

Full length article

Thermal treatment impact on self-written waveguide Fabry-Perot sensors for temperature and humidity detection

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ABSTRACT

UV curable photopolymers enable rapid fabrication of micro-optical structures that support highly integrated compact platforms. Light-induced polymerization can realize the formation of self-written waveguides, offering a scalable in-situ fabrication of functional microstructures. The thermal curing process of photopolymerizable materials can, however, have a significant impact on the mechanical and structural properties of the material. In this work, several self-written waveguides based on a UV-curable polymer are fabricated on the tip of single mode fibers by applying different laser powers. In a reflection scheme, a Fabry-Perot cavity arises from this structure, which is monitored for temperature and relative humidity variations. A study of the influence of two types of thermal curing is conducted for the sensor's stability as well as its response to the measurands concerning sensitivity, hysteresis, and resolution. Continuous high temperature curing allows for greater stability of the spectrum and lower hysteresis to temperature while slow curing – annealing – results in different temperature profiles of the sensors depending on the laser power during its fabrication. Conversely, a similar response for all curing laser powers is achieved to relative humidity after each curing treatment.

1. Introduction

In several applications, it is desired to achieve the measurement of several physical and chemical parameters accurately. The use of optical fiber sensors (OFS) comes about as an excellent option due to their flexibility [1]. These devices possess compact size as well as immunity to electromagnetic interference whilst being able to operate in harsh environments [2,3]. Despite these advantages, the functional diversity of silica-based OFS remains a limitation [4]. To overcome this, the interfacing of other materials with the pure silica OFS can potentiate higher sensitivity and offer greater versatility than the conventional silica OFS [1]. As such, lab-on-fiber technology has emerged, integrating functional microstructures directly onto the fiber end-face using various methods [3,4]. One approach is the light-induced self-written or self-growing polymer waveguide, which represents a highly efficient bottom-up fabrication method [4,5]. This process relies on the photopolymerization of a material through the light emanating from a fiber core, leading to a self-focused growth of a 3D structure perfectly aligned with the core [5,6].

This technique has been employed for several applications, including

sensing [1,2], optical coupling [7], beam-shaping [4,8], and imaging [4]. Regarding sensing, micro-tips have been developed which resulted in a Fabry-Perot interferometer (FPI) where the high thermo-optic coefficient of the polymer material was explored for a temperature sensing application [9]. Relative humidity (RH) detection with self-written waveguides (SWW) has also been explored with fast response (~ 0.6 s) and high sensitivity ($0.2 \text{ nm}/\% \text{RH}$) [10].

The curing process is fundamental to defining the optical performance and dimension of the SWW, as its geometry and refractive index (RI) profile are strictly controlled by parameters such as laser power, exposure time, and oxygen inhibition [4,5,8,11]. Empirical models have been developed for the RI evolution during polymerization, for instance, towards insertion losses in coupling applications, allowing for optimization of both step-index and graded-index profiles [6].

The influence of curing and annealing processes on the performance impact of sensors developed by SWWs is, however, unexplored. In this work we develop photopolymeric SWW sensors produced on single-mode fiber (SMF) tips, based on varying laser powers used during the SWW fabrication. A temperature and RH sensor was achieved on a SWW based on the Norland Adhesive (NOA) 68 polymer. Additionally, the

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influence of thermal curing and slow annealing was explored towards the temperature and RH response regarding sensitivity, hysteresis, and resolution, with respect to the laser power used for the device fabrication.

2. Sensor working principle

Photopolymer SWWs are formed through the polymerization of photocurable resins, irradiated by concentrated light from an optical fiber. It starts from the center of the fiber core, leading to an increase of RI in the irradiated region as the material gets polymerized. The RI difference between the exposed core region and the surrounding region leads to a self-focusing effect (also known as self-trapping effect [12]) where the material will continue to polymerize throughout the length of the irradiated region of the structure [5].

The shape of the resulting SWW microtip can be altered by controlling the parameters of the light source that is connected to the illuminating fiber. Power, exposure time, and wavelength all have an impact on the dimensions of the microtip [4]. Note that when the power of the light source is excessively high, a photopolymer cluster can be induced where a large area gets polymerized in a short time [13].

The implementation of a SWW for a sensing application can be realized with an FPI that arises in the cavity defined by the SWW. For this structure, the interfaces between the SMF and SWW and between the SWW and the external medium define the M_1 and M_2 mirrors and the FPI is formed along the axial direction, as shown in Fig. 1.

Considering the SWW as an FP cavity, due to the low reflectivity of the mirrors, this multiple reflection interferometer can be approximated to an only two-wave interference. Thus, the peak wavelength of this FPI is expressed as

$$\lambda_{FPI} = \frac{2L}{m} n_{eff} \quad (1)$$

where m is a positive integer that corresponds to the resonance order, n_{eff} denotes the effective RI of the polymer SWW and L is the length of the SWW. Its free spectral range (FSR) is given by

$$FSR_{FPI} = \frac{\lambda_{m+1} \lambda_m}{2L n_{eff}} \quad (2)$$

To ensure that the fringe contrast of the FP-based SWW is trackable, it is crucial that both the RI of the polymerized material is different from that of fused silica, and that the microtip has well-defined end-faces that denote highly parallel flat mirrors for the FPI. This results in more reflected light from the interface between the fiber and the microtip, revealing higher interferometric visibility [10].

When the sensor is subjected to external variations, such as temperature and RH, a variation of the cavity length, L , and the effective RI, n_{eff} , will be verified, giving rise to a wavelength shift in the spectrum. When temperature increases, an increase of the material dimension and a variation of its RI will be induced due to its thermal expansion and

thermo-optic properties, respectively. By considering a 1D approximation of the structure, the FPI sensitivity to temperature is then given by

$$\frac{\partial \lambda_{FPI}}{\partial T} = \left(\frac{1}{n_{eff}} \frac{\partial n_{eff}}{\partial T} + \frac{1}{L} \frac{\partial L}{\partial T} \right) \lambda_{FPI} \quad (3)$$

where $\frac{1}{L} \frac{\partial L}{\partial T}$ denotes the coefficient of thermal expansion (CTE) and $\frac{1}{n_{eff}} \frac{\partial n_{eff}}{\partial T}$ denotes the thermo-optic coefficient (TOC) of the cured polymer material. Similarly, a variation of RH can also induce a material expansion based on the absorption of water molecules, as well as an RI reduction due to the lower RI of water (1.318 at 25 °C [14]):

$$\frac{\partial \lambda_{FPI}}{\partial RH} = \left(\frac{1}{n_{eff}} \frac{\partial n_{eff}}{\partial RH} + \frac{1}{L} \frac{\partial L}{\partial RH} \right) \lambda_{FPI} \quad (4)$$

To use this technique as a temperature and/or RH sensor it is crucial to study the stability of the sensor, which is impacted by the thermal curing process of the SWW structure. The material used in this work is the photopolymerizable resin NOA68 (Norland Products Inc., US), which cures with ultra-violet (UV) irradiated light, and has a maximum absorption between 350–380 nm, with a transparent color, and showcasing great adhesion to glass. While uncured it has a RI of 1.51 and a viscosity of 5000 cps, while after curing its RI is of 1.54 at 25 °C (for a wavelength of 589 nm) [15].

When using this material as a curable resin, however, it is recommended by the manufacturer to cure the material at 50 °C for 12 h after UV curing [15]. This is due to the process of UV curing where photoinitiators aid in a chain reaction of crosslinking the free radicals in the material with the monomers, leading to a gel state that is not liquid, but also not completely solid. Thereafter, radical mobility drops, resulting in incomplete curing. The manufacturer states that this is the first of two curing steps, and that thermal curing finalizes the cross-linking reaction process [15].

Additionally, the process of annealing is also a thermal treatment process that has a direct impact on the chemical composition of the structure [16]. Given that the aim is to use the SWW as a sensor of physical measurands, it is crucial to study the impact of these thermal processes, particularly for temperature sensing. For this reason, the impact of temperature and RH sensitivity, resolution and spectral stability of FP sensors based on NOA68 SWWs throughout different stages of the curing and annealing was studied.

3. Sensor design, fabrication and spectral analysis

The fabrication of the SWW involved 4 main steps. Firstly, a droplet of the polymer NOA68 material was placed on a glass substrate (Fig. 2a). The SMF tip was positioned close to the droplet in a vertical holder attached to a motorized vertical translation stage (resolution of 10 µm and range of 25 mm), so that the subsequent submersion of the fiber tip in the polymer was slow and controlled. A CMOS Camera from Thorlabs® (Kiralux 5.0 MP Monochrome 35 fps Max) was used to better visualize the region of contact between the fiber and the polymer. Once the fiber was in the desired location in relation to the substrate, a laser diode connected to the fiber was used for the material curing (Fig. 2b). The setup involved a fiber-pigtailed laser with a laser diode connected to a temperature controller. The laser emits light around the 405 nm wavelength, and the laser power was controlled by varying the operating current. The power was measured with a power meter both at the laser pigtailed fiber and at the fiber tip where the SWW was, thereafter, written. The exposure time for the fabrication of the microtip was also studied, where the most successful waveguides were obtained for times as short as 1 s. Since the glass surface was not coated with any material, such as propyl-2-methyl-2-acrylate (TPM) as used in [9], longer exposure times led to sticking of the polymer to the substrate.

Once the laser was turned off, Fig. 2c, the fiber was slowly raised from the droplet, revealing the newly cured polymer structure with the

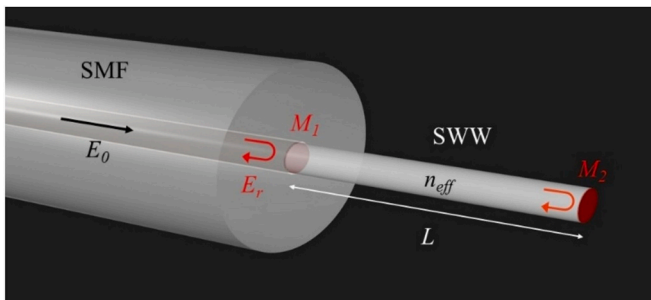


Fig. 1. Schematic illustration of the proposed SWW sensor based on an FP cavity.

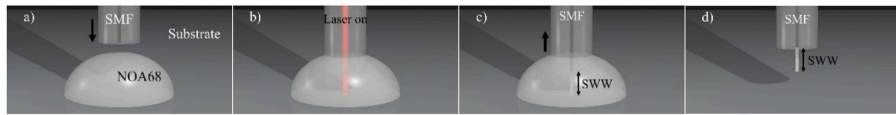


Fig. 2. Schematic of the SWW fabrication process. a) Fiber positioned in the droplet; b) Laser light polymerizes the material; c) Laser light off; d) Resulting SWW after cleaning excess polymer.

same length as the distance between the positioned fiber tip and the substrate when the laser was on. The uncured material surrounding the fiber and the SWW was removed by first quickly dipping (~ 0.2 s) in acetone and then submerging the structure in isopropanol (IPA) (~ 5 s). Acetone is a great option to remove most of the uncured material, however extended exposure to this solvent causes damage to the cured material, possibly corroding, undermining the shape of the SWW, or even breaking it, thus it was only used momentarily. The cleaning of any leftover material was, then, achieved with IPA, a less aggressive solvent for the polymer, and the final structure was obtained, Fig. 2d.

Different samples were fabricated with varying laser powers, whose power curve is shown in Fig. 3, where 3 regions were denoted, each containing two SWWs fabricated using powers within that region. Note that each laser has its unique power curve and Fig. 3 only describes the curve of the laser used in this work.

The micrographs of representative SWWs are shown in Fig. 4, revealing a thin cylinder of the cured material which accrues from the SMF core mode field, that is consistent throughout the entire length of the structure. Note, though, that Fig. 4b reveals some tapering, which is caused by the low laser power. The sensors were fabricated with varying lengths in order to assess the influence of this parameter on the sensitivity of the measurands, considering expressions (3) and (4).

The spectral analysis was performed in a reflection configuration using an optical fiber interrogator (FBG-scan 608 from FOS&S (now FBGS), operating in the C band with a wavelength range of 1525–1575 nm and a resolution of 0.01 nm). The reflection spectra of the sensors produced are provided in Fig. 5, where a longer FP-based SWW reveals a ripple with a narrower FSR than a sensor with a shorter FP-based SWW. The better defined the interfaces are, the higher the visibility of the spectrum. The fabricated sensors have a visibility between 34 % and 90 %, where visibility (V) is defined, within one oscillation period, by $V = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}}$ with $I_{\max}$ and $I_{\min}$ denoting the maximum and minimum linear optical power, respectively. The decrease in the reflected signal amplitude of some FPIs is attributed to cavity losses, either due to beam divergence, material absorption, or poorly defined mirror at the SWW edge [17,18]. Nonetheless, all sensors have clearly trackable peaks and valleys for a wavelength shift, regardless of the laser power used. The highest visibility was attained for longer sensors, therefore the ideal SWW length is in the 100–250 μm region. Beyond this length the decreased FSR would lead to a limited dynamic response range.

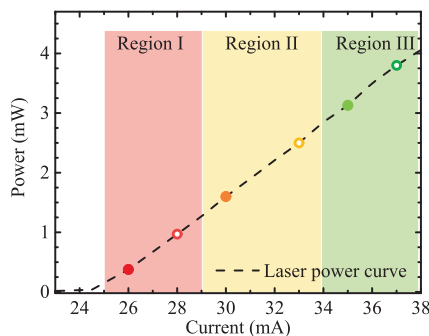


Fig. 3. Laser power curve against current. The dots represent the power at which each SWW was fabricated.

4. Material characterization

A known phenomenon in UV curing of a polymer is the introduction of internal stress. The rapid UV curing can trap polymer chains in disordered states. For this reason, an annealing procedure – which is defined by a slow heating followed by slow cooling process – can be done to help further stabilize the structure. With slow heating, these chains gain enough energy to move, rearrange, and align, resulting in a more stable structure while with gradual cooling, new stress is prevented [19]. The advantages of this thermal process include the increase in structural hardness and mechanical strength while relieving internal stress resultant from the UV curing [20]. There is, though, a temperature threshold above which the annealing treatment causes degradation of the material [21].

For this purpose, this configuration was studied in its composition before and after thermal treatment. Firstly, the samples were thermally cured according to the manufacturer's recommendations at 50 °C for 12 h followed by thermal annealing where temperature was slowly increased from 20 °C to 60 °C over 8 h and immediately after slowly cooled back to 20 °C during another 8 h. In order to comprehend the impact of these thermal processes on the morphology of the SWWs, SEM images were obtained pre- and post-annealing of three samples that were each fabricated in a different laser power region, as shown in Fig. 6.

From the SEM analysis, one can notice geometrical differences between these samples. One curious outcome is the end face of the samples. They all have a very flat shape; however, they are not perfectly circular. The higher the laser power used, the more irregular its edges are (Fig. 6o,r), revealing that the curing of a polymer with this method using an SMF does not occur with a single mode, since this fiber is not single-mode in the UV region. Furthermore, there are geometrical changes pre- and post-annealing, particularly more pronounced for the lower power samples: there is visible roughness both at the surface of the SWW (Fig. 6e) as well as the end face post-annealing (Fig. 6f); there is a curvature for the first sample post-annealing (Fig. 6d); there is also visible shedding accumulating at the fiber-polymer interface that occurred from the thermal treatment. None of this can be observed for the third sample. The length and diameter of the samples also changed from this process. There was a length decrease of 1.47 μm for the first sample (Fig. 6a,d), 0.96 μm for the second (Fig. 6g,j), and 0.19 μm for the third (Fig. 6m,p). On the other hand, there was also a slight shrinkage of the diameter of the samples, particularly at the center region, which shows greater tapering at this region than at the base and end of the structure. This phenomenon is verified in all samples, though, without showing preference for the laser power used. Overall, the greatest shrinkage is evidenced for the lower power samples, while the higher power samples undergo some decreased diameter while retaining the same SWW length.

Given these dimensional changes, particularly since the structure is not perfectly cylindrical throughout its length but rather reveals wrinkles that become shrunk from thermal processes, it is to be expected that the lower power samples experience a greater temperature sensitivity. The lower power used for cure led to an incompletely cured structure, with soft polymer in its composition. After thermal curing, some of the unbounded chains become polymerized, leading to an overall more homogeneous structure. With annealing, the material is further post-cured, which will increase the densification of the material, as visualized in the SEM images with the SWW shrinkage. This phenomenon is

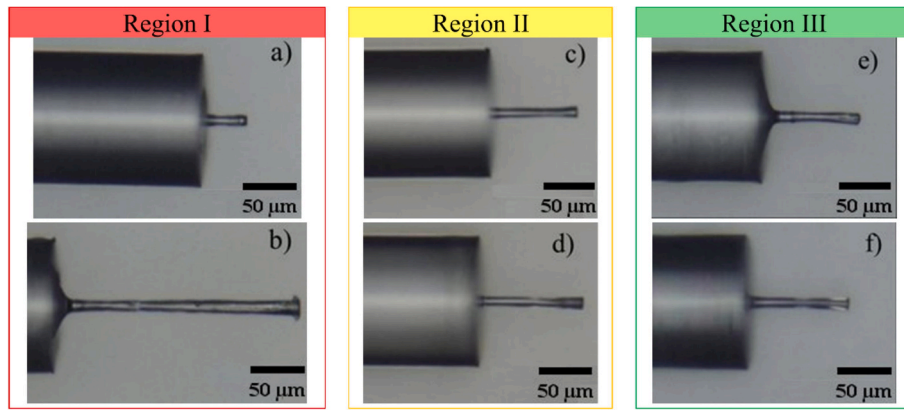


Fig. 4. Microscopic view of the SWWs fabricated on a SMF with a laser power of a) 0.4 mW; b) 1.0 mW; c) 1.6 mW; d) 2.5 mW; e) 3.1 mW; f) 3.8 mW.

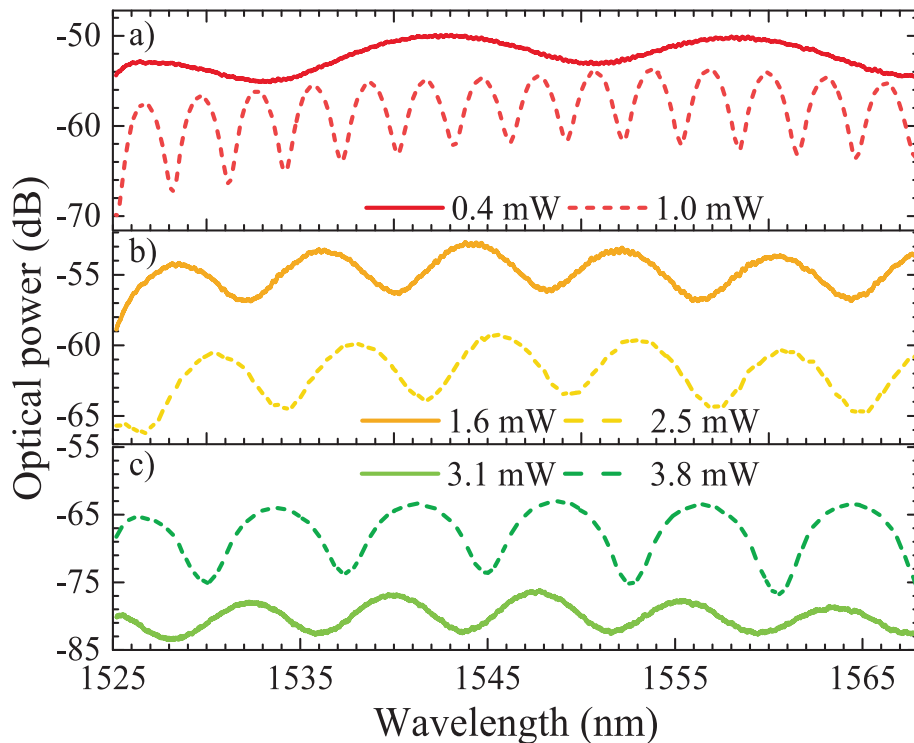


Fig. 5. Reflection spectra of the SWWs produced at different laser power powers for regions a) I, b) II, and c) III with respective lengths of a) 44 μm , 251 μm , b) 90 μm , 103 μm , c) 87 μm , and 96 μm . An offset in power was added for viewing purposes.

also corroborated in literature where annealing allowed to alleviate the internal stress, leading to polymerization shrinkage and attaining a material with an increased hardness [20–22].

The effective RI of the samples was also studied with the combination of the spectra pre- and post-annealing and the calculated lengths from the SEM analysis. Given (2), the RI of the samples was calculated, as shown in Table 1:

One notices the increase of the effective RI post-annealing for all samples, particularly for those fabricated with lower powers, indicating a further cured state from this thermal treatment, considering that the uncured RI was lower than that of the cured material. Since this variation was less noticeable for the higher power sample, it can be concluded that the material has a more advanced cured state from UV curing and that thermal treatments do not change the geometrical and material characteristics of the polymer as much as for the lower power fabricated SWWs. This analysis was also corroborated with the low shrinkage evidenced in the SEM analysis. As such, the material has low residual

curing stress from the initial UV curing step, with a more homogeneous network where most/all polymer chains are bound, thus the stress redistribution and increase of densification seen in the lower power samples was not as present.

Given this, the annealing of lower power samples will lead to a decrease of the CTE of the structure, due to its increased densification and higher hardness. Since, according to literature [23], this material has a negative TOC, it is expected that these samples will attain a decrease in temperature sensitivity post-annealing when compared with the pre-annealing state and when compared with the higher power samples. In contrast, for Region III sensors, one expects minimal temperature sensitivity change to occur given that the material was already mostly fully cured after the initial UV curing, and lower densification is verified for these samples, thus the CTE was expected not to decrease particularly.

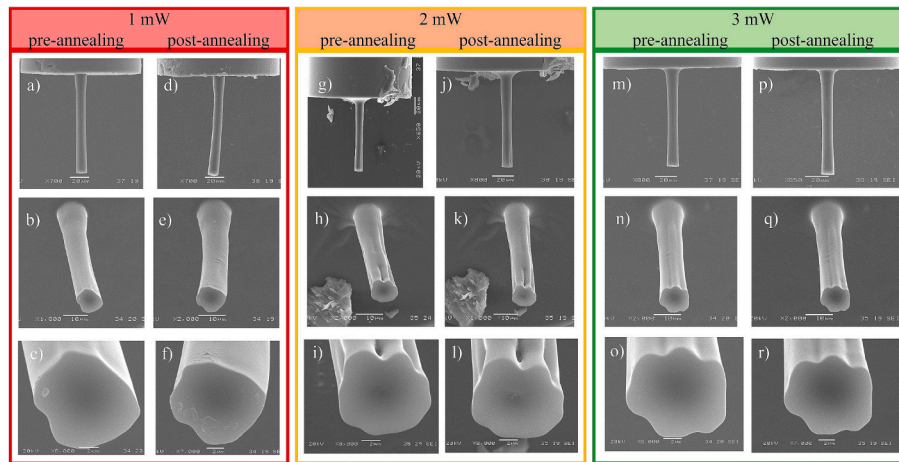


Fig. 6. SEM micrographs of the SWWs with different curing laser powers. Region I – pre-annealing: a) overview, b) top view c) end face; Region I – post-annealing: d)–f). Region II – pre-annealing: g)–i) and post-annealing: j)–l); Region III – pre-annealing: m)–o) and post-annealing: p)–r).

Table 1

Calculated effective RI of the SWWs with different curing powers pre- and post-annealing.

Sample	RI pre-annealing	RI post-annealing
1 mW	1.59	1.61
2 mW	1.55	1.56
3 mW	1.62	1.62

5. Results

Once the SWWs were fabricated, the following order of experiments was applied according to the flow chart shown in Fig. 7.

While, sequentially, both temperature and RH experiments were conducted in parallel following the thermal treatments, for clarity reasons the results for each parameter will be discussed in separate sections.

5.1. Temperature tests

The temperature characterization was conducted in a climatic chamber (CTS CS-40–200) where temperature was increased from 20 to 60 °C and decreased back to 20 °C in steps of 5 °C, waiting 1 h between each step, while relative humidity was kept constant at 40 % RH. The temperature range was chosen based on the limitations of a curable resin, which has a different plasticity for higher temperatures, particularly above 70 °C [15,24].

The post-cure wavelength shifts per step, shown in Fig. 8, where there is still some significant instability where the central wavelengths of certain steps deviate from the linear fit, particularly for the Region III sensors. One can conclude that a 12-hour curing step is insufficient for high-power fabricated SWWs. For the more stable sensors, the sensitivities obtained are between 20 and 40 pm/°C. According to the properties of these materials, while the value of CTE is not specified, according to [24,25] the temperature sensitivity of this material also had a positive response to the measurand. Additionally, according to [23], the experimental value of TOC was calculated to be $\sim -2.5 \times 10^{-4} \text{ K}^{-1}$. Since TOC is negative and the temperature response is positive, the CTE of NOA68 is dominant for this configuration. Considering the attained

sensitivity of 20–40 pm/°C, based on (3), as a first approximation, the experimental CTE is $\sim (2.63\text{--}2.76) \times 10^{-4} \text{ K}^{-1}$. The temperature experiment was then repeated after the annealing step with the responses seen in Fig. 9.

For the SWWs fabricated with a laser power in Region I, a complete flip in temperature response from a shift towards greater wavelengths (red shift) to a non-linear shift to shorter wavelengths (blue shift) was attained post-annealing, with greater wavelength shifts at lower temperatures. For Region II sensors, the response was also non-linear but remained with a red shift. Finally, for Region III sensors, the temperature response was linear with a red shift throughout the entire 20–60 °C temperature range.

While the response was not linear for Regions I and II, with an exponential fit being more suitable, a linear fit was considered in the aforementioned temperature ranges in order to compare linear sensitivity values between the 3 Regions. The average sensitivities and their respective fitting errors denoted as the error bars are represented in Fig. 10 for the Region II and III sensors, pre- and post-annealing.

For the Region I sensors, the sensitivity attained between 20 and 40 °C was $-95.4 \text{ pm}/^\circ\text{C}$ and $-72.7 \text{ pm}/^\circ\text{C}$, and from 40 to 60 °C it was $-10.41 \text{ pm}/^\circ\text{C}$ and $1.27 \text{ pm}/^\circ\text{C}$, for the 0.4 mW and 1.0 mW sensors, respectively. The R^2 of the Region I sensors was much lower post-annealing, decreasing from 0.988 to 0.940. For Region II, there was also a decrease from 0.966 to 0.919, and Region III has the highest value of R^2 post-annealing, of 0.988 and 0.996, respectively, for the 3.1 mW and 3.8 mW sensors. While the sensors have different lengths, it was found that the sensitivity to temperature of this material was not dependent on this parameter.

This three-region laser power temperature response profile is consistent with the analysis performed in section 4. The sensors that endured higher densification post-annealing attained a lower temperature sensitivity due to the decrease CTE. In Region II the sensors underwent some shrinkage but lower than the one for Region I, thus the decrease in CTE was not as pronounced. Finally, Region III sensors has the most consistent and linear response due to the little densification verified from the minimal post-curing during annealing.

In an application where this material is aiming to be used as a temperature sensor, it is ideal to use higher powers (in the Region III range)



Fig. 7. Flow chart of the order of thermal treatments and measurand characterizations.

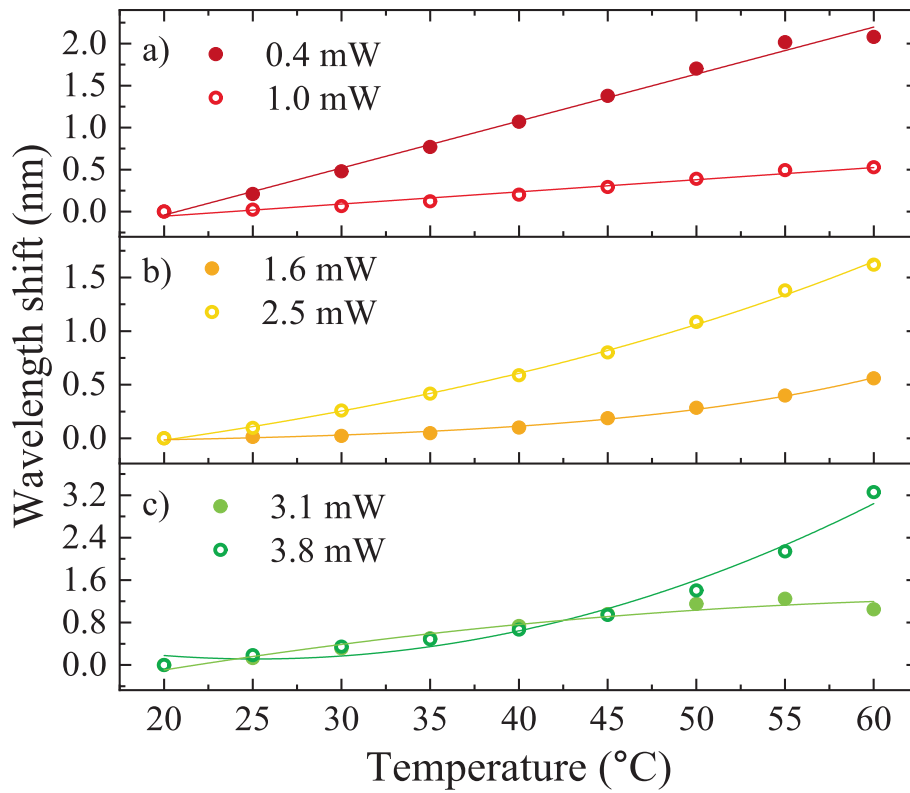


Fig. 8. Temperature characterization after thermal curing of SWWs for the: a) Region I, b) Region II and c) Region III sensors.

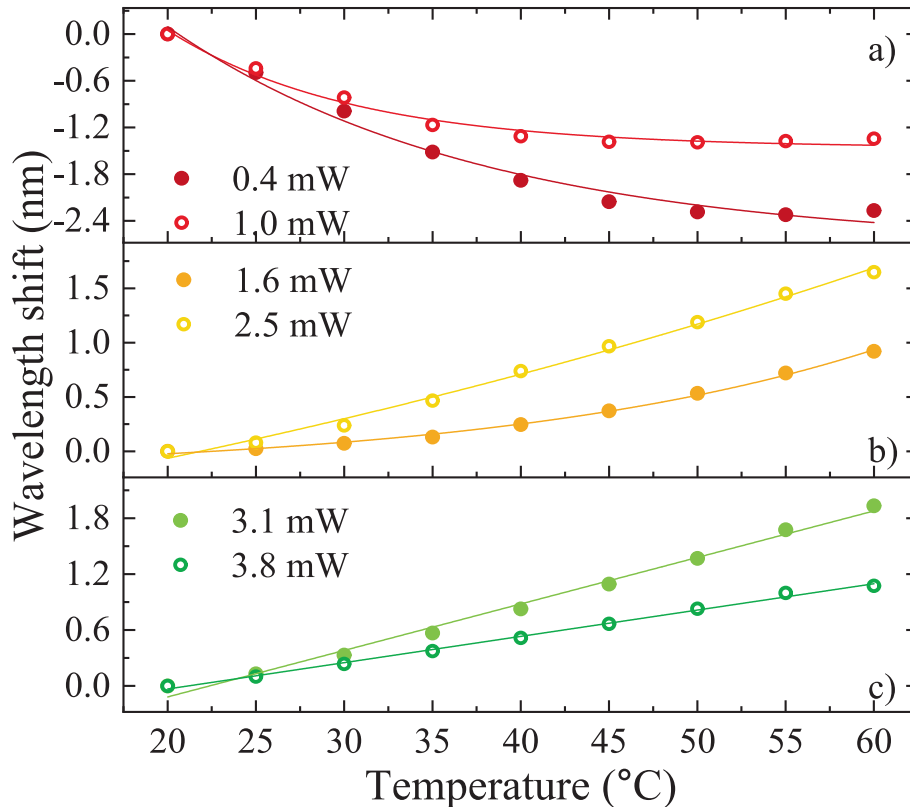


Fig. 9. Temperature characterization post-annealing of SWWs for the: a) Region I, b) Region II and c) Region III sensors.

for curing, and it is recommended to do a long thermal treatment that includes annealing. Note, however, that if the laser power is too high, a

material cluster is obtained, compromising the interferometric cavity [13].

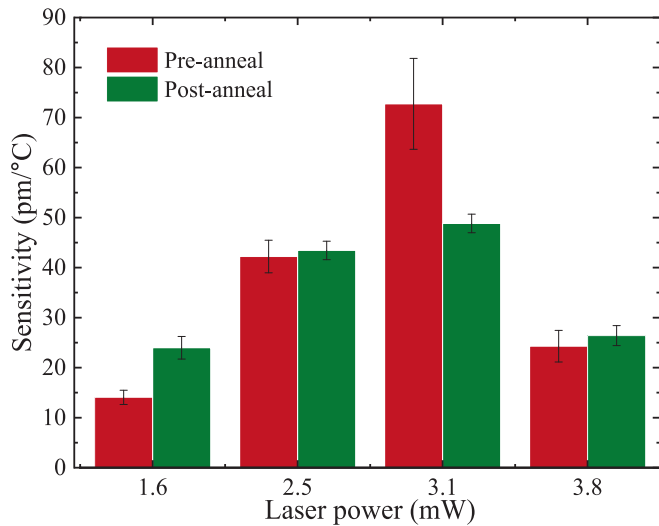


Fig. 10. Average temperature sensitivity and corresponding fitting error pre- and post-annealing for sensors fabricated with different laser powers.

To further confirm the importance of post UV exposure curing, a temperature experiment was carried out before any thermal curing on different sensors, and it was found that the response is highly unstable during the same temperature step. There was no linearity in the wavelength shift and hysteresis was very significant. On another case, annealing was done first instead of thermal curing, and it was found that initially the temperature response was unstable and unreliable, and as the sensor was thermally cured throughout the temperature experiments, the response was the same as the one attained for the flowchart in Fig. 7. This corroborates the requirement of curing in the first place, and

also that the order of thermal treatment does not have an impact in the end response of this sensing structure.

5.2. Relative humidity tests

Utilizing the same setup, the sensors were characterized toward variations of RH, after curing, in the climatic chamber, wherein this parameter was varied from 25 to 70 % RH, in 1 h steps of 5 % RH, with temperature fixed at 40 °C. As seen in Fig. 11, every sensor reveals high linearity to this parameter and a sensitivity of 60–80 pm/%RH. For all sensors, the stability of the SWWs to this parameter at the curing step is also significant, in contrast with the temperature response not being stable for all sensors at this curing step. Since the RI of the polymer is higher than that of water, the RI decreases as water is absorbed, and the wavelengths of the FP-based SWW would shift to shorter wavelengths. Therefore, since the RH sensitivity led to a red shift, the expansion of the polymer during water absorption is the dominant effect over the RI decrease.

The RH experiments were also tested for all sensors post-annealing, with the resulting wavelength shifts shown in Fig. 12.

It can be concluded that annealing has lower influence on this parameter, regardless of the laser power used to write the SWW sensors and that the ability of the material to absorb water molecules is rather an intrinsic property of NOA68 that is not influenced by thermal treatments. From Fig. 13, it is observed that the sensitivity of the sensors to RH does not dramatically change post-annealing, showing only a slight shift whilst maintaining a positive linear response and a similar linear regression error. The increase of densification and hardness from annealing did not have an impact on the swelling of the FPIs with increasing RH. The R^2 of the sensors in all regions was between 0.988–0.998 pre-annealing and 0.991–0.999 post-annealing.

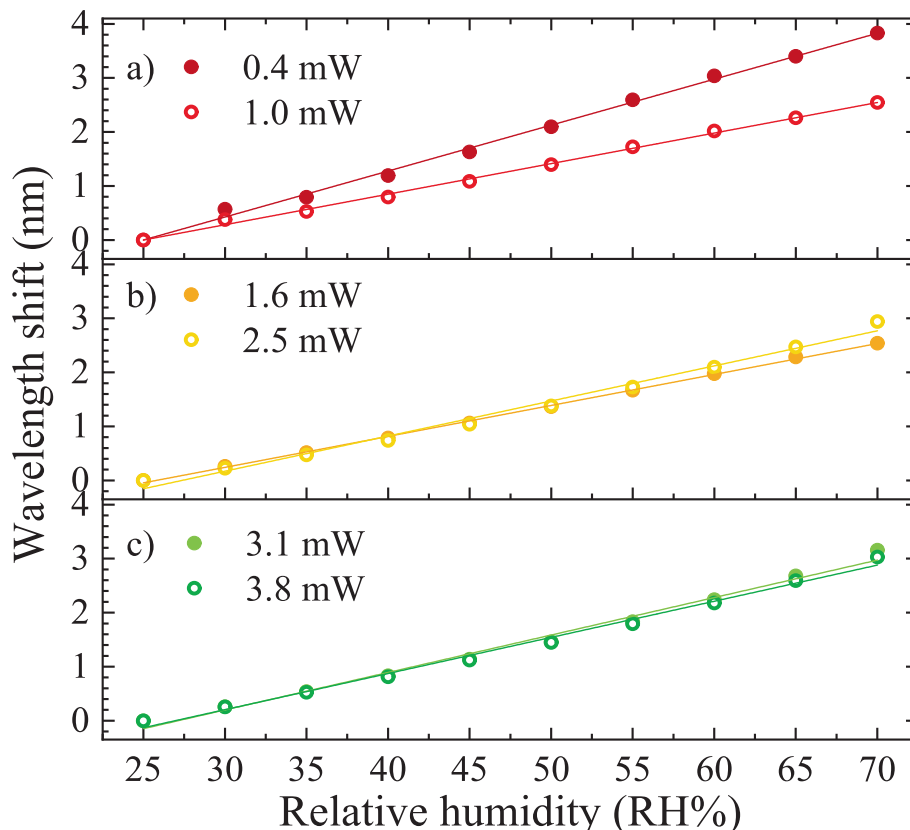


Fig. 11. Relative humidity characterization after thermal curing of SWWs for the: a) Region I, b) Region II and c) Region III sensors.

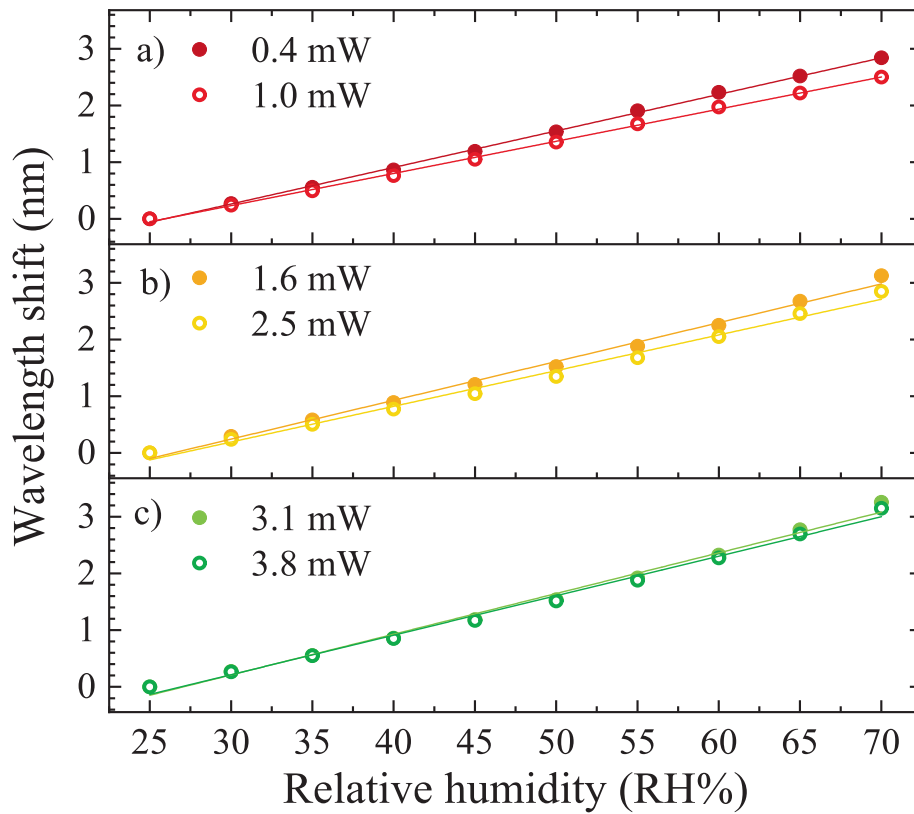


Fig. 12. Relative humidity characterization post-annealing of SWWs for the: a) Region I, b) Region II and c) Region III sensors.

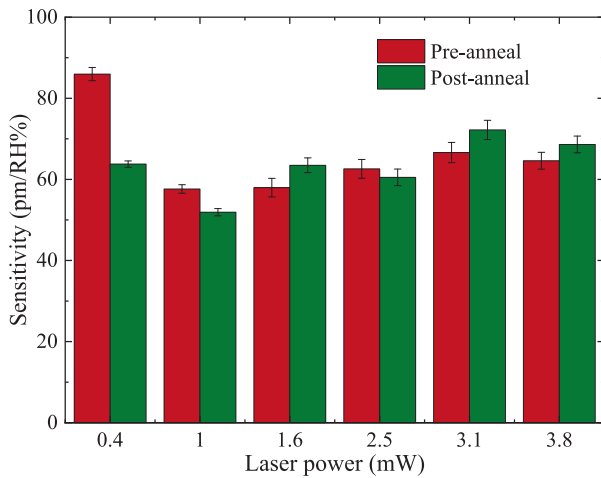


Fig. 13. Average RH sensitivity and corresponding fitting error pre- and post-annealing for sensors fabricated with different laser powers.

6. Discussion

In addition to analyzing the sensor behavior to the parameters of interest, it is, also, relevant to perform additional discussion on other aspects such as repeatability, hysteresis and resolution. To verify the repeatability and hysteresis of the experiments, as well as the resolution of the sensor for both temperature and RH, multiple cycles were performed with an increase and decrease of temperature post-annealed treatment, and one cycle of the RH experiment was compared pre- and post-annealing. In order to calculate the predicted temperature and RH shifts from the sensor, the obtained wavelength shift was multiplied with the previously attained sensitivity to the measurands, resulting in

the experimental measurand values. These results were superimposed with the climate chamber reading in Fig. 14.

From Fig. 14a, the reliability of this sensing structure towards temperature was verified, as the wavelength shift is quite consistent throughout the several increase and decrease step cycles, revealing lower hysteresis for the different cycles. The spikes at the beginning of each step are a result of the climate chamber stabilizing the assigned temperature and RH values. The measurement error of the sensitivities between the cycles is quite low at 3.4 %, with the average sensitivity corresponding to (47.5 ± 1.6) pm/°C. In Fig. 14b, the post-annealing reveals higher wavelength shifts per step and also that the steps are slightly more pronounced at higher values of RH, particularly above 50 %. Nonetheless, a linear fit was perfectly suitable for the characterization of this parameter. By zooming in on one step of the experiments, one can compare the response time and stabilization time of the climatic chamber response with that of the sensor. In a first approximation, the response time of the sensor is ~ 50 s, while the stabilization time is ~ 100 s pre-annealing and ~ 60 s post-annealing.

One poses, however, the matter of cross-sensitivity, since this FPI was only analyzed as a one-cavity, selecting one peak to track over the wavelength shift. The decoupling of two parameters is not possible in this configuration. The cross sensitivity of temperature to relative humidity was 0.615 °C/%RH and for relative humidity to temperature was 1.625 %RH/°C. Since these values are considerable, this device is only suitable for single-parameter detection under a controlled environment. As an alternative, for dual-parameter discrimination, an extra sensing head can be added to this sensing design (such as a fiber Bragg grating), in order to isolate one of the parameters.

When comparing with previously reported works, the implementation of a SWW for sensing has been explored. As shown in Table 2, SWW sensors for temperature and/or RH were developed with high sensitivity. The main contribution of our work is the in-depth study of resolution and hysteresis, which is often not reported or not compensated for. Overall, while higher sensitivities to RH and temperature using

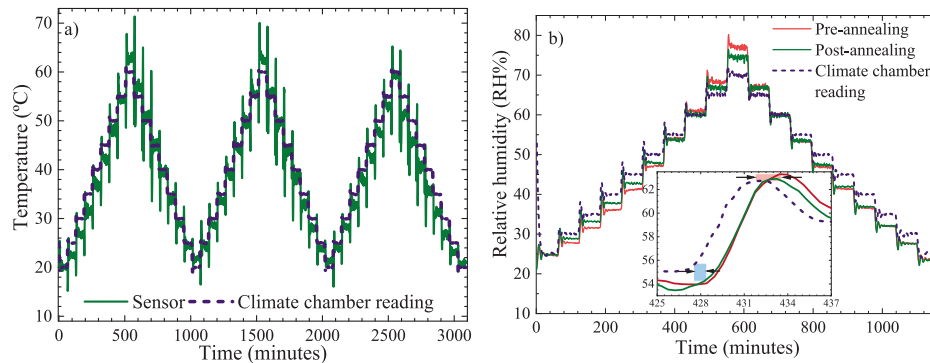


Fig. 14. a) Three-cycle temperature post-annealing experiment. b) One cycle RH experiment pre- and post-annealing: sensor response against the climatic chamber response. In-set: Zoom in on the RH setpoint increase. the blue square denotes the response time, and the pink square denotes the stabilization time.

Table 2

Comparison of sensing performance of FPI fiber tip polymer sensors.

Configuration	Temperature sensitivity (pm/°C)	Humidity sensitivity (pm/% RH)	Resolution T (°C)	Resolution RH (%)	Hysteresis	Cross sensitivity	Ref
2 SWW (PETA and PETA + PDMS)	1188	—	N.R.	—	Negligible	—	[1]
SWW (QUINSON)	−177	—	0.4 %	—	N.R.	—	[3]
SWW (PETA + PDMS + TPM)	1180	Insensitive	N.R.	—	Low	3 %	[9]
Dip coating (NOA61) and SWW (PETA)	24	202	N.R.	N.R.	Negligible	N.R.	[10]
Dip coating (Chitosan)	98.13	846 (nonlinear)	—	0.37	Low	0.1027 %RH/°C	[26]
2 SWW (NOA68 and PETA)	61	46	N.R.	N.R.	N.R.	0.49 % T	[27]
	25	202				1.64 % RH	
Micro rod (PMMA)	90 (nonlinear)	660	—	0.5 nm	Present	N.R.	[28]
SWW (PETA)	99 (nonlinear)	90	N.R.	N.R.	Present (Temp)	N.R.	[29]
					Low (RH)		
SWW (NOA68)	40	65	0.4	0.2	Negligible	1.625 %RH/°C 0.615 °C/%RH	Our work

SWWs have been attained, previous works suffered from: nonlinear RH response [26], nonlinear temperature response [29], high temperature hysteresis [28], used more complex polymers [1,3,9], had limited fabrication length method [1,3], with unreported resolution [1,9] and hysteresis [3] results. Despite these small advantages we report, the highlight of this work is the influence of thermal treatments on the material-state changes of a NOA68 SWW and how it results in differing wavelength shifts towards sensing.

To further corroborate the selected division of the laser power regions, several more samples were fabricated and fully characterized according to Fig. 7. From [30], considering an uncertainty of 95.4 % (“two sigma”), and the sensitivity, S , of the device given by the wavelength variation during one temperature step, $\Delta\lambda$, divided by the temperature step, ΔT , one can obtain the expression [31]

$$\delta T = \frac{2\sigma_\lambda \Delta T}{\Delta\lambda} \quad (5)$$

where the temperature resolution, δT , of a sensor can be calculated. σ_λ denotes the maximum wavelength standard deviation that happens during the same measurand step. For a 5 °C step, the resolution of the sensors was calculated, as shown in the statistical distribution in Fig. 15b. In Fig. 15a, the respective temperature sensitivity of each region is also shown.

There is, again, a clear difference in the post-annealing temperature sensitivity of Region I compared with the other regions. Regarding sensitivity, there is an improvement for the higher laser power regions and, overall, the resolution is better post-annealing for all regions.

Using an analogous derivation compared to (5) the RH resolution of the SWWs, whose RH sensitivities are shown in Fig. 16a, was also

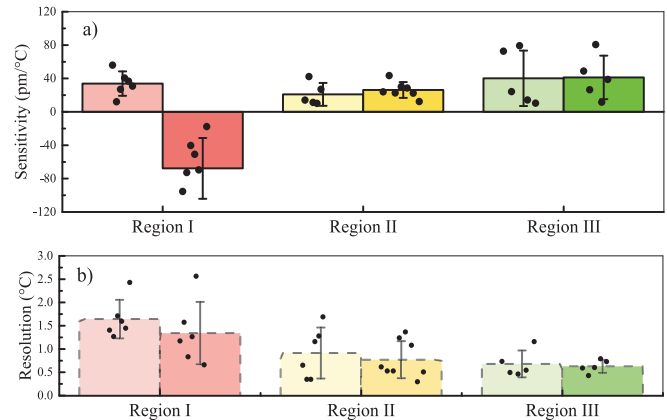


Fig. 15. Temperature sensitivity (a) and resolution (b) for each laser power region. The lighter colored bars indicate pre-annealing while the darker colored bars denote the post-annealing results.

calculated, as shown in Fig. 16b.

One notices not only an improvement of resolution for the higher regions, but also an improvement for all regions pre- and post-annealing. This technique allows for a fast fabrication low-cost process where a very small volume of material is used, and, in detriment of dip-coating and other SWW methods, grants the versatility of defining the desired length of the structure with very well-defined FP mirrors that result in high visibility spectra. While the desired application of this structure for our work was for the detection of measurands, from a thermo-

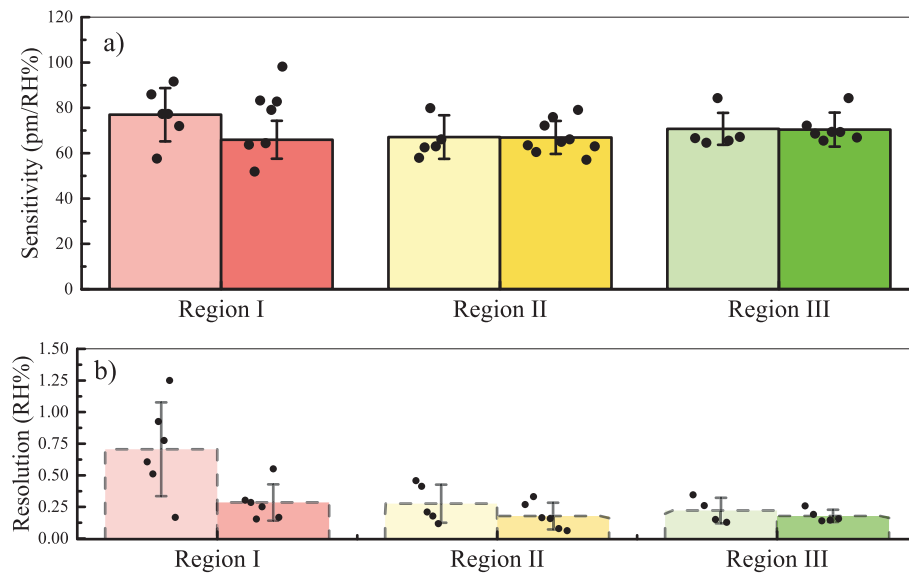


Fig. 16. RH sensitivity (a) and resolution (b) for each laser power region. The lighter colored bars indicate pre-annealing while the darker colored bars denote the post-annealing results.

mechanical point of view, the understanding of the response of this device with varying levels of power curing and thermal treatment is crucial. This study provides insight into how the degree of conversion, crosslink density, and residual stress state influence the dimensional stability of this structure. The varying sensitivities of the FP cavities can be directly interpreted as manifestations of changes of the material's thermal expansion, elasticity, and internal stress. This informs how the mechanical design of this kind of UV-curable polymer microstructures should be processed, given its direct impact in dimensional and thermal stability, as well as long term environmental robustness, which are key parameters to the reliability of this material. For this purpose, the most stable SWW structure was found to be fabricated at higher laser powers, in the 3–4 mW range, and after both a long curing and slow annealing process. Specifically in a sensing application, temperature stability is ensured after thermal curing and resolution for both temperature and RH are maximized when the device undergoes thermal annealing. One also points out, however, that for all powers used, a stable structure was attained, with a well-defined structure whose length only depended on the distance between the fiber and the substrate, where the desired interferometer was obtained in a reflection spectrum. The three laser power regions only have an impact on the measurand response.

To further understand the material parameters evolution and to complement the findings of our work, we suggest, as future work, the use of complimentary techniques, such as Raman spectroscopy, dynamic mechanical analysis, thermomechanical analysis, or ellipsometry.

7. Conclusion

In summary, SWWs based on Norland Adhesive UV curable polymer (NOA68) were fabricated on SMF tips with varying laser powers. The structure resulted in an FP cavity which was characterized in a reflection scheme to temperature and RH variations throughout a two-step thermal curing process. It was found that the stability of the sensor highly improves after thermal curing at a fixed temperature for several hours, obtaining a temperature sensitivity of approximately 40 pm/°C and an RH sensitivity of 65 pm/%RH. An annealing step was also performed for the sensors where it was seen that the temperature profile remains the same post-annealing for SWWs fabricated with high power (≥ 3 mW) while for lower power (≤ 1 mW) laser written SWWs, the temperature result flips to a two-region profile: at low temperatures the sensitivity was high and shifts towards shorter wavelengths, and at higher

temperatures the sensitivity was lower and almost insensitive. For sensors with intermediate laser writing powers, the temperature profile was unchanged at high temperatures, with lower sensitivity attained at lower temperatures. The response of the sensors to variations of RH behaves similarly pre- and post-annealing, regardless of the laser power used, with only slight improvements in sensitivity and resolution. A material characterization was performed to visualize the geometry changes of the samples. The hysteresis of the structure post-annealing was quite low for both parameters, showcasing great repeatability during the several characterization cycles. High resolution was attained, with the best values achieved post-annealing of 0.4 °C and 0.19 % RH for Region III sensors.

CRediT authorship contribution statement

João P. Santos: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Jeroen Missinne:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Micael Nascimento:** Writing – review & editing, Supervision, Methodology. **Marta S. Ferreira:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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