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Development of FBG humidity sensors for stone condition monitoring

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***Abstract* – Novel relative humidity (RH) sensors, based on fibre Bragg gratings (FBGs) technology, have been fabricated using different moisture-sensitive materials, namely agar, poly(vinylalcohol) and poly(ethyleneoxide), on the fiber sensor.The sensor is specifically intended to monitor stones of cultural heritage buildings and monuments, in particular for being positionedunder hydrophobic coatings used for protecting stones from absorption of water and dissolved chemicals, thus providing early warning of coating deterioration by RH monitoring. The feature of the prototypes was first tested exposing the sensors in controlled environment and then assessed by simulated 'in-the-field' operating conditions, with sensors installed in a marble block. Experimental results are presented and discussed.**

I. INTRODUCTION

In the preservation of cultural heritage, the use of protective coatings for the consolidation and protection of artworks with historical and artistic value is widely accepted. In the case of stone artworks, consolidant properties and water repellence are the most important requirements of a protective coating. Unfortunately the protective coatings do not always have chemical and photochemical stability, and can undergo deterioration, losing in this way their hydrophobic protective properties [1]. It is important to detect this deterioration for a well-timed intervention. The monitoring of moisture in stones under the protective coating can be an efficient method for early detection of coating deterioration.

The most commonly used techniques for measuring moisture in building materials are gravimetric, electrical and mechanical and chilled-mirror hygrometric methods [2]. Recently, sensors based on fiber optic technology have gained interest in the measure of this parameter [3]:they offer specific advantages such as small size and low weight, are less invasive, are resistant to environment adverse condition and do not require power supply at the sensing point.

A wide range of fiber optic sensors for RH monitoring have been reported in the literature, including techniques based on in-fibergratings, evanescent wave techniques, interferometric methods, hybrid approaches and absorptionmethods [4]. In particular, the in-fiber grating sensors represent a class ofintrinsic fiber optic sensors, that are sensitive to environmental parameters such as temperature and strain. Depending on thegrating structure of the sensing element, in-fiber grating sensors can be classified in FBG and Long Period Grating (LPG).

FBG-based humidity sensors work on the basis of a hygroscopic material layered on the optical fiber. FBG sensors intrinsically act as strain gauges, providing a signal proportional to the experienced deformation. The hygroscopic material layered on the FBG modifies its swelling according to humidity, which in turn induces a related deformation of the FBG thus allowing RH monitoring.Several coatingshave been investigated, such as silica/di-ureasil, polyimide, polyvinyl alcohol,and Pyralin [4 and references therein].

This work is aimed to provide a novel solution to achievea direct measurement of the moisture content in stones through thedevelopment and evaluation of a minimally-invasive opticalfibre humidity sensor.Thus,Agar, poly(vinylalcohol) (PVA) and poly(ethyleneoxide) (PEO) were tested to fabricate prototype sensorsbased on coated FBGs.

II. WORKING PRINCIPLE AND FABRICATION OF THE SENSORS

A. Workingprinciple

The proposed sensor exploits thestrain effect induced in a FBG through the swelling of a thinlayer of applied polymer coating. The swelling of the polymer coating, arising from the absorption of moisture,affects the FBG signal, which can becalibrated to give a direct indication of the humidity level.

Regarding the FBG sensors, they can be seen as a notch filter which reflects back a narrow-bandlight spectrum centered around a peak wavelength, named the Bragg wavelength (λ_B), that satisfies theBragg condition [5]. The value of $\lambda_B$can be expressed as:

$$\lambda_B = 2 \cdot \Lambda \cdot \eta_{eff} \quad (1)$$

where Λ is the spatial period of the grating, and $\eta_{eff}$is the effective refractive index of the fiber opticat the grating location.As a function of the change in the strain or temperature, the Bragg wavelength shift can be expressed as

$$\frac{\Delta\lambda_B}{\lambda_B} = (1 - P_e)\,\varepsilon + [(1 - P_e)\,\alpha + \xi]\,\Delta T \qquad (2)$$

where *Pe*represents the photo-elastic constant of the fiber, ε is the longitudinal strain, and ξ is the fiberthermo-optical coefficient. The two terms of (2)represent the longitudinal strain effect on the FBGand the thermal effect due to the change in temperature, respectively.

B. Fabrication of the sensors

There are a number of possible polymeric coating materials, but suitable ones must satisfy a number of criteria. Firstly it should be hygroscopic and should have anappreciable swelling with respect to RH. Another important requirement is that the preparation of the coating solution and the coating process should be simple. The material should also possess a good adhesion to fiber and finally the coating material should exhibit good long term stability.

Agar, PVA and PEO meet these requirements and were selected as a suitable material with appropriate RH sensitivity. PVA and PEO can readily absorb and desorb water; moreover a fast equilibrium with atmospheric moisture can be established. They are able to form gels containing as little as 2-3wt% polymer in water. Agar is a complex mixture of polysaccharides, it is soluble in hot water and the solution forms a gel on cooling to about 35° -45° C that does not melt below 85°C.

To fabricate the humidity sensor, a steel mold with dimensions 3×3×80[mm]with a hairline notch in the middle was designed to coat the FBG with selected polymers. Commercial FBGs (Broptics Technology Inc., Taipei, Taiwan; 1 cm length of sensing area) were fixed straight and horizontally in the middle of the notch and then coated with polymer solution. In the case of PVA and PEO, the powder was mixed with deionized water to form a 5 wt%polymer solution. The agar solution was prepared by dissolving 1 wt % agar in distilled water at 100°C with help of a heater combined with a magnetic stirrer. The mixture has to be deposited on the optical fiber when the temperature of the solution is above the gelling point, that is, when the mixture has not gellified yet, and is still in liquid form. In all cases, the coated fibres were kept in the mold for one day at room temperature until the gelwaspartially dehydrated and reached equilibrium with the ambient room humidity.

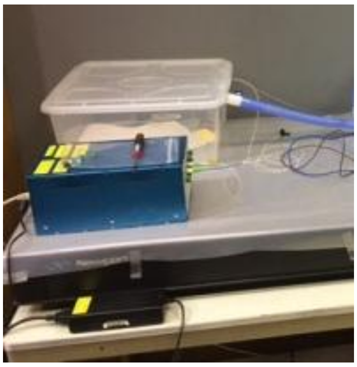

Fig. 1. Experimental setup for testing prototype sensors exposed to controlled RH environment.

III. EXPERIMENTAL SETUP

Two experimental setup were used, to test the prototype sensors under two conditions: *i*) exposed to the ambient within a RH controlled enclosure, to evaluate the relationship between the output of the FBG (λ_B) and RH (Fig.1); *ii*) installed on stone, to simulate expected 'in-the-field' operating conditions (Fig. 2).

In both conditions a reference FBG acting as a pure temperature sensor (insensitive to RH) was used to accomplish temperature compensation.

The output ofthe RH sensors and the output of the reference FBG are collectedby a fiber Bragg grating interrogator system (FS22, HBM Fiber Sensing, Portugal; wavelengthmeasurement range: from 1500 nm to 1600 nm, resolution= 1.0 pm) at a frequency of 1 Hz.A capacitive humidity sensor (EasyLog, EL-USB_2, USB interface, resolution 0.5%RH) is used to provide the RH reference value.

Allprototypesensors were tested in the controlled RH enclosurethat is conditioned by a RH controlled pressurized airflow. Setup is shown in Fig.1.

The experimental set-up is able to test the sensor in a wide range of RH: indeed, it allows dryingthe air within the enclosure up to RH= 5%–10% or humidifying the air up to RH= 100%.

The output of the reference FBG sensor is used with a twofold aim: to monitorthe temperature during the experiments, and to compensate the output changes of the three RHsensors due to temperature changes.

Agar sensor prototype resulted the most promising one for the intended application, which is providing alarm in case of protection coating deterioration, by monitoring of moisture in stones underneath the coating itself.The sensor was thus tested by simulating expected 'in-the-field' operating conditions, using a type of stones quite common in roman archaeological building and artworks, namely marble. Setup is shown in Fig. 2.

Two grooves were dug on the upper surface of a marble block. One of the grooves and the nearby stone upper surface was coated byRhodorsilRC80 (RhodiaSilicones, Italy), a commercial polyethysiloxanewith marked hydrophobic properties, largely used for conservation of stone monument.

Fig. 2. The layout of the sensors in a marble block to simulate "in-field" condition.

Two Agar sensor prototypes were prepared and placed each in one groove, in close contact with the inner stone surface (Fig. 2). The sensor prototype inside the non coated groove is representative of a heavily deteriorated protective coating. Then the stone was subjected to a "capillary rise" test: the bottom surface, in dry condition at environment equilibrium, of the block was put on a soaked pad and the moisture changes at the grooves on the upper surface was monitored. After the stone block was fully saturated with moisture, the stone went back to a dry condition at environment equilibrium.

IV. RESULTS AND DISCUSSIONS

Before testing the prototype sensors in the two experimental setup, their spectra were acquired and compared before and after the coating deposition. That was intended to check that no appreciable distortion of the spectra had occurred, mainly with respect to the typical narrow-peaked bell-shaped feature which allows automatic detection of λ_B. This explorative analysis has been performed at two different values of RH (about 15% and about 45%) and at an almost constant temperature (about 20 °C). Spectra did not show any appreciable distortion at both RH values. Moreover, this explorative analysis also allowed to roughly verify that effective adhesion between the optical fibre and the coating had occurred. In fact the spectra of the coated sensors shifted toward the longerwavelength with RH increase as expected after swelling of the coating material.

A. Measuring sensitivity of the sensors

Experiments with setup shown in Fig. 1 were carried out to assess the response of the PVA, PEO and Agar coated FBG sensors to a wide range of RH values. The sensors under investigation were placed in a controlled RH enclosureand a first set of measurements was performed by applying cycles, in which the RH was increased and decreased from an almost dry condition to a saturated one.Indeed, each cycle consist of two phases: a first phase with ascending RH levels, starting from dry air (RH < 10%) to condition of air with high content of water vapour (RH>90%); the second phase with descending RH levels, starting from the end of the first phase (RH>90%) to almost dry air (RH < 10%). The duration of both the increasing and the decreasing phases was varied and each cycles were repeated in order to determine the response velocity of the sensors and the reproducibility of the measurements.

The best behaviorwas recorded for the Agar-coated sensor, for RH range and for sensitivity, besides it is able to follow the changes of RH as fast as the reference sensor or faster. The Agar sensor prototype is able to follow RH changes in a wide range of measurement, from ~4% to ~98% andthe maximum $\Delta\lambda B$ is about 1.2 nm. This sensor followed the RH changes, so its mean sensitivity can beestimated as the ratio between the $\Delta\lambda B$ recorded in correspondence to the two extreme RH valuesapplied during the test and the difference between these two RH values. The sensitivity is about 0.15nm/%and the degree ofhysteresis is acceptable,*i.e.*about 10%.

PVA- and PEO- sensor prototypes showed a narrower range of operation, because they behaves as humidity sensors only for high value of RH, *i.e.* from 50% to 90%. Anyway they have a reversible, fast and repeatable response to humidity, together with a good sensitivity in this range of RH.

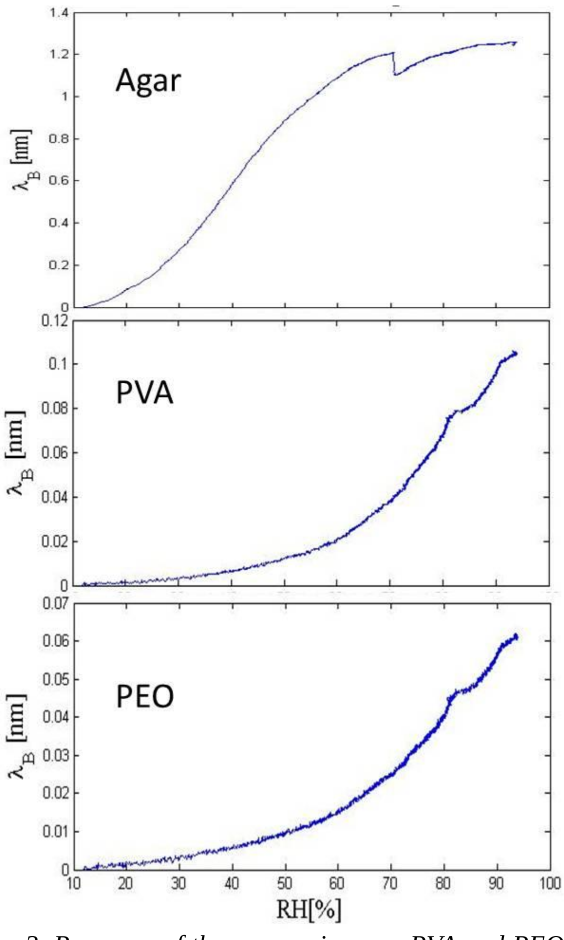

Fig. 3. Response of the sensors in agar, PVA and PEO to RH changes.

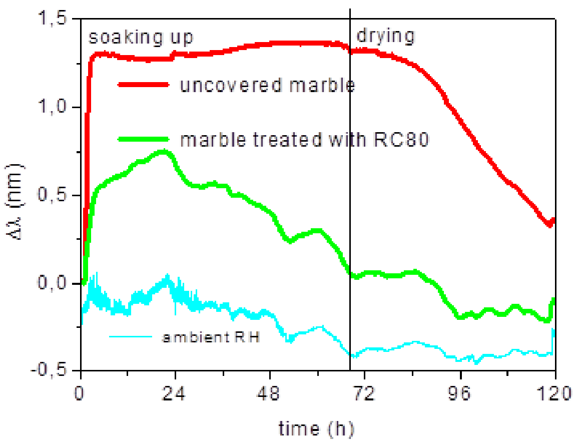

Fig. 4. Response of the Agar sensors placed on the marble block together with reference sensor during capillary rise test.

B. Testing "in-the-field" application

The Agar sensor prototype showed the best performance as RH sensor, so we decided to test its capability to monitor the moisture in the stone and at the same time the capability to distinguish between nude and protected stone.

The figure show the response of the two sensors embedded in the stone, when they were subjected to capillary rise test.

First of all we can note that the RH values, as measured by Agar-coated sensors in the "nude" grooves, increased as soon as the stones were immersed in water, while decrease gradually with time whilst the stones were drying, showing a clear correlation between the response of this humidity sensor and the hydration and drying process of the stones. This is a clear evidence that the measurement made using the opticalfibre humidity sensor allows the movement of thewater vapour in this porous medium to be monitoredaccurately. In fact the conclusion drawn that the drying is a very slow process. This arises from the fact that the major watertransport process is governed by diffusion. Thus, even afterthe drying process was carried out over 3 days, the RH levelinside the stone block continued to decrease very slowly.

From the data analysis, it comes out that the response of the sensors placed in groove "protected" by RC80 are different. In particular there is no steep slope in correspondence with the immersion of the stones in water and in general the sensors followed the variation of ambient RH, as registered by commercial RH sensor. This suggest that the surfaces of the grooves protected by RC80 are in some way "isolated" from the stones and the agar sensor can distinguish these different behaviours.

V. CONCLUSIONS

In this work we investigate a number of possible hygroscopic coating materials with the intent to assess their use for monitoring stone humidity with the aim of Cultural Heritage conservation. Agar, PVA and PEOwere chosen to fabricate prototype sensors. They offer a series of advantages compared with other materials whose use is reported in literature: a wide operating humidity range with a simple coating procedure; few impurities and no ethical concerns; high melting points, stability and low material degradation. They are soluble in hot water, so the preparation and coating procedure are easy. Moreover they have a good adhesion to fibre and easily form a thin coating film on fiber.

Measurements intended at evaluating if the FBG-based RH sensor is able to follow RH changes in a wide range of value were carried out. All the sensors are able to follow the change of RH as fast as a commercial electronic RH sensor used as a reference. However agar sensor prototype is able to follow RH changes in a wide range of measurement (from ~4% to ~98%) and with a resolution better than 1%, while PVA- and PEO- sensor prototypes showed a similar sensitivity (about 1%) but only for high value of RH (from 50% to 100%).

Furthermore, the Agar sensor prototype was tested for its capability for the intended use, which is providing alarm in case of protection coating deterioration, by monitoring of moisture in stones. Results clearly show that our sensors allows to monitor the movement of the water vapor inside the stones. Furthermore we have noticed differences in the response of the sensors placed in groove "protected" by RC80. These are encouraged outcomes for detection of coating deterioration by using FBG RH sensors.

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