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► **To cite this version:**

Gaia Maria Berruti, Marco Consales, Anna Borriello, Michele Giordano, Salvatore Buontempo, et al.. Radiation Tolerant Fiber Optic Humidity Sensors for High Energy Physics Applications. EWSHM - 7th European Workshop on Structural Health Monitoring, IFFSTTAR, Inria, Université de Nantes, Jul 2014, Nantes, France. hal-01021040

**HAL Id: hal-01021040**

**<https://inria.hal.science/hal-01021040>**

Submitted on 9 Jul 2014

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## RADIATION TOLERANT FIBER OPTIC HUMIDITY SENSORS FOR HIGH ENERGY PHYSICS APPLICATIONS

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### ABSTRACT

In this contribution we present investigations developed in the last years by our multidisciplinary research group concerning the possibility to use fiber grating based sensors for relative humidity monitoring in the Compact Muon Solenoid (CMS) experiment at CERN, in Geneva. In particular our research, firstly focused on the development of relative humidity fiber Bragg grating (FBG) sensors coated with micrometer thin polyimide overlays, has been recently extended to long period grating (LPG) sensors, coated with a finely tuned titanium dioxide (TiO<sub>2</sub>) thin layer (~100 nm thick). Experimental tests in the range [0-75] %RH and at different temperatures were carried out to assess the FBG and LPG humidity sensors performances in real operative conditions required in experiments running at CERN. Progressive irradiation campaigns with  $\gamma$  ionizing radiations were also performed. Obtained results demonstrate the strong potentialities of the two proposed technologies in light of their future exploitation as robust and valid alternative to currently used commercial hygrometers in High Energy Physics (HEP) applications.

**KEYWORDS:** *Fiber Bragg Grating, Long Period Grating, CERN, Relative Humidity, High Energy Physics*

### INTRODUCTION

Relative humidity (RH) monitoring has a significant impact in various application fields and many sensing schemes and solutions have been proposed, according to the specific applications. Here we present the use of fiber optic sensors (FOS) in High Energy Physics (HEP) environments, as the Compact Muon Solenoid (CMS), one of the detectors of the accelerator running at the European Organization for Nuclear Research (CERN) in Geneva. The detector is a multi-layered cylinder, 25 meters long and 15 meters in diameter, weighing more than 13000 tons, built around a large solenoid generating a 4 Tesla uniform magnetic field. Its innermost layer is a silicon-based particle tracker where silicon pixel and micro-strip sensors measure momentum and position of the particles emerging from the collisions. In order to reduce the radiation induced loss of performance of the silicon sensors, the CMS tracker must operate under the condition that the warmest point on the surface of each silicon sensor is at a temperature below -10 °C. For this reason, all tracker sub-detectors are cooled by circulating fluids between -20 °C and -25 °C. To avoid condensation of water and the growth of ice, which can inflict major damage at such low temperature, the whole tracking volume needs to be kept dry. In particular a distributed thermal and hygrometric monitoring of the air has mostly concern the external area of the tracker. Indeed, the inner part of the volume has been hermetically sealed during assembly in laboratory and dry conditions of the air

are guaranteed. On the contrary, the surrounding area is not in a perfectly sealed environment. In particular the humidity level has to be controlled by blowing in large quantities of dry gas to force out the water vapor. In addition, the thermal insulation of the nearby coolant pipes, which are also at low temperature because of the circulating coolant, is not always optimal due to space and geometry constraints. For all these reasons, temperature and humidity monitoring is crucial, in order to prevent the possible damage of the detector and its expensive service infrastructures. In addition, each tracking detector needs to be as much as possible transparent to particles; the high-luminosity upgrade of the accelerator will extend the expected integrated luminosity to the new target of 3000 fb<sup>-1</sup>. Such increase in luminosity translates directly to a corresponding increase in the radiation dose that the detectors components will have to withstand. All those needs and constraints together translate to requirements for environmental sensors in terms of radiation tolerance from 10 kGray up to 1 MGray, miniaturized dimensions, minimal mass, minimal need of services and calibration unaffected by magnetic field. Almost all miniaturized humidity sensors available in the market are capacitive and they require multi-wiring, which makes the remote powering and read-out undesirably complex and space consuming. Furthermore, the electrical sensors suffer from inherent limitations as they are not designed with radiation tolerance characteristics and only a few ones exhibit a level of accuracy below  $\pm 3$  %RH [1]. On the contrary, fiber optic sensors (FOS) provide many attractive features, which overcome the limitations presented by the conventional instruments used at the present. Indeed, modern fabrication technologies allow obtaining fibers tolerating high radiation levels [2]. This aspect, together with the undeniable advantages of fiber optic technology in terms of reduced size and weight, multiparametric sensing, immunity to electromagnetic interference and water and corrosion resistance, has promoted renewed interest in the application of FOS in high radiation environments. Numerous FOS have been proposed for humidity detection over the years. An extensive review concerning the use of FOS for humidity monitoring has been reported by Yeo et al [3]. In 2011, our research group has been involved in the development of new generation RH-FOS sensors in collaboration with CERN. The research was firstly focused on the development of radiation tolerant relative humidity fiber Bragg grating (FBG) sensors coated with micrometer thin polyimide overlays [4]. Collected results provided a clear proof that this innovative technology is a robust alternative to currently used commercial hygrometers. For this reason, this platform has been selected for hygrometric control of the air in critical areas of CMS. In the meanwhile, the characterization of a second generation of optical sensors based on high-sensitivity TiO<sub>2</sub>-coated long period grating (LPG) sensors for RH measurements in high radiation applications has been launched within the same collaboration framework. In this paper we present results obtained during the experimental campaigns carried out in the CERN laboratories, showing the strong potentialities of both FBG and LPG technologies in light of their use in HEP environments.

## 1 SENSING PRINCIPLE OF FIBER GRATING BASED SENSORS FOR HUMIDITY MEASUREMENTS

Fiber gratings consist of a periodic perturbation of the refractive index of the core of the optical fiber and fall in two general classification based on the period of the grating (typically a sub-micron period for FBGs and from 100 $\mu$ m to 1mm for LPGs).

In the next sections we explain how it is possible to use both the two kinds of gratings as relative humidity sensors.

### 1.1 Fiber Bragg Grating as humidity sensor

A FBG behaves as a wavelength selective filter which reflects light signals at a specific wavelength, named the Bragg wavelength ( $\lambda_B$ ), which depends on the fiber effective refractive index ( $n_{\text{eff}}$ ) and the grating pitch ( $\Lambda$ ):

$$\lambda_B = 2n_{\text{eff}}\Lambda \quad (1)$$

Both  $n_{\text{eff}}$  and  $\Lambda$  can be affected by strain and temperature and, consequently, the Bragg wavelength shift ( $\Delta\lambda_B$ ) due to change in strain ( $\epsilon$ ) and thermal effect ( $\Delta T$ ) can be expressed as:

$$\Delta\lambda_B/\lambda_B = (1 - P_\epsilon)\epsilon + [(1 - P_\epsilon)\alpha + \zeta]\Delta T \quad (2)$$

where  $P_e$  is the photo elastic-constant, and  $\alpha$  and  $\zeta$  are the thermal-expansion and thermo-optic coefficients, respectively.

Bare silica fibers are insensitive to humidity. However, it is possible to develop FBG humidity sensors by coating the grating with a hygroscopic material that swells in presence of water molecules adsorption, thus inducing mechanical strain to the grating [5, 6].

In presence of  $\Delta RH$  and  $\Delta T$ , in linear assumption,  $\Delta\lambda_B$  is expressed as:

$$\Delta\lambda_B = S_{RH}\Delta RH + S_T\Delta T \quad (3)$$

where  $S_{RH}$  and  $S_T$  are the relative humidity and temperature sensitivities of the sensor, respectively. The typical value of  $S_T$  is 10 pm/°C while previous studies demonstrated that the  $S_{RH}$  for a 10  $\mu\text{m}$  polyimide coated FBG is at best about 1.0÷1.5 pm/%RH [4]. A very precise temperature compensation scheme is required to decouple the cross-sensitivity to temperature in order to avoid large errors induced on the RH reading (e.g. a temperature reading error of  $\pm 1$  °C corresponds to an error of 7÷10 %RH in the humidity reading if no compensation is applied). The solution proposed to CMS and currently used in the experiment is represented by an optical thermo-hygrometer made of two coupled FBGs, one polyimide coated and another bare, for RH and T readings, respectively.

## 1.2 Long period grating as humidity sensor

LPGs are photonic devices allowing the power transfer from the fundamental guided core mode to a discrete number of forward propagating cladding modes and to each of them at a distinct wavelength where the so-called phase matching condition is satisfied:

$$\lambda_{res,0i} = (n_{eff,co} - n_{eff,cl}^{0i})\Lambda \quad (4)$$

where  $n_{eff,co}$  and  $n_{eff,cl}^{0i}$  are the core and  $i^{\text{th}}$  cladding mode effective indices respectively, and  $\Lambda$  is the grating period. As a result of this process (referred to as mode coupling) the LPG transmission spectrum shows several attenuation bands or dips related to the different excited optical fields: the cladding modes. A portion of the electromagnetic field of the cladding modes penetrates into the surrounding medium in the form of evanescent wave, thus making  $n_{eff,cl}^{0i}$  sensitive to the chemical and physical properties of the surrounding environment. The surrounding refractive index (SRI) sensitivity affects the spectral features of LPG, which, for this reason, is used in chemical and biological sensing applications through the integration of functional overlays [7,8]. Moreover, the SRI sensitivity can be optimized for a specific application, by acting on the thickness and optical properties of the functional overlay [9]. Several kinds of materials have been explored as coating for the development of LPG-based RH sensors, including polymers [9] [10], hydrogels [11], gelatine [12], cobalt chloride based materials [13] and SiO<sub>2</sub> nanosphere [14]. In all these cases, the water absorption/desorption in the hygroscopic coating in presence of RH variations modify its RI (and thickness), thus creating a spectral variation and amplitude change in the LPG attenuation bands, independently from the adhesion properties of the coating onto the grating itself. However, in literature there is no evidence of the characterization of coated LPGs at low humidity values (below 20 %RH) and temperatures below 15 °C. Moreover, while the radiations effects have been investigated in case of bare LPGs up to 1.54 MGy [15,16], their influence on the sensing performance of coated LPG seems to be completely unexplored. We proposed for the first time to the best of our knowledge the use of titanium dioxide (TiO<sub>2</sub>) as coating material, due to its high refractive index ( $n=1.96$ ) [17] and hygroscopic characteristics [18,19]. Furthermore, the use of oxide layers is expected to avoid the well-known aging problems, typical of polymeric materials.

## 2 EXPERIMENTAL SET-UP

The sensor performances of FBG and LPG based RH sensors have been investigated in [0-75] % humidity range at different temperatures. In addition, the effects induced by the sensors' exposure to very high  $\gamma$  - ionizing radiation doses have been also studied (post irradiation stage).

Fig. 1 shows the set-up used for the experimental tests of the fiber optic grating sensors in the PH-DT (Physics Department-Detector Technology Section) laboratory at CERN. The facility for experimental tests is provided with a thermo-regulation circuit for temperature and a manual control

of humidity levels. A 1 pm wavelength resolution optical interrogator with a measurement range from 1510 nm to 1590 nm, was provided for the acquisition of the FOS readings. A high performance Chilled Mirror Dew Point Hygrometer was used to measure the reference relative humidity. Four resistance thermometers (PT-100) were installed close to the optical sensors to guarantee the homogeneity and stability conditions of the temperature in the chamber.

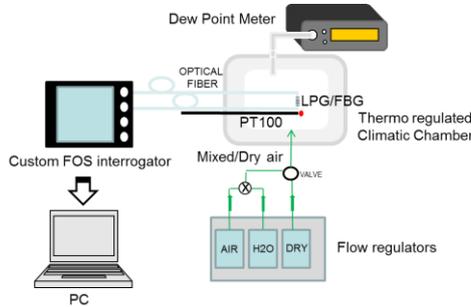


Figure 1: Experimental set-up used for the tests of the FOS sensors.

### 3 EXPERIMENTAL RESULTS

In the next two sections we separately describe the procedure applied for the FBG and LPG sensors' characterization, before and after their exposure to high  $\gamma$  radiation doses.

#### 3.1 Characterization of FBG sensors for relative humidity monitoring

As to the temperature sensors, commercial FBGs (Micron Optics os4300) were selected. The sensors were fully characterized in the range  $[-20, 20]$  °C. In fig 2 (a) an example of T reconstruction for 8 samples is shown. Despite the different dynamics of the FOS and the reference sensors, residuals are inside  $\pm 0.15$  °C in steady states, as shown in fig. 2 (b).

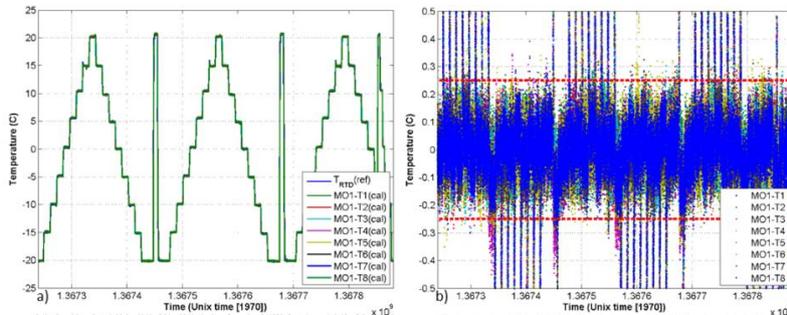


Figure 2: T reconstruction from 8 FBG sensors (a);  $T_{FOS} - T_{ref}$  (b).

The FBG sensors selected for the RH measurements were produced under specification by an external company with a polyimide coating of  $10 \mu\text{m}$  ( $\pm 2 \mu\text{m}$ ) [4]. RH tests were performed in the range  $[0 - 60]$  % at 4 different temperatures (20, 10, 0, -5 °C), as shown in fig. 3 (a), and  $S_{RH}$  and  $S_T$  were evaluated in order to find the linear model (equation 3).

Slight dependences of  $S_{RH}$  on T and of  $S_T$  on RH were observed ( $S_{RH} = 1.41 \pm 0.07 \text{ pm}/\%RH$ ;  $S_T = 11.40 \pm 0.11 \text{ pm}/^\circ\text{C}$ ), as shown in fig. 3 (b, c). A higher order method was introduced to model the above mentioned cross-sensitivity dependencies and RH was expressed as

$$RH(\lambda, T) = p_{00} + p_{10}\lambda + p_{01}T + p_{20}\lambda^2 + p_{11}\lambda T + p_{02}T^2 \quad (5)$$

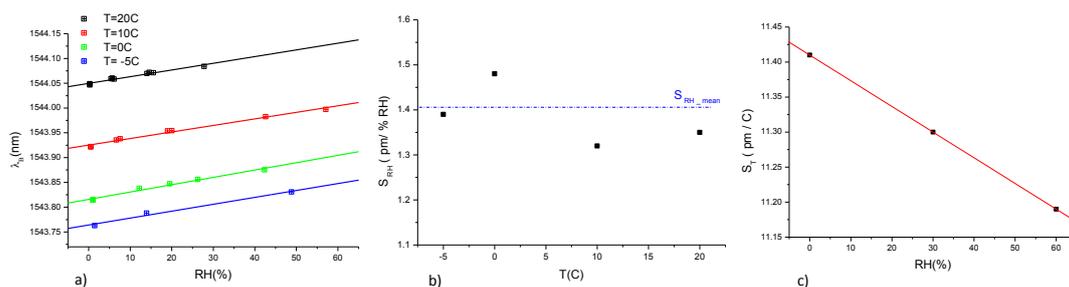


Figure 3: Calibration points at different T (a);  $S_{RH}$  versus T (b);  $S_T$  versus RH (c).

where  $p_{00}..p_{02}$  are the surface parameters. Fig. 4 shows performances of 4 FOS thermo-hygrometers using this model.

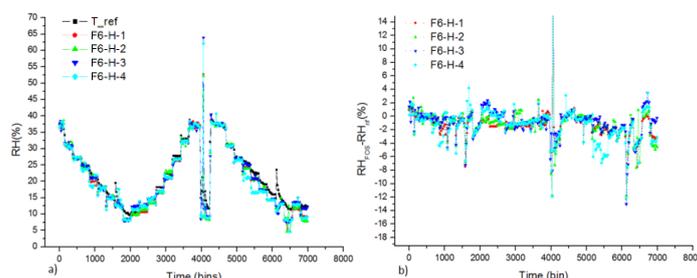
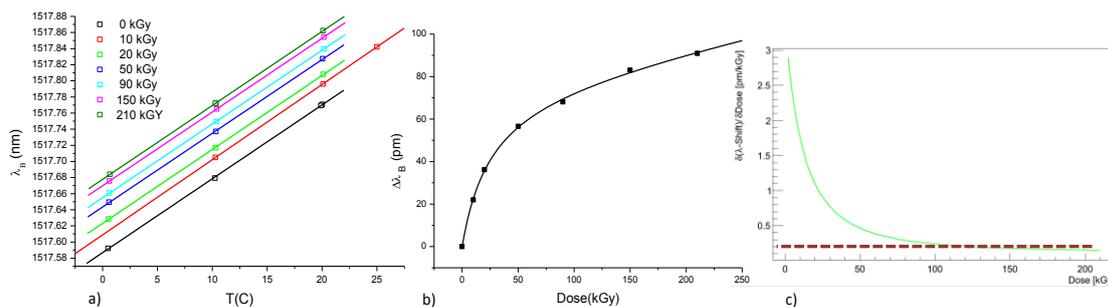


Figure 4: RH reconstruction from 4 FBG thermo-hygrometers (a)  $RH_{FOS} - RH_{ref}$  ( $\pm 2\%$  in steady states) (b).

The radiation tolerance of the FBG thermo-hygrometers was also investigated. In particular incremental irradiation campaigns were performed on samples of RH and T FBG sensors in order to understand the effect of  $\gamma$ -ionizing radiation on the sensing performances.

In Fig. 5, results from one sample of the FOS T sensor up to 210 kGy  $\gamma$ -radiation dose are summarized. A radiation-induced red shift was observed after each irradiation step. The  $S_T$  value, evaluated as the slope of the calibration curve at each dose, was found to be unchanged all over the six campaigns. The wavelength shift is not saturated up to 210 kGy, however, above 100 kGy total adsorbed dose the shift near linearly increases with it, as shown in Fig. 5 (b).



Fig

Figure 5:  $\Delta\lambda_B$  versus T at different doses (a);  $\Delta\lambda_B$  versus Dose (b);  $S_{\gamma-rad}$  versus Dose (c).

Fig. 5 (c) shows that the sensitivity of the sensor to radiation ( $S_{\gamma-rad}$ ) is very low ( $\approx 0.15$  pm/kGy) after 150 kGy. The result suggests that the FOS T sensors can be pre-irradiated with 200 kGy total adsorbed dose in order to achieve high precision T measurement once installed in HEP environments. Similar investigations have been performed on FBG-RH sensors. In particular 4 samples have been irradiated up to 210 kGy and results after each campaign demonstrated that the effect of ionizing radiation is negligible on  $S_{RH}$  (below the measurement error). In Fig. 6, results from one sample of FOS RH sensor exposed to radiation at 20 °C are summarized. Similarly to the

T sensors, the RH-FBGs also show radiation induced wavelength shift. In this case, a pre-irradiation step helps to decrease the sensitivity to further radiation.

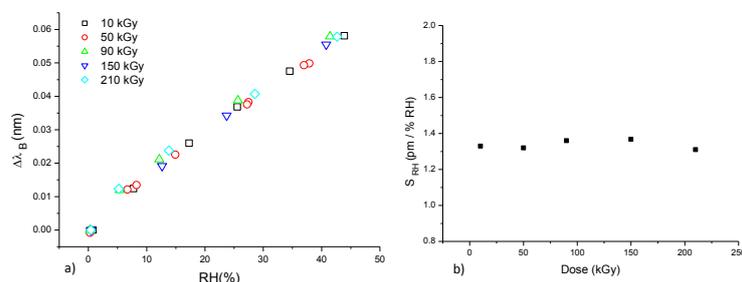


Figure 6:  $\Delta\lambda_B$  as a function of RH at 20 °C (a); invariance of  $S_{RH}$  (b) at each irradiation step up to 210 kGy.

### 3.2 Characterization of LPG sensors for relative humidity monitoring

The same characterizations, performed on the FBG thermo-hygrometers and discussed in the previous sections, were applied to TiO<sub>2</sub> coated LPG sensors. In particular several samples with different TiO<sub>2</sub> thicknesses were successfully produced, using sol-gel dip coating method. Data reported here refer to the sample named LPG1 ( $\Lambda=404 \mu\text{m}$ ). Fig. 7 (a) reports a 20x optical microscope image of the sample after the deposition. The estimated TiO<sub>2</sub> thickness is ~100 nm. Fig. 7 (b) shows the transmittance spectra, before and after the deposition. The bare device exhibits an attenuation band (related to the 5th cladding mode) centred at  $\lambda_{res,05}=1589.0 \text{ nm}$ , while the deposition of the TiO<sub>2</sub> layer causes a resonance blue shift of ~24.1 together with a ~5 dB increase in its depth.

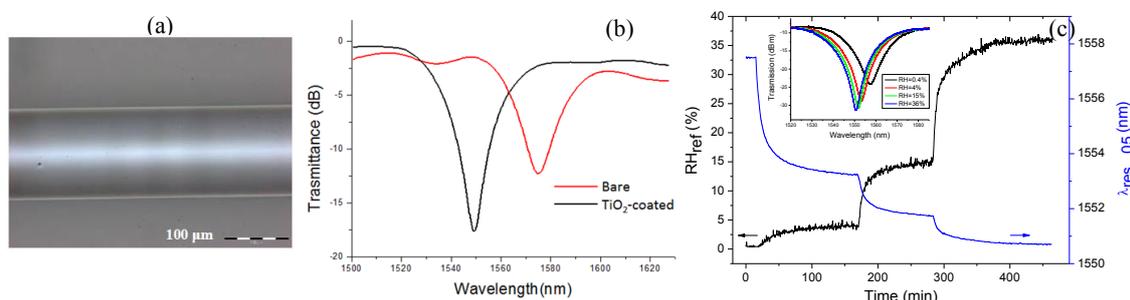


Figure 7: Microscope image of a TiO<sub>2</sub>-coated LPG (a); transmitted spectra of bare and coated LPG (b); LPG response during a RH test at 25 °C (c); Transmittance spectra of LPG1 at different RH values (Inset).

The LPG1's performances have been investigated in [0-75] %RH and between -10 °C and 25 °C. Fig. 7 (c) reports the  $\lambda_{res,05}$  variations during a test at 25 °C. The response provided by the optical sensor is in very good agreement with the reference device readings (black curve). As theoretically expected, increasing the humidity content inside the test chamber causes a blue shift of the  $\lambda_{res,05}$ . This is due to the TiO<sub>2</sub> coating RI increase promoted by the higher amount of adsorbed water molecules, which in turn leads to an increase of  $n_{eff,co}$  [18]. The inset in Fig. 7 (c) also reveals that, by increasing the RH value, the attenuation band visibility increases. This is an important aspect as, in most cases, LPGs coated with "lossy" overlays experience partial or full resonance fading when operating in transition mode [20]. Similar results have also been obtained at 10, 0 and -10 °C. Fig. 8 (a) shows the calibration curves of LPG1 at the four temperatures during the pre-irradiation stage. In contrast with what observed with poly-coated FBGs [4], they have found to be non-linear, as a larger  $\lambda_{res,05}$  shift was registered at low humidity. This can also be observed in Fig. 8 (b), where the  $S_{RH}$  curves versus RH are plotted. At 25 °C,  $S_{RH}$  values are between 1.4 and 0.11 nm/%RH in the range 0-10 % and decrease down to 0.01 nm/%RH at high humidity. Obtained results evidence that

TiO<sub>2</sub>-coated LPGs are able to provide  $S_{RH}$  values from one to three orders of magnitude higher than those exhibited by micrometer thin polyimide coated FBGs.

From Fig. 8 (b), a slight dependence of  $S_{RH}$  on  $T$  can be appreciated as a small decrease occurs when  $T$  decreases. In addition, starting from the curves reported in Fig. 8 (a), working at constant RH, the temperature sensitivity was retrieved. As a result, the  $S_T$  slightly decreases with RH; however a mean  $S_T$  of  $\sim 0,250 \pm 0.015$  nm/°C was evaluated in the full RH range. As observed for FBG RH sensors, temperature compensation is required also in this case to avoid errors induced on the RH reading due to the cross sensitivity to  $T$ . However, in the critical operative range [0-10] %RH, assuming a  $S_{RHmean}$  of  $\sim 0.5$  nm/%RH, a  $T$  change of  $\pm 1$  °C would only induce a  $0.5 \div 1$  %RH error if any compensation is applied (against the  $7 \div 10$  %RH in case of FBG RH sensors).

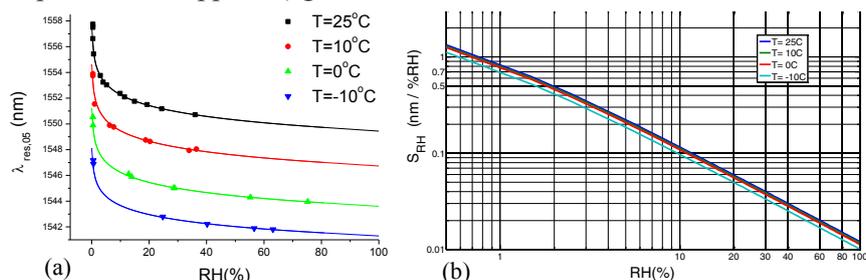


Figure 8: Characteristic curves of LPG1 at the four investigated  $T$  (a);  $S_{RH}$  as a function of RH (b).

The radiation tolerance of LPG1 has also been investigated by exposing it to a 10 kGy ionizing radiation dose. Preliminary results evidence a radiation induced dip wavelength shift of about 4.4 nm in correspondence of an RH value of 30 %. Even if further irradiation campaigns at progressively higher doses are currently in progress to confirm these preliminary results, we point out that the observed shift is almost in agreement with radiation hardness investigations conducted on different types of bare LPGs [21]. Results are shown in Fig. 9, where the  $\Delta\lambda_{res,05}$  versus RH is depicted at 25°C, before (black curve) and after (red curve) the irradiation dose.

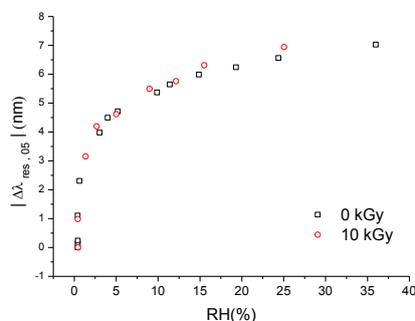


Figure 9:  $\Delta\lambda_{res,05}$  before and after the 10 kGy exposure for sample LPG1.

Interestingly, the LPG sensor does not lose its amazing capability to respond to RH changes and, apart some variations (of about 16 %), its characteristic curve still exhibits the same behavior shown at 0 kGy.

## CONCLUSION

In this paper we presented our research activity, developed in collaboration with CERN of Geneva, concerning the use of fiber optic grating (both coated FBGs and LPGs) based sensors for relative humidity monitoring in high energy physics environments. In particular, the RH detection performances of polyimide coated FBG sensors and of TiO<sub>2</sub>-coated LPG sensors were investigated in the range [0-75] % at different temperatures, as well as after high radiation exposures. Collected results give a clear demonstration that this innovative technology is a robust and valid alternative to

polymer-based electronic hygrometers currently used in high energy physics environments as the CMS experiment at CERN.

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