Evaluation of the durability and performance of FBG-based sensors for monitoring moisture in an aggressive gaseous waste sewer environment

Lourdes S. M. Alwis, Heriberto Bustamante, Bernhard Roth, Kort Bremer, Tong Sun and Kenneth T. V. Grattan

***Abstract*— Measurements of the rate of corrosion in concrete sewers need to take into consideration the humidity in the environment and thus its accurate measurement becomes critically important. Introducing a novel approach to do so, tailored Fiber Bragg grating (FBG)-based humidity sensors have been evaluated *in situ* to examine their durability, time response and stability when used in measurements over an extended period of time under the aggressive gaseous environment of a gravity sewer, experiencing high levels of both humidity and hydrogen sulfide gas. The critical, humidity monitoring element in the probe is based on a moisture-sensitive polyimide coated FBG, using the calibrated and reproducible peak wavelength shift in response to moisture variation, in this case operationally in the sewer. To optimize the device for this environment, two different designs of the probe assembly were configured using different material, thus aiming to provide durability in the harsh environment in the long term. The aim of the probe design evaluated was to achieve good sensitivity to humidity as well as to protect the sensing elements from the aggressive environment and which had rendered ineffective the electrical sensors placed in the sewer and used for cross-comparison. A full evaluation of the packaged sensors *in situ* was undertaken over a period of 5 months, during which the sensors were constantly subjected to high, but varying levels of humidity and wet hydrogen sulfide gas. The results are highly encouraging, showing superior performance of the configured fiber optic sensors used over a conventional electrical sensor when the results of the cross-comparison study of the performance were evaluated. These outcomes show a promising future for optical fiber sensors to be employed for measurement of humidity in the long term in harsh environmental applications such as this.**

***Index Terms*— Fiber Bragg grating, Humidity sensor, Optical fiber sensor, Polyimide**

I. Introduction

ACCELERATED corrosion in the major infrastructure assets worldwide is an important problem which must be tackled rapidly in order to avoid long term damage and expensive replacement strategies. According to Brongers et al. [1] sewer assets are being lost, due to corrosion, at an estimated annual cost of approximately US$14 billion in USA. Sydney Water (SW), the collaborating industrial partner in this work, has a focus on effective waste water management with approximately 800 km of corrodible gravity concrete sewers of at least one dimension of ≥400 mm, and 22 000 km of sewers with corrodible manholes and vent shafts, this being typical of major utility companies in the developed world. This significant facility carries with it large operational costs for mitigation and significant capital outlays for rehabilitation, repair and importantly, odor management. If uncontrolled, corrosion can reach the reinforcement and cause collapses, which then results in major repair and replacement costs to asset owner. In addition, moisture acts as the dissolving medium for the gaseous hydrogen sulfide, H_2S, which comes in contact with both concrete and bacterial biofilms [4]. Chemical corrosion in the "dry" part, above the waste water, of the gravity sewer is due to microbiologically generated sulfuric acid that corrodes the concrete and, if left unattended, will reach the rebar leading to the chemical corrosion of the rebar. This needs to be avoided as it can lead to sewer collapses and expensive repairs. Although forced ventilation can be used to remove the H_2S, the malodorous nature of H_2S requires expensive air treatment prior to any air to be discharged to the atmosphere through ventilation. [2]. In addition, forced ventilation is only effective over a limited distance and consequently, many sewers – especially those in warmer climates and those that carry industrial discharges or ageing/septic sewage – suffer from H_2S-related corrosion [2]. This is the key source of the sewer degradation underpinning the need for this study.

Concrete sewer corrosion is thus dependent on humidity which, although variable, is high in the overhead space and this issue is evident in this project. It is clear that to be able to predict corrosion rates (and thus schedule maintenance more efficiently) it is necessary to have reliable humidity measurements on the surface of the concrete. The influence of humidity on the rate of corrosion of sewer concrete is over-looked, partly because it is difficult to measure under the

L. Alwis is with the School of Engineering and the Built Environment, Edinburgh Napier University, UK (email: l.alwis@napier.ac.uk). H. Bustamante is with Sydney Water, Parramatta, Australia (heri.bustamante@sydneywater.com.au). K. Bremer and B. Roth are with Leibniz Universität Hannover, Germany (kort.bremer@hot.uni-hannover.de, bernhard.roth@hot.uni-hannover.de). T. Sun and K.T.V. Grattan are with the School of Mathematics, Computer Science and Engineering, City University London, UK (t.sun@city.ac.uk, k.t.v.grattan@city.ac.uk)

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additional constraint of the acid conditions seen when H_2S is present. A problem that has been known for some time is that the aggressive sewer environment rapidly destroys the current group of available electronic sensors that have been evaluated previously in this environment by SW because they corrode in the constant high humidity and low pH (acidic) environment and thus these desired data cannot readily be obtained using conventional approaches. A further problem of the conventional electronic sensors is a susceptibility to becoming and remaining 'wet' in use and thus causing errors in the readings due to a limited dynamic range. They are also difficult to multiplex and to use at long distances from each other or from the 'base station', due to the need for long cable runs which may be inconvenient and also expensive.

This collaborative research tackles this issue through the design, calibration and long term evaluation of an alternative sensor system which will give the data needed. In light of the above discussion, there are several key requirements that underpin the design and thus allow creating the specification for the sensor itself as no suitable commercial devices exist. Sewer operators needed sensors that can last as long as possible with minimum maintenance. Therefore, the photonics sensors were designed to undergo extremely high relative humidity in a corrosive gaseous environment. Based on the sewer operator's requirements sensors should be able to function reliably and in the long term in this environment – in this case for a period of at least several weeks or longer with minimum or no maintenance (and be capable of use in other similar, challenging environments and potentially reused after exposure such as this). The sensor should measure RH from a low level (minimum 20 %RH) up to 100 %RH, and be fully compensated for temperature changes in the working environment. The dynamic response to humidity changes should match or better a typical commercial electronic sensor and thus have a response time (noting that this changes with RH) which allows a 5% RH change in typically 15 mins at 80+ %RH (and less at lower RH values). The sensitivity of the device should be such as to allow the discrimination of RH changes of <1% RH. Most importantly, it should work in a sewer where there are high levels of wet H_2S gas present continuously. The sensor should be specified to be physically robust to be installed and when removed from the sewer by operators, demonstrate that it has been unaffected by the aggressive environment.

To achieve this, it was recognized that fiber optic sensor technology offered a strong prospect for the basic sensor design. Conventional electronic humidity sensors have been seen to fail to operate for more than a few days in tests carried out in the sewer environment. Fiber optic-based sensor systems, as discussed below, play a full role in providing a response to the needs that such a choice provides, possessing a number of advantages over conventional electrical/electronic sensors in general: such as immunity to electromagnetic interference, chemical inertness, light weight and low mass (which facilitates 'drying' after use), a multiplexing capability, high thermal stability and remote sensing ability. All of these make them well suited to both general and remote sensing for this application. In light of the deficiencies of conventional technologies for this particular environment, these advantages make them ideal candidates for measurement applications where such conventional electrical/electronic sensors are found to be inappropriate or simply would not function. Thus considerable research effort on fiber-optic (FO)-based techniques for humidity sensing has been seen [10-12], much of it by the authors, and this provides the basis for the sensor design approach taken in this work and the expansion to operation in a sewer for an extended period.

This paper describes the collaborative project with SW, City University London and Edinburgh Napier University. It provides an evaluation of the design of several packaged sensors, tailored to this environment, illustrating the detailed laboratory fabrication and calibration carried out followed by the results of the evaluations conducted in the sewer environment in Sydney. The data obtained are reviewed in detail and the conclusions that could thus be reached discussed, together with recommendations for further work that arise from the study.

II. Sensor Evaluation under Aggressive Environment

An optical fiber grating-based technique has been chosen in this work as the basis to show that the key challenges arising from the highly aggressive sewer environment could be addressed, ensuring both the durability of the sensor design and ease of operation within the scope of the evaluation defined by the industrial partner, SW.

A. Test Procedure

In order to assess the feasibility of optical fiber sensors, a pilot test was conducted at a SW site for a period of 4 weeks. This followed from a period of design and preparation work undertaken in the laboratories to create a series of sensors tailored to the needs of this application. Upon satisfactory results, the sensors were then left in the sewer for a period of 5 months. At the end of this period of intensive exposure, the sensors were then re-calibrated to evaluate their performance.

B. Fabrication and calibration of the sensors

For this particular application FBGs were fabricated using phase masks illuminated by light from a 248 nm KrF excimer laser with a pulse energy of 12 mJ and a pulse frequency of 200 Hz. In this way, there was maximum understanding, and thus control of the 'baseline' characteristics of the gratings which underpinned the sensor design itself. The period of the phase masks used were carefully chosen so that their reflection wavelengths were within the C-band, for ease of interrogation. Thus the FBGs used of the humidity sensors were written at wavelengths of 1558.8 nm, 1552.9 nm, 1559.4 nm and 1558.8 nm; similarly, for the temperature-only sensors these were written at wavelengths of 1540.4 nm, 1543.6 nm, 1542.4 nm and 1542.7 nm: thus to allow each of these easily to be differentiated by their primary wavelength characteristic.

An in-house dip-coating system was used for coating the FBGs. The in-house dip-coating setup consists of a stepper motor that controls the dipping and rotation of the holder (containing the grating) into the solution and to the oven

respectively. Prior to the coating of the polyimide (PI) polymer layer, the grating was annealed at 185 °C for 3 hours to ensure stability. The PI material used was supplied by HD Microsystems in liquid form, having a refractive index of 1.70. In the sensor fabrication, each grating was first treated with 3-aminopropyltriethoxysilane (3-APTS) in order to obtain an aspect of the device design that is very important for long term stability – a good bond between the fiber surface and the PI liquid. This was achieved by preparing a 0.01% 3-APTS solution which was dip-coated on the grating surface at a speed of 13 mm/min following which the grating was placed in the oven at 130 °C for 15 minutes and the PI solution was used to coat multiple layers of PI on the fiber. The coating speed again was 13 mm/min and the grating was placed in the oven at 150 °C for 5 minutes for each layer and after depositing the final layer, the PI coating was cured in the oven at 180 °C for an hour.

Previous work by some of the authors has shown the need to optimize the coating thickness to enhance the performance of the sensors [12]. Coatings are built up from multiple layers of the polymer material which is applied and then baked between each layer. Two different coating thicknesses were first analyzed in order to determine the suitable coating thickness that needs to be applied for effective sensor design. After careful trials, 7 and 15 layers of moisture sensitive polyimide were chosen for the task. Upon coating the polymer using the setup described above, the sensors were subjected to a RH calibration over cycles between 85 and 90 %RH. Fig. 1 shows the comparison here between these two coating thicknesses: the RH response of the thinner and thicker polymer coated sensors to humidity variations while temperature was kept constant at 40 °C.

It can be seen from the results that while the thinner coating takes less than 15 minutes to saturate, the thicker coating takes nearly an hour for such saturation to occur. However, the sensitivity of the thicker coating is approximately more than 2.5 times that of the thinner coating. Depending on whether it is more important to achieve a faster response or to obtain a higher sensitivity, the appropriate coating thickness needs to be specified. In this case, the thinner coating thickness was chosen so that a rapid response could be achieved but this can be tailored to the specification required by the environment in which the sensor is used.

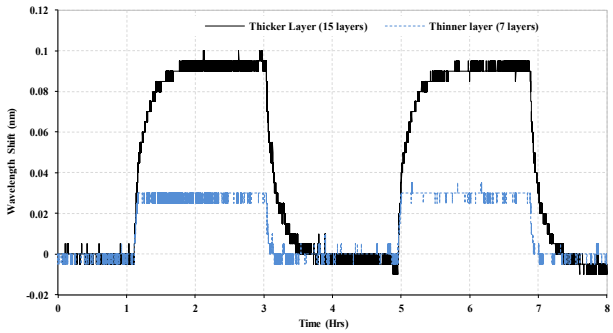

Fig. 1. Sensor RH response performance: comparison between thinner and thicker polymer coating thicknesses.

C. Sensor design and packaging for use in the aggressive gaseous sewer environment

Following the above analysis, four different sensor elements were fabricated for evaluation – to allow for breakage or damage during installation, two of each type were used. In each, the sensor package contains two FBGs in series, one for temperature measurement only and the other a FBG coated with a thinner coating of moisture sensitive polyimide for RH measurement. As discussed above, this was because a more rapid response could be achieved with a thinner coating and the sensitivity is adequate enough for the target application. The four (at this stage un-packaged) sensors were then calibrated using an environmental chamber in a setup shown in Fig. 2.

The sensitivities for each sensor both to temperature and RH were recorded so that the calibration can be established when used for actual RH/temperature measurement when they are placed inside the sewer plant environment.

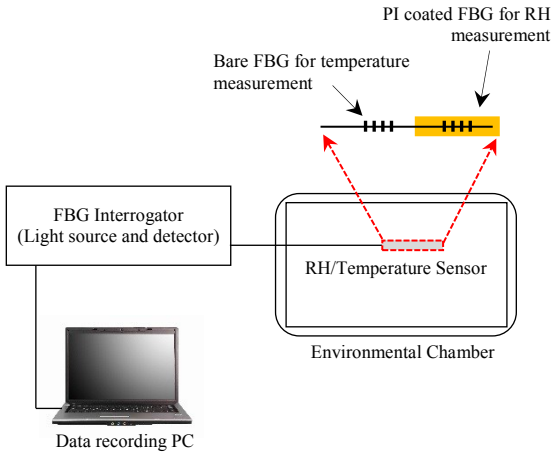

Fig. 2. Experimental calibration setup of a single sensor probe.

Once the sensors were calibrated, several potentially suitable packaging designs were evaluated to optimize performance in the known aggressive environment. Two different packaging options that were seen to be the best candidates for the evaluations to be carried out were explored:

Sensor A - 3D printed epoxy resin packaging

This approach was chosen as it was inexpensive, quick to fabricate and could be done 'in-house'. The design comprised a printed epoxy rod, with a small hole in the middle into which the fibers could be inserted. The region of the fiber containing the sensing regions was exposed to air on one end which is protected with a 3D-printed perforated cap, which could easily be removed for inspection (or indeed redesigned and replaced if it became contaminated). This sensor packaging is illustrated in Fig. 3 where a comparison to the commercial electrical sensor can also be seen, (as used for in the in-sewer evaluation tests).

Sensor B – PEEK (Polyether ether ketone) packaging

A second approach to the sensor design was used, based on a PEEK packaging design. This was a more complex design to implement, but potentially more robust and chemically

resistant. These packaging were fabricated by drilling a cavity in the middle of commercially sourced PEEK rods, while the walls of the cylinder shape (that results from the drilling) is drilled with holes to allow the atmospheric gases in the sewer to interact with the sensor. This packaging consists of 2 layers of drilled PEEK rods in which the inner rod contains the sensor followed by a Teflon wrap surrounding the internal rod, which is then protected again with the second PEEK drilled rod. This creates a robust configuration for the sensor system for the aggressive environment and the exterior view of this packaging can be seen in Fig. 4.

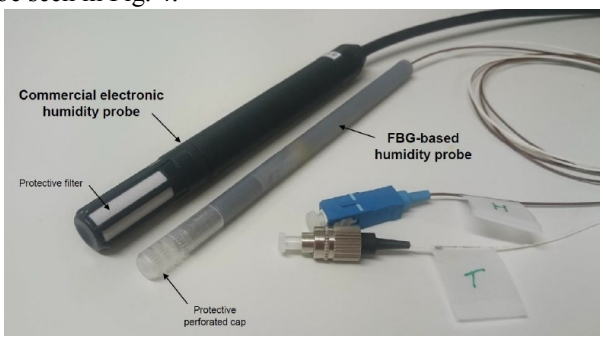

Fig. 3. Sensor system designed using 3D-printed epoxy packaging (with fiber optic connections to the interrogation system) (on right) and for comparison a conventional commercial electronic humidity probe (left).

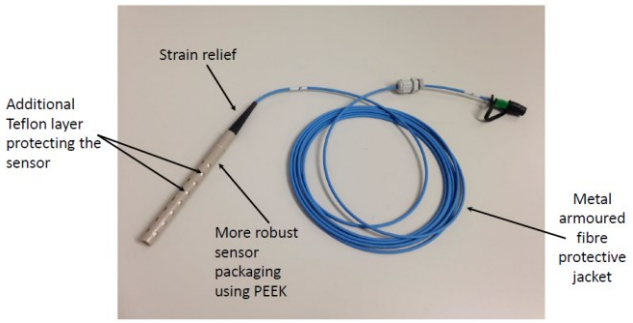

Fig. 4. Sensor system designed using PEEK packaging (with fiber optic connections to the interrogation system).

Two Sensor B RH monitoring grating-based sensors were fabricated using this PEEK-based design while two other sensors, of Sensor A type were produced using the 3D printed epoxy design. All 4 sensors were calibrated in a RH/temperature chamber prior to shipping to SW for the actual tests on site.

III. Results and Discussion

A. Results from preliminary evaluation over 4 week period (prior to the extended exposure period)

Following the calibration of the sensors to a range of humidity and temperature excursions, the responses of the four probes (2 x Sensor A and 2 x sensor B) were evaluated using a commercial 4 channel FBG interrogation system (Micron Optics SM125), to varying humidity and acid environment and the effect of prolonged exposure to the sewer environment, over a period of 12 days, can be seen from Fig. 5.

For ease of analysis the outputs from only two of the sensors (1 each of Sensor A and Sensor B types) is shown. The results indicate that both these representative sensors produced a strong dynamic response, recording the humidity levels present, both at or near 100% (as would be expected from the high humidity present in the extreme environment) and in two other cases where the humidity was rapidly lowered (to ~30 %RH by being taken out of the sewer tank to room conditions at the test site) and then recovering rapidly to measure ~100 %RH humidity when put back in the sewer. Thus *both* sensor system designs showed a clear dynamic capability in that they could respond to changes, both increasing and decreasing of humidity, from an initial level. Also, at the end of the 12-day test period, again this major RH fluctuation was repeated several times using the same technique. A very consistent response behavior was observed for the major fluctuations in humidity which were created (when the sensors were taken in and out of the tank) as well as after the sensors being left inside the sewer for over 10 days with no intervention.

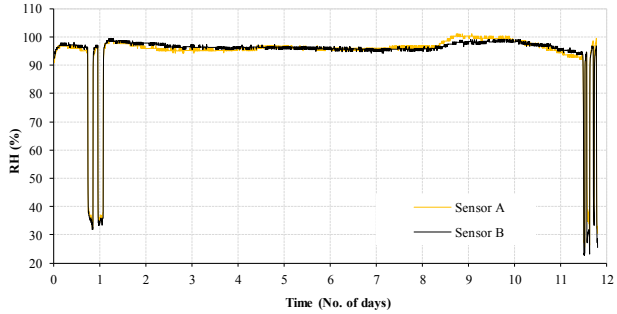

Fig. 5. Performance of the optical sensors to varying humidity conditions. At the beginning and end of the tests, the humidity was reduced from ~100 % to ~30 %RH and back, as shown, for both Sensor A and Sensor B types.

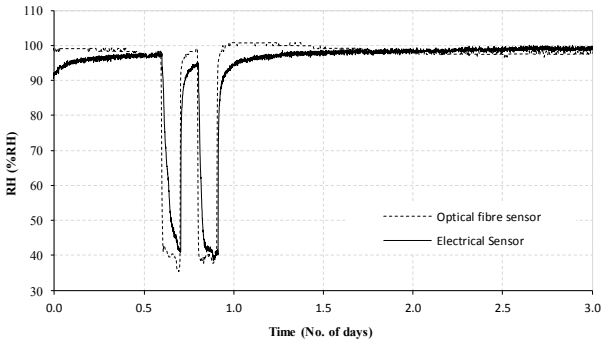

Fig. 6. Comparison between the responses of the optical and electrical sensors.

Since all 4 sensors used were seen to respond similarly to the changes in the RH, one of them was chosen and used for a more detailed analysis of the response of the RH sensors, this being representative of the performance of all. The rapid fluctuation in humidity that was created to test this eventuality was captured by the sensor very successfully. This response was then compared with that of the commercial electrical sensor response, noted in Fig. 6, where it can be seen that the response time of the electrical sensor is much longer than that of the optical sensor. This is an important consideration and shows

one of the major advantages of the low mass optical fiber-based sensor design when compared to the electrical sensor.

From the results obtained from these initial tests undertaken, the following key outcomes were seen and comparisons were made:

Response time: The electrical sensor takes a longer time to saturate and once saturation is obtained it takes a longer time to respond and return to the original RH reading. This effect is likely due to the condensation of moisture on the sensing part of the capacitance based RH probe. By contrast, the optical sensors respond more rapidly and gave values of RH which were seen to stabilize as would be expected, in less than 15 minutes. The delay demonstrated by the electrical sensor also involves the larger volume of the capacitive sensor probe and the location of the opening allowing moisture into the sensing part of the probe, when being placed on the side rather than at the tip of the probe.

Durability: It was visible from a simple inspection that the packaging of the electronic sensor is hugely altered after exposure to the sewer environment. This can be seen from Figs. 7 and 8 where the degradation of the packaging is evident (the internal condition was not examined as the sensor was not easily demountable). By contrast the optical fiber sensors, both Sensors types A and B perform well and little degradation can be seen – indeed what is seen occurs only to the outside packaging, which is superficial. It should be noted that the sensor design was not yet optimized but still functioned well.

Fig. 7. Visible significant degradation of the electrical sensor at the end of the test period (left): comparison with an unused electrical sensor (right).

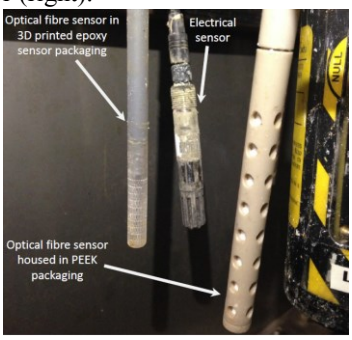

Fig. 8. Visible status of the electrical and the optical sensor packaging after the test.

Sensitivity: It can be observed from Fig. 6 that the indication from the electrical sensor probe was always ~100 %RH, whereas the measurements made using the optical fiber sensor probe showed a variation in RH from less than 100 %RH and between 97–100 %RH, this varying with time. This demonstrates that the electrical sensor, once saturated, tends to remain so throughout the remaining exposure time until a significant drop in the RH occurs — indicating the failure of the sensor to monitor accurately the true conditions of the test environment. This may be due to the high mass and failure to dry out properly when wet initially during use.

B. Evaluation and calibration after 6 months exposure period

In order to assess the long term functionality of the sensors, they were left in the sewer environment for a period of several further months and at the end of a 5 month period, they were removed and taken to the laboratory for evaluation and re-calibration. This was done to evaluate their functionality and sensitivity following the continuous exposure to the aggressive environment during that period. All 4 probes (2 x Sensor A and 2 x Sensor B) were re-evaluated in this way and it was found that all four PI-coated sensor elements had survived exposure to the aggressive environment. Figure 9 shows four views of the probes after the extended exposure, with the Sensor A type (the 3D-printed probes) showing a higher level of encrustation than the Sensor B type (the PEEK probes), which, given the more inert nature of the material, is not unexpected.

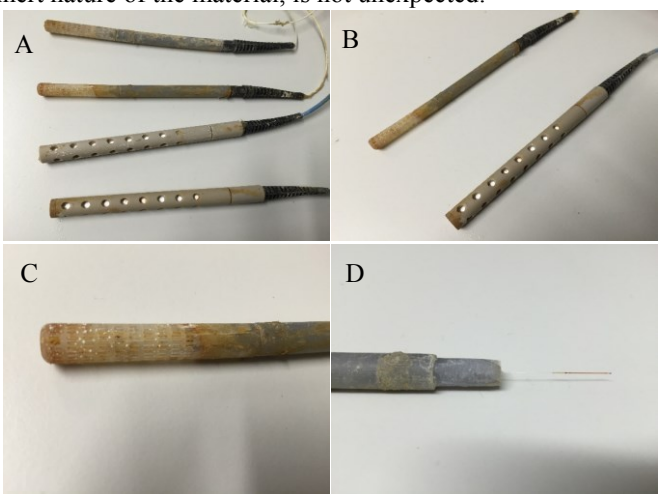

Fig. 9. The sensors after the extended exposure for 5 months in the aggressive sewer environment; A – 2 x Sensor A and 2 x Sensor B showing visible surface degradation; B – closer view of 1 x Sensor A and 1 x Sensor B; C – close-up view of the end of Sensor A and the cap tip; D – Sensor A with the cap removed, showing the undamaged sensor element.

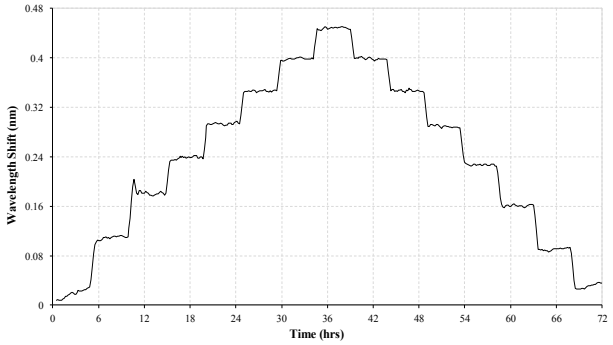

Fig. 10. RH calibration results of Sensor B subjected to extended exposure to RH saturation levels over a range from 20 – 90 %RH.

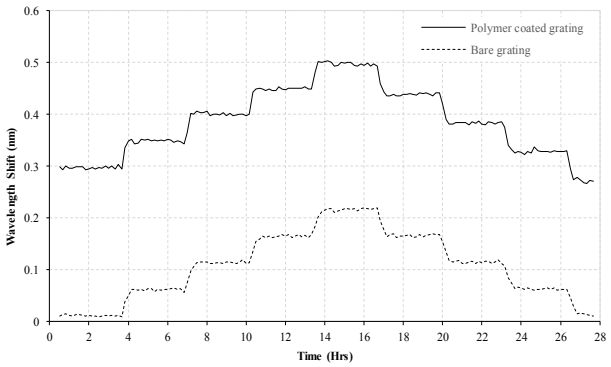

Fig. 11. Temperature calibration results of Sensor B over a range from 30-50 ºC showing both the bare grating and the polymer coated grating.

The re-calibration and performance re-evaluation tests were then performed by subjecting the sensors to an extended duration of saturated humidity levels, initially at a stable temperature. Figures 10 and 11 show the dynamic response of the RH element of one of the probes (Sensor B with the PEEK packaging) under varying humidity ranges from 20-90 %RH, undertaken in steps of 10 % in RH and in temperature over a range of 30-50 ºC, in steps of 5 ºC respectively. The bare grating has a temperature sensitivity of 10.3 pm/ºC and this is maintained (within the uncertainty of the measurement of ± 0.5 pm) for the coated grating (at 10 pm/ºC), as can be seen from Fig. 11. Thus it can be seen that the sensor still responds well to the re-calibration through the humidity and temperature variations created in the laboratory in a commercial stable humidity/temperature chamber.

Figures 12 and 13, together with Table 1, show the effect of the re-calibration of two sensors, one from each of the Sensor A and Sensor B designs, to both rising/falling humidity and temperature respectively. As can be seen from Fig. 12, the sensitivities obtained from the RH calibration of Sensors A and B at the end of the exposure tests to the aggressive environment were on average 4 pm/%RH and 6 pm/%RH respectively which compared very favorably to the calibration of the sensor carried out after fabrication and before being put into the aggressive environment, of 4-6 pm/%RH, and showing overall negligible hysteresis and retains a linear response (with an uncertainly of ± 0.2 pm). The small difference in sensitivity results from the slightly different fabrication conditions for each of these sensors – they are individually made and calibrated for this experiment and thus show minor, but acceptable, differences of coating thicknesses.

Figure 13 shows the same re-calibration of the probe for temperature where within experimental uncertainty (data are shown in Table 1) the average slope (averaged over 7 repeated cycles) is 10.5 pm/ºC and 9 pm/ºC, which compares favorably with the original calibration of the probe after fabrication and before insertion into the aggressive environment of 10 pm/ºC.

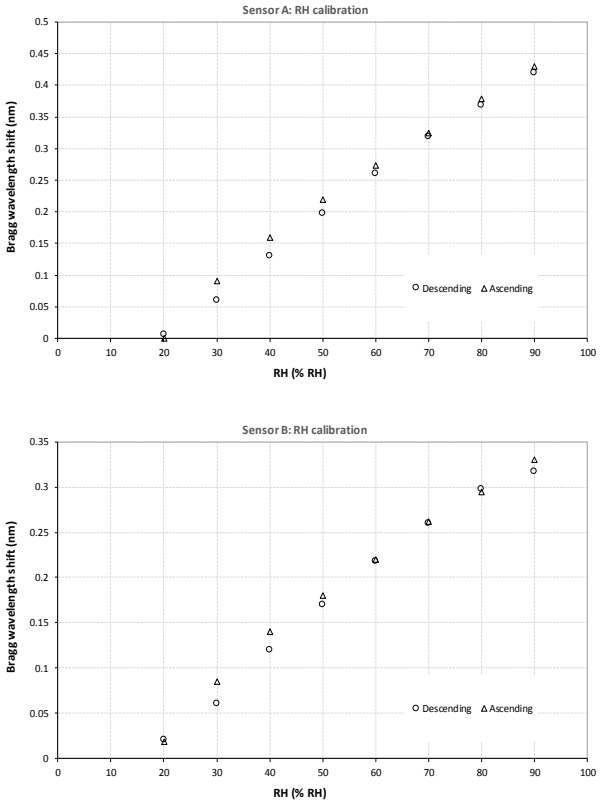

Fig. 12. RH recalibration results (average over 7 repeated cycles) on the two types of probes to both ascending and then descending levels of RH.

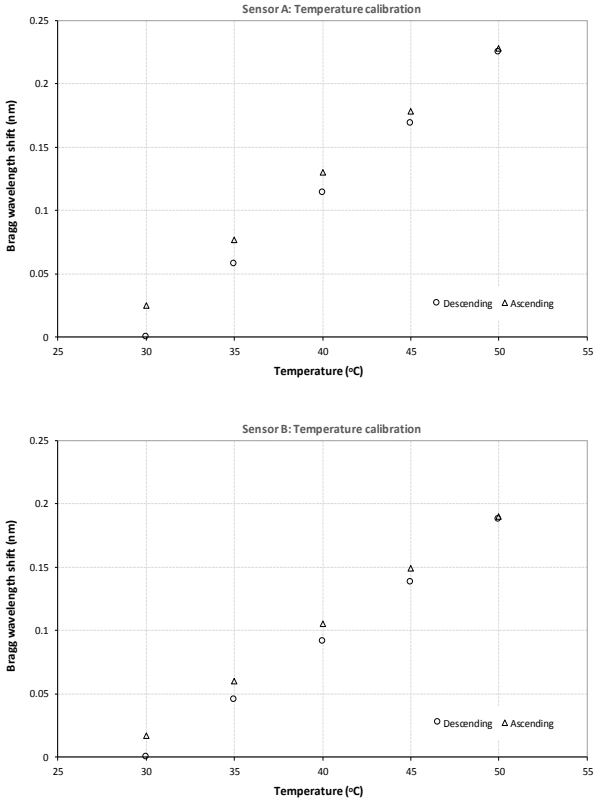

Fig. 13. Temperature calibration on the two types of probes to both ascending and then descending levels of temperature.

Sensor type and material	Ascending/ Descending	Sensitivity Prior to exposure (with ± uncertainty)	Sensitivity after exposure (with ± uncertainty)
(Sensor A) Epoxy design	Asc. RH	4.4 ± 0.2 pm/%RH	4.3 ± 0.2 pm/%RH
	Desc. RH	4.5 ± 0.2 pm/%RH	4.4 ± 0.2 pm/%RH
	Asc. T	9.5 ± 0.5 pm/°C	8.7 ± 0.5 pm/°C
	Desc. T	9.5 ± 0.5 pm/°C	9.4 ± 0.5 pm/°C
(Sensor B) PEEK design	Asc. RH	6.3 pm ± 0.2 /%RH	6 pm ± 0.2 /%RH
	Desc. RH	6.2 pm ± 0.2 /%RH	6 pm ± 0.2 /%RH
	Asc. T	10 ± 0.5 pm/°C	10.1 ± 0.5 pm/°C
	Desc. T	10 ± 0.5 pm/°C	11.2 ± 0.5 pm/°C

Table 1. Temperature and RH calibration results of the two types of probes.

IV. Discussion and Conclusion

The results obtained have shown clearly that the fiber optic sensor system designed and developed for use in the very high humidity, aggressive sewer environment have performed satisfactorily and highly reliably over a significant test period. This contrasts with the performance of conventional electronic humidity sensors which were evaluated at the same time and failed within days under the same exposure. The sensor systems showed a rapid dynamic range at the outset of the series of tests and this was reproduced after months of exposure to the aggressive environment, when the sensors were recalibrated after a 5 month period. This showed the consistent performance over the period of the tests in spite of the effect of the aggressive environment on the surface of the probes, especially evident in the Sensor A type design. This recalibration showed that the humidity sensitivity remained essentially unchanged (within the uncertainty of the experiment) at ~4–6 pm/%RH (depending on the design and fabrication parameters), with the temperature sensitivity again being consistent at ~9–11 pm/°C (again depending on the design and fabrication parameters). Inspection of the sensors themselves (illustrated by the photographs in Fig. 9 A-D) showed the extent to which they suffered some fouling due to the aggressive environment (more evident for Sensor A), but this had no deleterious effect on the performance of the sensors. It should be noted that this study was an experiment conducted in a live environment – not under the controlled conditions of a laboratory. The calibration carried out attempted to represent the measurement in the extreme conditions outside the sewer.

The sensor packaging plays a vital role in the durability of the sensor as the sensing element needs to be effectively protected against harsh environment. Although both the packaging survived, the PEEK packaging used for Sensor B is now recommended for even longer periods of exposure, as the sensor element is completely protected via a Teflon layer and the material seems to show less fouling.

In light of the results obtained from the study, it can be concluded that the use of optical fiber sensor systems for the target application of humidity and temperature measurement in a highly aggressive sewer environment can be successfully achieved. All of the qualities observed from the optical fiber sensors are paramount to achieve sensitive, accurate and effective data in order to assess the sewer corrosion condition, allow for better maintenance and reduce cost to both operator and consumer as well as be readily configurable in an optical fiber sensor network.

The next stage of the research work will focus on the physical durability of the sensor system: thus it will be important to analyze the material properties of the specific PI grade used under the influence of H_2S and see whether this could be improved by implementing the material or using another material. The work showed the stability of the PI coating over the period – it will be valuable to expose this to even longer term testing to see if there are effects not noticed in the tests carried out thus far. Work is also ongoing to tackle the small level of hysteresis seen in the final results and examine the cause in light of the above.

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