field of the waveguide to penetrate the chemical used to replace the etched cladding. The device was then fibre pigtailed using standard telecoms single mode fibre. A diagrammatic representation of the device is shown in figure 1. Two similar sensors were fabricated in this way, one used for studies of water, the other used with liquid crystals.

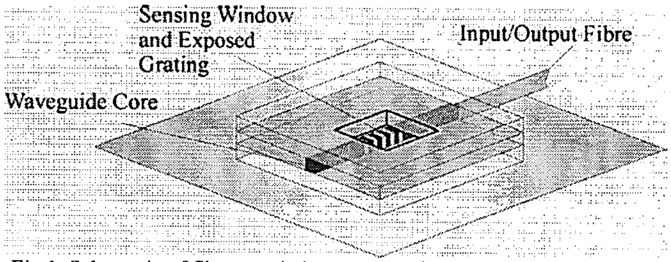

Fig 1. Schematic of fibre coupled sensor showing exposed Bragg grating.

EXPERIMENTAL

To induce changes in the physical state of the chemicals used here, temperature control of the samples was required. Mounting the device on a resistive heater or a thermoelectric cooler provided the facility to heat the sample to 100°C or to control between +35 and -5°C respectively.

The inherent temperature response of the samples used was taken prior to them being used as sensors to determine that the variation of Bragg wavelength was linear as expected. The positive temperature coefficient response is shown in figure 2 and can be seen to be linear when heating from room temperature to 100°C.

Optical measurements were taken using a broadband ASE source fibre coupled into the waveguide. Reflection spectra were recorded using an optical spectrum analyser. The input polarisation was optimised to provide a strong reflection peak and then held constant throughout. By design, the Bragg gratings were chosen to reflect at around 1550nm so that the advantages of long distance, low loss, single mode propagation associated with this wavelength could be exploited.

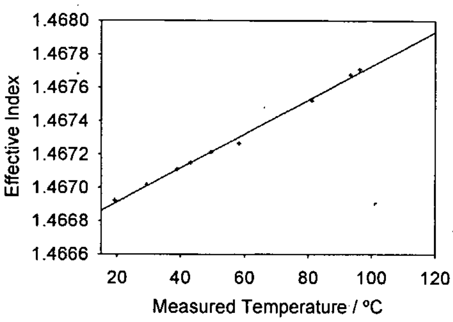

Fig 2. The linear temperature response of a sensor device in air.

RESULTS

Figures 3 and 4 clearly show the transitions from one physical state to another for different sensor configurations. The data shown in figure 3 was recorded whilst the exposed grating was covered with a nematic liquid crystal (Merck 18523). Nematic liquid crystals are known to exhibit orientational order amongst their molecules. Above a particular temperature, known as the clearing point, this level of order is lost and the liquid crystal becomes an isotropic liquid. As the temperature of the liquid crystal covered sensor increases, the effective index as measured from Bragg reflection spectra, decreases until the very obvious discontinuity at approximately 65°C. This spike corresponds to the clearing point of the liquid crystal, the sudden first order phase transition from ordered to isotropic state. It is unsurprising that this transition from one phase to another is so clearly visible as it is a result of molecular reordering of a chemical deliberately engineered to have a significant birefringence below the clearing point.

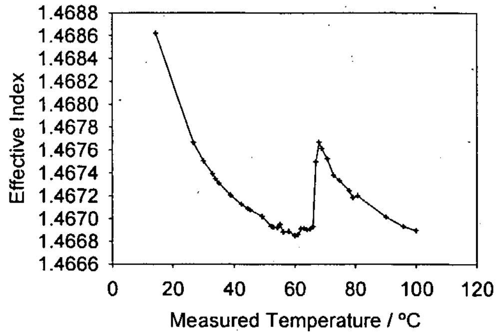

Fig 3. The variation of effective index with temperature when sensor is covered with nematic liquid crystal.

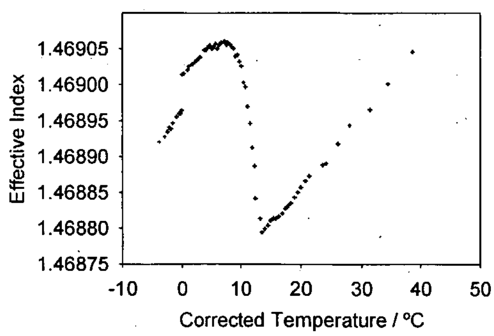

Fig 4. The variation of effective index with temperature as water covered sample is cooled to below freezing.

In contrast to the order-disorder transition of the liquid crystal, figure 4 clearly shows the phase transition from one disordered state to another. In this case, the bare sensor was exposed to air above room temperature. Using a thermoelectric cooler the temperature of the device was slowly reduced. The temperature scale shown in the figure is corrected by 4.0°C to compensate for an offset between the sensor surface and the measuring thermistor which was bonded to the edge of the thermoelectric cooler. The expected linear variation of effective index with temperature is seen as the temperature drops from 40°C to 13°C. At 13°C the refractive index is observed to begin to increase steadily due to the formation of condensation on the surface. With an approximately constant cooling rate and a regular period between each data point, it can be seen that there is a steady increase in the density of water droplets formed across the sample. In this way, the build up of condensed water can be monitored until, at approximately 5°C the surface of the sample is entirely covered with a complete layer of water. As the temperature cools further, the effective index resumes an approximately linear decrease down to 0°C. At this point there is a sharp discontinuity as the condensed liquid freezes across the sensor. The drop in the measured effective index is due to the anomalous behaviour of water whereby the solid state has a lower density than the liquid form.

Further investigation into the behaviour of water around phase transitions highlight the ability of the device to readily distinguish between water in both its solid and liquid states which occur naturally below 0°C. The phenomenon of supercooling of liquids has been known for many years and although the melting point of water occurs at 0°C it does not necessarily freeze at this temperature and can remain liquid until approximately -40°C[8]. Figure 5 shows the results obtained when liquid water was allowed to cool several degrees below zero before freezing. Initially, reflection spectra were recorded as the device was covered in water at room temperature. Gradual cooling of the device (triangular data points) showed a linear decrease in effective index until approximately -5°C. Until this point the water over the sensor was visibly still a liquid. At this temperature however, all of the water covering the sensor suddenly froze, taking the temperature at the surface of the sensor sharply back up to 0°C. After this, the temperature could be reduced further. The double-value behaviour of the data shown in figure 5 below zero degrees make it clear that the device can distinguish between the supercooled liquid and solid states of water. As the temperature was slowly taken back up above zero degrees (square data points) there is, as expected, a discontinuity at zero degrees as the water returns to its liquid state, with linear behaviour either side.

Although it is not shown here, similar tests demonstrate how evaporation from a saturated surface can also be monitored. As the volume of water decreases in thickness the evanescent field will begin to interact not just with the liquid, but also with the atmosphere above it. Thus the measured effective index drops back towards that for a dry sensor as evaporation proceeds.

These results demonstrate how phase changes of water and perhaps more significantly the point at which condensation begins to form (dew point) can be easily and simply measured with a compact and low cost sensor. Although the magnitude of the refractive index changes in question are considerably smaller than those observed when using liquid crystals, the phase transitions can nonetheless be clearly identified.

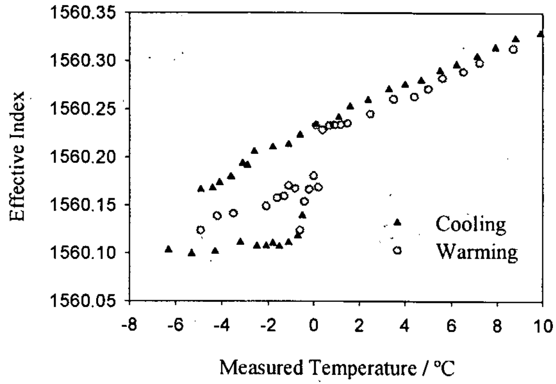

Fig 5. The variation of effective index with temperature of a water covered sensor showing the effect of supercooling when the temperature is slowly reduced.

CONCLUSIONS

We have presented a new route to using Bragg gratings as refractometers allowing highly flexible, robust and low cost sensor devices which are ideally suited to optical integration. The nature of the UV writing procedure used to define the waveguides allows arrays of sensors to be created in a single processing step, each operating at different wavelengths. Operating as single mode waveguides at 1550nm the sensors are ideally suited to being used remotely and can be readily multiplexed into an extensive sensor network using existing telecommunications technology. Using a nematic liquid crystal as well as gaseous, liquid and solid water we have demonstrated how phase transitions can be detected and, in the case of gradual condensation, monitored during intermediate stages. Such results are not limited to the two chemicals described here and the same techniques can be used for a huge range of chemicals. The nature of the UV grating writing process allows the Bragg gratings to designed to operate at any wavelength, thus allowing the sensors to take advantage of any available light source.

REFERENCES

1. Y-J. Rao, "In-fibre Bragg grating sensors", Meas. Sci. Technol **8**, 355-375, 1997
2. G. Meltz, S. J. Hewlett and J. D. Love, "Fiber grating evanescent-wave sensors", *Proc. SPIE Chemical, Biochemical, and Environmental Fiber Sensors VIII*, Robert A. Lieberman, Vol. 2836, p. 342-350, 1996
3. A. Asseh, S. Sandgren, H. Åhlfeldt, B. Sahlgren, R. Stubbe and G. Edwall, "Fibre Optical Bragg Grating Refractomer" Fiber and Integrated Optics, **17,** 51-62, 1998
4. K. Schroeder, W. Ecke, R.Mueller, R.Willsch and A.Andreev, "A fibre Bragg grating refractometer", Meas. Sci. Technol. **12**, 757-764, 2001
5. G.J. Veldhuist, J.H. Berends, R.G. Heideman and P.V. Lambeck, "An integrated optical Bragg-reflector used as a chemo-optical sensor", Pure App Opt , **7,** L23-L26, 1998
6. J. Weremczuk , Z. Wawrzyniak and R. S. Jachowicz, "A new method of super-cooled water recognition in dew point hygrometer by morphology-based image processing", Sens. Actuators, A, Physical, **111**, 240-244, 2004
7. G.D.Emmerson, S.P.Watts, C.B.E. Gawith, V.Albanis, M.Ibsen, R.B. Williams and P.G.R.Smith, "Fabrication of directly UV written channel waveguides with simultaneously defined integral gratings", Electron. Lett., **38**, 1531-1532, 2002
8. B.J.Mason, "The Supercooling and Nucleation of Water", Adv. Phys. **7**, 221-234, 1958