ECIO'05: 12th EUROPEAN CONFERENCE ON INTEGRATED OPTICS
- ABSTRACT -

PLANAR WAVEGUIDE WATER STATE SENSOR ALLOWING DETECTION OF SUPERCOOLING

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SUMMARY

A sensor believed to be the first truly integrated optical sensor demonstrating the detection of the liquid-solid phase transition of water is presented. The condensation, freezing, melting and evaporation of water are all detected with a planar silica Bragg grating operating in the 1.5 micron telecommunications window. Additionally, use of the sensor allows recognition of supercooled liquid at temperatures below the melting point of water. The device is fabricated by direct UV writing with simultaneous definition of the grating, a method inherently suited to integration with other technologies. The Bragg grating is exposed and water is allowed to condense over it. Interaction with the evanescent field causes small changes in effective index (5×10^{-6}) which can be detected, a sufficient sensitivity to identify the phase transitions of water clearly.

KEYWORD
Bragg grating, Planar waveguide, Water, Refractometer, Integrated Optical Sensors

ABSTRACT

INTRODUCTION

The formation of ice and the humidity of air are critical parameters in fields as diverse as aircraft safety and agriculture but are often given little more than passing interest in our everyday lives. As an example, airflow over an aircraft wing can be severely disrupted by the formation of ice, resulting in potentially fatal consequences. A variety of agricultural and storage applications also exist where atmospheric moisture levels directly impact costs and profitability. Thus a compact and low cost means of monitoring the state of atmospheric water is desirable for many reasons.

A number of methods exist to determine the point at which condensation begins to occur[1], making use of electrical, acoustic[2] and optical properties of materials [3]. Optically measuring the change from liquid to solid water is less common and to date has relied on reflecting signals from the water surface[4]. Here we present a planar waveguide device, inherently suited to high levels of integration, capable of both condensation and freezing point detection. Previous detection devices with the potential for optical integration have been based on the use of grating couplers or phase measurement[5] but still require external components such as prisms and polarisers. Relief gratings over optical waveguides[6] have also been demonstrated, but are not suitable for detection of ice formation as they have a high potential for damage due to the expansion of trapped water upon freezing.

The properties of real liquids can be considerably more complex than those of simple idealised fluids, and one phenomenon displayed by water and other liquids is that of supercooling. Although the melting point of pure water is known to be zero degrees centigrade, water does not necessarily freeze at this temperature and can remain in a liquid state until below approximately –40 ºC[7]. The transition from liquid to solid requires a finite amount of energy to initiate the transformation and, under some circumstances, it is energetically favourable to remain a liquid below the melting point. The distinction between ice and supercooled water has previously required human intervention, multiple sensor designs or significant processing of the sensor output[8].

In addition to identifying the formation of condensation and ice the presented device is also able to clearly distinguishing between ice and supercooled water. In addition to this previously unavailable combination of abilities, it is also possible to use the device to simultaneously measure temperature, making it a highly versatile and compact sensor. The design is based around a Bragg grating written using UV exposure, into a planar silica substrate [9]. Removal of a portion of the cladding to expose the grating results in the evanescent field interacting with the water or air above. Very small changes in the moisture content of the air or the state of the water cause a corresponding change in the effective index of the optical mode within the waveguide. This results in a change in the Bragg wavelength of the grating according to

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

Where λ_b is the Bragg wavelength, n_{eff} is the effective index and Λ is the period of the Bragg grating. Measurement of the Bragg wavelength, for example with an optical spectrum analyser, allows small changes (less than $1\text{x}10^{-5}$) in refractive index to be easily detected.
An advantage of this particular device is that it is designed to operate at wavelengths around 1550nm, the widely used telecommunications window. This is significant for two reasons. Firstly, there is wide availability of well developed optical sources and detectors with which to use the sensor. Secondly, and perhaps more importantly, the use of such wavelengths allows signals to be transmitted over large distances. Consequently, any device utilising fibre optic technology can be used at a location remote from the analysis station.

DEVICE FABRICATION

The combination of waveguide and grating used in this device is written into a 3 layer silica on silicon structure. The three layers were deposited using flame hydrolysis deposition such that the central core layer was co-doped with germanium to provide photosensitivity to 244nm radiation. To further enhance the photosensitive response, the sample was stored in hydrogen at 150 bar for over 3 days to allow in-diffusion of hydrogen into the silica core. The Direct Grating Writing technique[9] was then used to define waveguide and grating simultaneously using a continuous Ar+ laser. This technique provides a versatile means of defining single mode waveguiding structures in a single processing step. Following the waveguide definition, a wet etch process was carried out to remove the overclad layer above the grating section of the device. Hydrofluoric acid was used in a timed etch for cladding removal without waveguide core damage. To allow liquid water to be collected above the sensor, an aluminium window, approximately half a millimetre thick was bonded to the surface of the sensor to provide side walls. In order to keep the light coupling into and out of the device constant, it was fibre pigtailed. This prevents thermal effects altering the fibre to waveguide coupling and allows spectra to be taken without realignment of the fibre. A diagrammatic representation of the device is shown in fig.1.

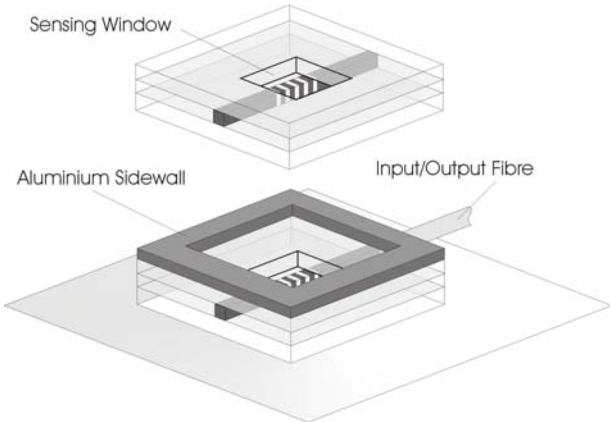

Fig. 1: Schematic of sensor device showing etched window to expose Bragg grating.

EXPERIMENTAL

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.
Initial measurements were taken with the sample heated above room temperature to determine the change of grating response in air, with no surface condensation. The variation of centre wavelength was found to be linear with a temperature coefficient of 1.01×10^{-5} C^{-1}. Subsequent measurements were carried out by heating and cooling the sample as appropriate to allow the formation of condensation and to cause freezing or melting of the condensed water.

RESULTS AND DISCUSSION

Figure 2 shows the result of melting ice, formed by cooling the sample and allowing the condensed water to freeze. As the sample was slowly allowed to warm back to room temperature, reflection spectra were recorded. A discrete step in the spectra is clearly visible at a measured temperature very close to zero degrees. This corresponds to the density (and associated refractive index) increase as the ice turns to water. Either side of the phase transition the spectra displays the expected linear variation of effective index with temperature.

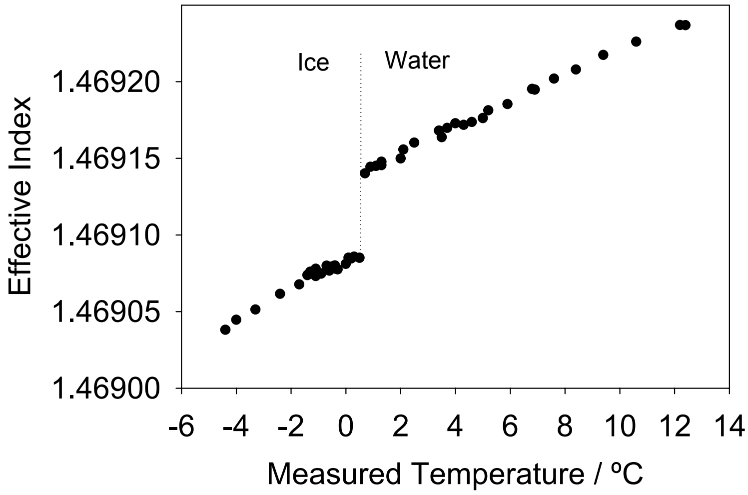

Fig. 2: Variation of effective index when allowing ice over the sensor to melt

Figure 3 shows the result of first cooling the sample from room temperature, until the resulting condensation froze, and then allowing it to re-warm. The high temperature end of the cooling curve shows the linear response of the sample with no condensation. Condensation begins to form at approximately sixteen degrees C when the effective index sharply increases due to water being present over the grating. After the initial formation of condensation and before total water coverage, with approximately five minutes stabilisation between each temperature step, there is time for coalescence and formation of water drops upon the sample. Upon coalescence, a region of the sensor with a depleted amount of water is temporarily created before condensation reoccurs. This depletion and re-condensation is believed to be the reason for the apparent noise on fig. 6 in the condensation phase. At just over zero degrees when the sample was totally covered by just two or three water drops, it was held at temperature to allow further condensation and final coalescence of the drops to form a complete water layer.

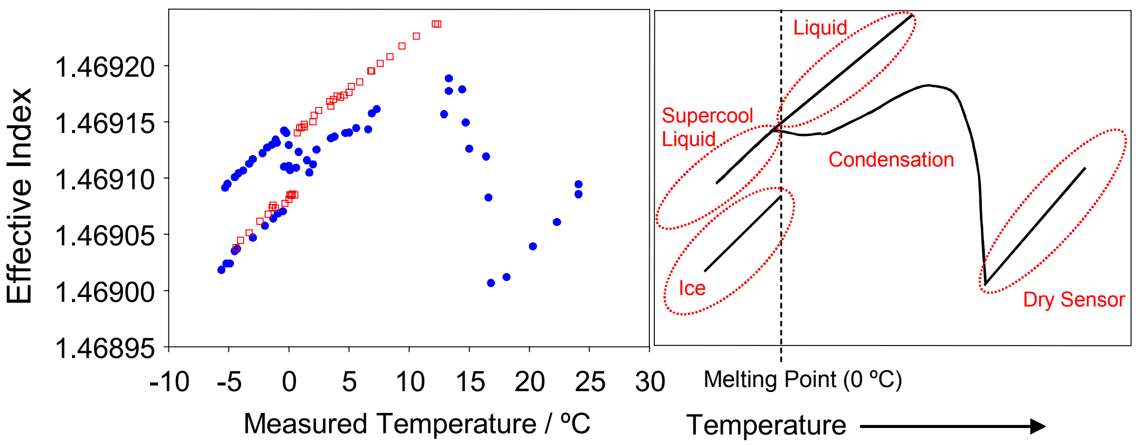

Fig. 3: The cooling and warming of the sensor. *Left:* Measured data as sample is first cooled (solid circular data points) and then allowed to warm (open square data points). *Right:* Line drawing to represent the trend shown by measured data. Labelled ovals indicate the state of the water over the sensor at each point.

Below zero degrees the cooling curve appears to show that the condensed water is in two physical states with different refractive indices. This is due to the liquid entering a supercool state. The effective indices caused by this state are given by the upper of the two lines on the cooling curve. The water remains liquid until approximately minus five degrees when the liquid to solid transition suddenly occurs and all of the water on the sensor freezes. The latent heat of freezing takes the sensor temperature back to zero degrees, the melting point of water. As the ice cools back below zero, the measured spectra follow the lower of the two traces shown on the cooling curve. When the sample is allowed to warm, the effective index of the frozen sample retraces itself until the melting point is reached and the solid-liquid transition, as shown in fig. 4 is seen. As expected, fig. 6 shows that the data taken with a total covering of condensed water meets that of the melted water and forms a continuous data set as the water is allowed to warm.

FURTHER DEVELOPMENT

The linear temperature response of the device allows the additional use as a temperature sensor. Integrating two gratings in the same waveguide operating at different wavelengths allows one unexposed grating to measure temperature whilst the other acts as the sensor as described above. Similar techniques have previously been used to allow for temperature compensation in fibre Bragg grating refractometers[10]. Such integration of multiple gratings is a trivial procedure when using the Direct Grating Writing fabrication technique. The addition of extra gratings to a device design is purely a matter of modifying the software used in the UV writing process.

Devices such as that presented here are ideal for integration with other functions. The UV writing process does not require high post-exposure anneal temperatures which may damage pre-existing structures on the substrate. Other sensor technologies or more complex waveguiding structures can therefore realistically be incorporated without many of the issues that make integration of multiple technologies problematic.

SUMMARY

We have demonstrated a highly sensitive sensor that can be used to measure the condensation, freezing transition and evaporation of water. The presented data shows that supercool liquid water can be readily differentiated from solid ice, a feature that distinguishes this design from previous optical methods. The level of measurement sensitivity and flexibility of the fabrication approach makes the sensor particularly suited to high levels of integration and of use in a variety of applications. Although we have concentrated on the sensor's use in studying pure water, the same method can equally well be applied to other liquids to monitor formation and phase transitions.

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