27th International Conference on Optical Fiber Sensors © OSA 2020

Humidity Effect on Acrylate- and Polyimide-Coated Fibres for Distributed Sensing Applications

Tiago Neves*[ab], **Regina Magalhães**[c], **Lorenzo Scherino**[ad], **Sonia Martin-Lopez**[c],
Hugo F. Martins[e], **Paolo Petagna**[a], **Luc Thévenaz** [b]

[a]*Experimental Department, Detector Technology Group, CERN - European Organization for Nuclear Research, CH-1211 Geneva, Switzerland*
[b]*EPFL - Swiss Federal Institute of Technology, Group for Fibre Optics, SCI-STI- LT Station 11, 1015 Lausanne, Switzerland*
[c]*Dpto. de Electrónica, Universidad de Alcalá, 28805, Alcalá de Henares (Madrid), Spain*
[d]*University of Sannio, Department of Engineering, Optoelectronics Group, I-82100 Benevento, Italy*
[e]*Instituto de Óptica, Consejo Superior de Investigaciones Científicas, 28006 Madrid, Spain*
**tiago.neves@cern.ch*

Abstract:
A study of the effect of humidity on acrylate- and polyimide-coated fibres is analysed over a large range of humidity and temperature (-20 °C to +50 °C) using phase-sensitive Rayleigh reflectometry.

1. Introduction

Nowadays, fibre optic sensors (FOS) are increasingly gaining the attention of researchers and companies due to their insensitivity to electromagnetic interference, small dimensions, reliable readings across long distances and ease of multiplexing to form large network of sensors. Most of the successful FOS applications focus on temperature and strain monitoring as well as acoustic and humidity sensing [1,2]. FOS technology can be divided in two different categories. The first is made of point FOS, such as Fibre Bragg Gratings (FBG) and Long Period Gratings (LPG), while the second is based on Distributed Fibre Optic Sensors (DFOS), in which the whole fibre actually makes a continuous and linear sensor. 3 main different types of DFOS can be devised, based on the exploited back-scattering effect: Rayleigh, Brillouin and Raman and, crosswise to all of them, the backscattered signal inside the material is directly dependent on temperature and stress applied on the fibre. Relative humidity (RH) is the other fundamental environmental parameter influencing the signal in the fibre beside temperature: the fibre coating expand/contract when absorbing/desorbing water vapour, transmitting stress to the fibre. While this physical effect looks quite obvious at first glance, only few authors studied the effect of humidity on optical fibres. In 2008 Galindez et al. [3] actually showed that the temperature measurement in a distributed Brillouin sensor can be significantly affected by RH and this factor should not be considered negligible. However, in most state-of-the-art studies, the effect of humidity variation on temperature measurements is completely overlooked.

Taking advantage of this effect, some distributed RH sensors have been very recently developed for different applications and using different techniques, such as Optical Frequency Domain Reflectometry (OFDR) [4,5] and Optical Time Domain Reflectometry (OTDR) [6]. The OFDR technique provides higher spatial resolution, of the order of few centimetres, but a sensing distance range limited to few tens of metres, while the spatial resolution of an OTDR is slightly larger but with a sensing distance range that can be extended to several kilometres. In the previous studies, the referenced authors made a comparison between acrylate- and polyimide-coated fibre in the RH range from 10% to 90% at a temperature range from 10 °C to 50 °C.

In this work, a Chirped-Pulse Phase-Sensitive OTDR (CP-ϕOTDR) interrogator [7] is used to study the RH effect in air at different temperatures (-20 °C to 50 °C) on different acrylate- and polyimide-coated silica fibres. The RH range - from 0% to 40% at 25 °C - naturally expands for decreasing temperatures, covering the range from 0% to 70% at -20 °C. The temperature and humidity studies were repeated afterwards in a saturated Carbon Dioxide (CO_2) environment, in order to understand the differences in the fibre RH absorption properties under different environments. All the results presented in this study are temperature compensated using 40 metres of coating-stripped fibre and applying a corrective factor to adjust each fibre to their own temperature sensitivity.

2. Sensing Principles

The ϕ-OTDR interrogation technique measures the noise-like but static Rayleigh backscattering signal caused by the frozen and random longitudinal entropic fluctuations of the refractive index (RI) alongside the optical fibre. A deterministic spectral shift of these random signal variations is caused by the stress induced by the deformed coating in presence of water molecules. The humidity causes an expansion of the coating, stressing the fibre and, if the fibre is totally free of static mechanical strain, the total backscattered signal is only function of the temperature and humidity shift. In a traditional ϕ-OTDR, the Rayleigh intensity traces are repeatedly acquired using different optical frequencies and the scanning range determines the maximum detectable temperature or strain change. On the other hand, if the probe pulse has a linear frequency variation along the pulse width, the system is called CP-ϕOTDR, which is, nowadays, a powerful and a highly sensitive refractive index sensor for distributed acoustic sensing (DAS) [8].

3. Experimental Setup

The CP-ϕOTDR interrogator used for this experiment is described in detail in [9]. In our implementation, we used 60 ns pulses (corresponding to a spatial resolution of 6 metres), linearly chirped with ≈700 MHz of total pulse spectral content. For the acquisition, a 2 kHz pulse repetition rate was implemented with an averaging of 40, resulting in an effective sampling rate of 50 Hz. The detection scheme enabled a sensitivity below 1 MHz, being the minimum measurable perturbation limited in practice by cross-sensitivities to other perturbations (1 MHz, equivalent to ≈1 mK temperature shift). Using this configurations, we monitored the fibre under test (FUT), which was 1.2 kilometres long and made of: two pieces of 500 and 300 metres of acrylate-coated (AC1) fibres from Manufacturer1 drawn in the 1990's; 300 metres of acrylate-coated (AC2) fibre from Manufacturer2 drawn in the 2000's; 25 metres of recent polyimide-coated (PI) fibre; 40 metres of bare (i.e. with stripped coating) silica fibre. The FUT was subject to a temperature test at constant RH and several RH tests at different constant temperatures. The tests were performed in a climatic chamber, especially designed at CERN (European Organization for Nuclear Research), showing an excellent control of low levels of humidity and equipped with several temperature, relative humidity and dew point reference sensors [10]. The first set of tests was performed with normal compressed dry air, while the second was carried out with CO_2.

4. Results

4.1. *Compressed Air*

The experimental procedure started with a temperature characterization from -20 °C to 25 °C at constant RH (≈32%) and the results from all fibres are grouped in Fig. 1 ("a" and "b" represent the slope coefficient and zero intercept of the best linear fit, respectively). All fibres are coiled in a stress-free packaging placed in a uniform environment chamber and each shown point represents the value averaged over the entire fibre length.

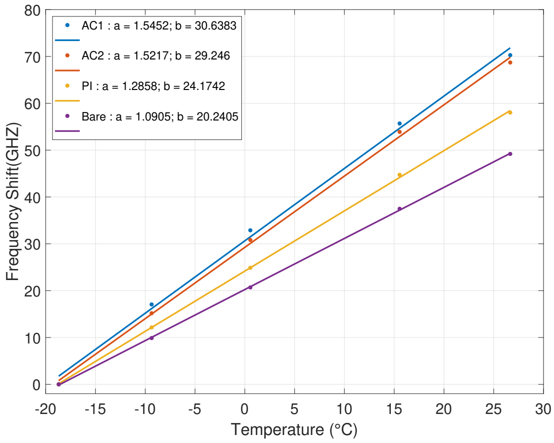

Fig. 1. Temperature calibration fittings of all fibres.

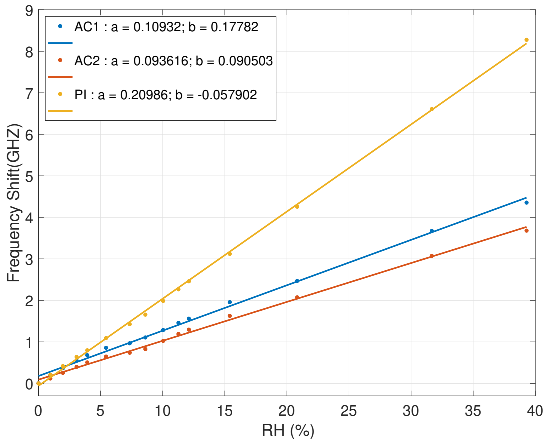

Fig. 2. RH calibration fittings of all fibres.

Analysing the results, one can conclude that the temperature sensitivity (S_T) of both acrylate-coated fibres is constant to a very good approximation in the observed temperature range and nearly the same, of 1.545 and 1.522 GHz/°C respectively, but it is slightly higher than the polyimide-coated fibre showing 1.289 GHz/°C. The bare fibre shows the lowest temperature sensitivity of 1.091 GHz/°C. These differences are related to the thermal expansion coefficient of the specific coatings and their thickness. The acrylate-coated fibres have a coating diameter of ≈60

μm, while the polyimide coating is thinner down to ≈15 μm. After the temperature calibration at constant RH, a RH test at six preset constant temperatures (-20 °C, -10 °C, 0 °C, 15 °C, 25 °C and 50 °C) was carried out. Fig. 2 illustrates the RH sensitivities (S_{RH}) of each fibre at 25 °C. As previously shown in [6], the polyimide-coated fibre presents a higher sensitivity to RH than the acrylate-coated fibres and, as observed for the temperature sensitivities, the differences are again due to the nature and thickness of the coating. The polyimide coating, even being thinner, shows a higher RH sensitivity due to its higher hygroscopic capacity.

Analysing Fig. 3, where the temporal response of each fibre at 25 °C is represented, we can conclude that all tested fibres follow perfectly the RH variation inside the climatic chamber (calibrated reference in the inset plot), but showing different sensitivities and different response times. As expected, the higher is the magnitude of RH variation, the longer is the stabilization time. However, the more sensitive the fibre is, the longer is the stabilization time.

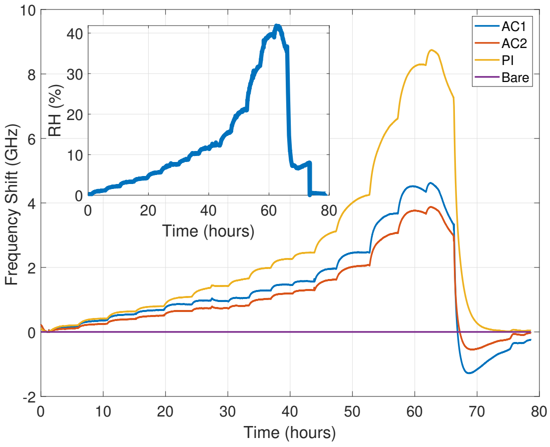

Fig. 3. Fibres frequency shift over a RH test.

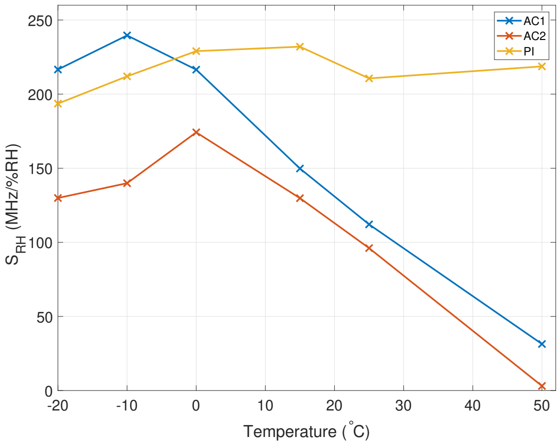

Fig. 4. RH Sensitivity vs Temperature.

Table 1. RH sensitivities comparison in (MHz/%RH).

Temperature	AC1	AC2	PI
-20°C	216.6	129.9	193.6
-10°C	239.6	139.9	212.0
0°C	218.9	177.8	230.0
15°C	147.6	127.7	229.5
25°C	109.3	93.6	209.9
50°C	31.4	–	218.6

Polyimide polymers are known for their stability over a large range of temperatures [11] and this is confirmed by the sensitivity data presented in Fig. 4 and Table 1, which summarises the RH sensitivities at different temperatures. The maximum variation in the RH sensitivity over this range of temperatures is ≈15% for the polyimide-coated fibres, while the acrylate-coated fibres show a completely different behaviour. The first point to raise is the drastic temperature dependence of RH sensitivity for acrylate-coated fibres. Considering for instance AC1 from 25 °C to -20 °C, the response varies by a factor 2 and reaches its maximum sensitivity at -10 °C. At 50 °C, the RH sensitivity is strongly decreased with a value ≈13% of the RH sensitivity at -10 °C. At -10 °C, its sensitivity is even higher than the maximum sensitivity of a polyimide-coated fibre. AC2 also shows a similar behavior to AC1 but the maximum is reached at 0 °C. At 50 °C, the RH sensitivity of this fibre is insignificant and impossible to calibrate due to its nonlinear response. The interesting feature of acrylate-coated fibres is that the difference between two distinct commercial fibres is significant. For some given temperatures, for instance at -20 °C, the AC1 shows a RH sensitivity 1.67 times higher than the AC2, which means that the RH sensitivities should be addressed individually and can not be considered similar to all standard acrylate-coated fibres.

4.2. Carbon Dioxide

A temperature test from -20 °C to 25 °C and then a RH test from 0% to 50% at 25 °C were performed under pure CO_2 atmosphere and comparative results with air are shown in Table 2. As expected, the temperature sensitivity of the bare fibre is not affected in a significant way from air to CO_2 due to the absence of coating, in contrast with the coated fibres. The temperature sensitivity changes is significantly lowered by ≈16%, ≈11% and ≈10% for AC1, AC2 and PI, respectively. Regarding the RH sensitivities of the acrylate-coated fibres, the decrease is ≈6%, ≈10% for AC1 and AC2 respectively, while the polyimide-coated fibre shows an even higher loss of sensitivity of

≈30%. These differences are substantial and we can conclude that the CO_2 concentration in the environment also impacts the fibre sensitivity.

Table 2. Comparison between Air and CO_2.

Sensitivity	AC1	AC2	PI	Bare
S_T Air (GHz/°C)	1.545	1.522	1.289	1.091
S_T CO_2 (GHz/°C)	1.302	1.356	1.167	1.116
S_{RH} Air (MHz/%RH)	109.3	93.6	209.9	–
S_{RH} CO_2 (MHz/%RH)	102.9	84.7	144.8	–

5. Conclusions

An important conclusion of this study is the demonstration that the acrylate-coated fibres show drastic differences in their response in presence of humidity at different temperatures. The RH sensitivities of the acrylate-coated fibres are not negligible and surprisingly, they increase significantly at lower temperatures and vanish almost entirely at high temperatures (50 °C), probably as a result of a drastic change of plasticity. Even if fibres are prepared using the same type of acrylate in their coating composition, the RH sensitivities may be entirely different and should be individually evaluated. RH should be carefully controlled when doing temperature or strain measurements using acrylate-coated commercial fibres, because, at certain temperatures, a small variation of 10% of RH can induce an error of ≈1.5 °C. This value can be even higher in the case of the polyimide-coated fibres due to their higher RH sensitivity. On the other hand, a single correction factor for RH can be used at all temperatures for the polyimide-coated fibres. This also indicates that a polyimide coating seems to be best suited for fibres to be used for humidity sensing. Another factor that affects both temperature and RH sensitivities is the surrounding atmosphere. The test carried out using pure CO_2 actually demonstrated that, depending on the composition of the surrounding atmosphere, the different equilibrium of water absorption in the coating can impact the response in a non-negligible way.

Acknowledgements: This research was funded in part by: the European Commission (FINESSE, MSCA-ITN-ETN-722509); Ministerio de Ciencia, Innovación y Universidades (IJCI-2017-33856, RTI2018-097957-B-C31, RTI2018-097957-B-C33); Comunidad de Madrid and FEDER program (SINFOTON2-CM: P2018/NMT-4326).

References

1. Alan D Kersey. A review of recent developments in fiber optic sensor technology. *Optical fiber technology*, 2(3):291–317, 1996.
2. L Alwis, T Sun, and KTV Grattan. Optical fibre-based sensor technology for humidity and moisture measurement: Review of recent progress. *Measurement*, 46(10):4052–4074, 2013.
3. Carlos A Galindez, Francisco J Madruga, M Lomer, A Cobo, and Jose M Lopez-Higuera. Effect of humidity on optical fiber distributed sensor based on brillouin scattering. In *19th International Conference on Optical Fibre Sensors*, volume 7004, page 70044W. International Society for Optics and Photonics, 2008.
4. Peter J Thomas and Jon O Hellevang. A fully distributed fibre optic sensor for relative humidity measurements. *Sensors and Actuators B: Chemical*, 247:284–289, 2017.
5. Pavol Stajanca, Konstantin Hicke, and Katerina Krebber. Distributed fiberoptic sensor for simultaneous humidity and temperature monitoring based on polyimide-coated optical fibers. *Sensors*, 19(23):5279, 2019.
6. Tiago FP Neves, Li Zhang, Fan Yang, Kenny H Tow, Paolo Petagna, and Luc Thévenaz. A kilometre-range distributed relative humidity sensor. In *Seventh European Workshop on Optical Fibre Sensors*, volume 11199, page 1119922. International Society for Optics and Photonics, 2019.
7. Juan Pastor-Graells, HF Martins, Andrés Garcia-Ruiz, Sonia Martin-Lopez, and Miguel Gonzalez-Herraez. Single-shot distributed temperature and strain tracking using direct detection phase-sensitive otdr with chirped pulses. *Optics express*, 24(12):13121–13133, 2016.
8. María R Fernández-Ruiz, Luis Costa, and Hugo F Martins. Distributed acoustic sensing using chirped-pulse phase-sensitive otdr technology. *Sensors*, 19(20):4368, 2019.
9. Regina Magalhães, João Pereira, Andrés Garcia-Ruiz, Walter Margulis, Sonia Martin-Lopez, Miguel Gonzalez-Herraez, and Hugo F Martins. Distributed detection of quadratic kerr effect in silica fibers using chirped-pulse φotdr. In *Seventh European Workshop on Optical Fibre Sensors*, volume 11199, page 1119929. International Society for Optics and Photonics, 2019.
10. G Berruti, M Consales, M Giordano, L Sansone, P Petagna, S Buontempo, G Breglio, and A Cusano. Radiation hard humidity sensors for high energy physics applications using polyimide-coated fiber bragg gratings sensors. *Sensors and Actuators B: Chemical*, 177:94–102, 2013.
11. John A Kreuz and James R Edman. Polyimide films. *Advanced Materials*, 10(15):1229–1232, 1998.