Research Article

PVC formulation study for the manufacturing of a skin smart structure based in optical fiber elements

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The development of smart solutions based on optical fiber technology for any kind of structure such as buildings, aircrafts, or even for human body kinematics, is becoming more and more common. The ability to provide coverings for different structures that can enable monitoring functions beside the esthetic purpose of the covering is an important add-value characteristic. Nevertheless, an open issue is to find an effective solution for the fabrication and application procedure, preferably that scales at a production level. This article explores the insertion of optical fibers into polymeric PVC foils based on the spread-coating fabrication process. The success of this integration approach allows the use of photonic technology in different fields with minor application issues. The material choice for the substrate is a crucial step when choosing integrated solution. Three PVC paste formulations were explored in order to guarantee the optimal integration of optical fiber. A high-viscosity and not monolithic paste formulation emerged as the best choice. This formulation provided the best adhesion, reducing in great scale the surface undulation and paste displacement that the optical fiber tends to do and, its 362% stretch capability is sufficient for special applications, as strain sensitive one. Copyright © 2010 John Wiley & Sons, Ltd.

Keywords: polyvinyl chloride; plasticizers; spread-coating; formulation; integration techniques; optical fibers

INTRODUCTION

The most commercially successful devices based on optical fiber technology were aimed to markets where conventional technology was not applicable. The cost of optical fiber technology is still more expensive than electronic and/or mechanical solutions and, consequently, they have only penetrated where their unique advantages justify the investment or where they are the only advisable option. As component prices have been falling down and quality has been improving, optical fiber technology is starting to replace standard technology in different applications, from communications to sensing.[1,2]

Nevertheless, still about 50% of the cost of such systems is for installation, which is labor demanding.[3–5] In the majority of the cases, the fiber optic element is mounted over a host structure surface, using epoxy resins,[6] or by welding methods,[7] in a very labor-demanding work. Both approaches present few concerns due to the fragility of the optical fibers.

The solution for overcoming these concerns has been already proposed in different works, in which, the optical fibers are integrated in substrates that are then applied to the host structure.[8–10] Even considering the success of such approaches, there is a need for an extensive study about the polymer formulation to manufacture smart structures based on optical fiber sensors in industrial environments.

Overall, the integration concept is a problem of great complexity due to the different areas that it addresses, from mechanical to thermal, sensing, and electronics.[11] The research on the integration of optical fiber sensor has mainly been pushed by the textile approaches, especially in the field of geo-

textiles[12,13] and wearable devices.[14,15] Even though the polymer approach may resemble the textile one on its broad concept, it presents its specific issues. Such concerns include weight, resistance, bond between the optical fiber and the polymeric matrix, capacity to transfer thermal and mechanical stimulus to the sensors, or even the ability to block one of the components, for example, to avoid the temperature influence in the sensor and measure only the strain.

This paper presents an introductory study about the integration approach, material selection and foil layout that provides the best solution for the smart structure manufacturing.

Besides, the inexistence of such smart structures, manufactured in a production-line, is responsible for the relative low-market acceptance of optical fiber systems, despite the superior quality and performance in comparison to the conventional solutions.

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Therefore, industrial ways for optical fiber integration in smart structures is a crucial point. The benefits of this smart structure come from the easier applicability of foils modules in the monitored structure and easy access for replacement. Plus, the substrate has to ensure not only the full protection of the optical fiber but also, for sensing application, the correct transfer of stimulus from the host structure in which the smart structure is applied. Finally, any kind of functional devices should be integrated in the most discrete and elegant manner, in harmony with the surroundings.

The design of a smart structure fabricated by technology that enables the direct industrial transition is explored in detail. The industrial process selection is the starting point of the solution design. From the fabrication technique constraint, the structure layout and materials are defined. The main focus is achieving the correct polymer formulation for the structure, with respect to the chosen process and the desired smart structure performance.

SMART MATERIAL DESIGN

As the main goal is the structure fabrication at industrial environment, the manufacturing process needed to be first defined.

Integration technology

Spread-coating technology allows the manufacturing of foils for a broad range of applications, such as clothing, footwear, home decoration, waterproof tablecloths, tarpaulins, conveyor belts, wallpapers, floor mats, artificial leather, and automotive interior trimmings.[16] It is also one of the techniques that best enables the integration of optical fibers.

The spread-coating process (Fig. 1) consists on the spreading of polymer pastes over a moving substrate or carrier (e.g. release paper). The running substrate drives the polymer through a gap between the knife and the substrate, defining the layer thickness. After the coating application as a uniform layer over the substrate, the whole frame is inserted in the oven to cure. After heated above the curing-temperature (between 130 and 400°C, depending on the substrate formulation), the polymer becomes homogenized and a solid phase appears.

Structure layout

When thinking about a flexible foil, the need for an easy to apply product becomes evident. In this context the product should be handled without difficulties or, at least, without rubbing or damaging the embedded optical elements.
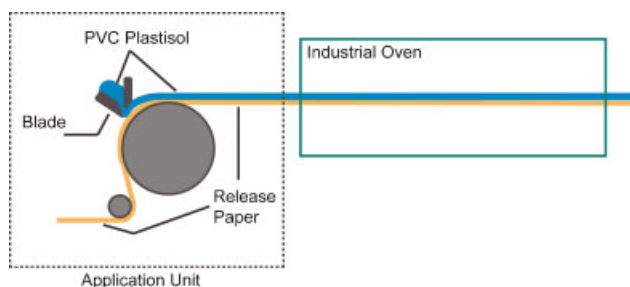
The type of foil produced by the spread-coating method plus the optical fibers insertion advise the integration of the fiber in the polymer carrier structure itself and not in other substrate (as a fabric) that is later integrated in the carrier. The direct integration reduces the friction and damage risk of the optical elements. This approach is more advantageous in respect to the protection given to the photonic elements. Furthermore, the insertion into the carrier ensures a better bonding between the structure elements and subsequently a good transference of external stimuli from the host material to the sensor. As the main concept is based on the use of glass fibers as optical elements, a multilayer structure approach is the most suitable (Fig. 2).

In the advanced manufacturing process, the materials choice plays a crucial role in the final performance of the structure. The material first needs to fulfill the requirements of the industrial process. The final formulation has to ensure that the smart structure can be applied to regular and non-regular surfaces by a diversity of methods. For specific application as the sensing one, it has to guarantee that the substrate interference in the sensor structures is not overlooked.

Flexible foil material selection

For the development of flexible sensing foil based on optical fiber sensors, the manufacturing technology needs to enable mass-production with customization of foil size, fiber layout and finishing styles. Thus, it was decided to choose a polymer matrix not only with an acceptable average quality and price to be as much compatible as industry's strategies, but also with characteristics that would best enable the sensing performance of the smart structure on its application field.[17,18]

Although flexible skin-like foils can be made of different polymers, with respect to the described design constraints, three main materials were considered to discussion, polyurethane (PUR), polyolefin (TPO), and polyvinyl chloride (PVC). Table 1 presents a comparison between their core properties.

PUR presents itself as the option with best performance. It feels like leather, with very long durability and high-performance in regard to flexibility. Nevertheless, PUR-based artificial leather is one of the most expensive skin materials in the field of flexible polymeric foils.[19,20]

TPO based artificial skin is a suitable alternative for the required objective with a much lower price and lighter weight. However, its flexibility and elastic limit are poorer than the TPU case.

The choice was set on plasticized PVC, for its general good cost/performance ratio and easy use during manufacturing processes.[21] In characteristics as yield, flexural, and shear
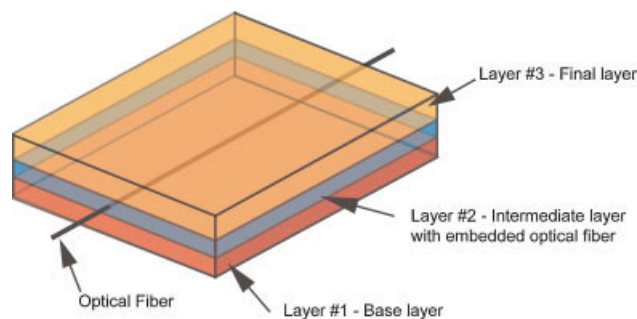

Figure 1. Spread-coating process. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

Figure 2. Multilayer structure for embedded optical elements in flexible PVC foil. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

Table 1. Polymers comparison[25]			
	TPU	TPO	PVC
Price (€/kg)	8.94–9.84	1.33–1.47	1.33–1.47
Density (kg/m³)	1100	893	1260
Shear modulus (GPa)	0.0114	0.00841	0.00395
Yield strength (elastic limit) (MPa)	42.90	7.23	19.00
Compressive strength (MPa)	51.40	8.68	22.80
Flexural strength (MPa)	69.3	5.25	35.7
Shear strength (MPa)	42.90	7.23	20.20
Elongation at yield (%)	597	796	346
Fatigue strength at 10^7 cycles (MPa)	17.10	2.89	8.08

strength, PVC places itself close to the PUR option (Table 1). PVC certainly is one of the most versatile plastics, still playing a major role in the building, packaging, and automotive market. Furthermore, PVC exhibits many advantages like highly competitive production cost, versatility, resistance to ageing, and maintenance simplicity.[22]

SMART MATERIAL FABRICATION

The PVC matrix had to be roughly defined without neglecting the important compromise between its flexibility and dimensional stability, which is important to ensure protection to the optical fibers and sensors embedded in the foil. The elasticity or flexibility, as well as the dimensional stability are mainly dependent on the choice of the PVC resin itself, besides the plasticizer type and concentration.

In addition to the elasticity and hardness, some other important aspects like price and organic volatile emissions are of crucial importance and have to be considered at the early development stage. Volatile organic compound emissions are in the basis of a lot of severe quality standards. Such emissions in flexible PVC products are mainly due to the volatility or

migration ability of the used plasticizers. Weight loss tests play a major role in the quality control, as they deliver the amount of volatilized plasticizer when the flexible PVC foil is submitted to high temperatures.

As it is also known, the optical fibers have a residual tension. When bending an optical fiber, it tends to recover its initial state after releasing the bending load. Consequently, it is important that the intermediate layer, where the fiber is embedded, is able to keep the fiber steady in place after its deposition.

Considering all the industrial conditions (maximum heating cycles and process temperatures) and the desired structure, the recommended process conditions are expressed in Table 2.

Formulation compounds influence

Before dealing with the formulation, it is important to understand the effects and the choice of the used basic compounds for PVC pastes. At this stage, plasticizer hardness and volatility, and PVC resins are discussed.

The effect of plasticizer was tested with a basis formulation composed of a widely used micro-suspension polymerization PVC resin without any additives. This PVC resin is of low-viscosity and perfectly suitable from light to heavy plasticized applications. For each formulation with the same basis formulation, but with different plasticizer type and concentration, the hardness was measured in the *Shore A* scale according to DIN 53505 and plotted in function of the plasticizer concentration (Fig. 3).

The plasticizer influence analysis shows, for high molecular weight plasticizers, a low plasticizing effect. Figure 3 also shows that for a given plasticizer concentration, the smallest hardness value is obtained with the plasticizer of low molecular weight, like diisobutyl phthalate (DIBP). This means that specific foil hardness can be achieved, using less plasticizer quantity if the plasticizer is of lower molecular weight as DIBP. This is not of practical interest, as this may result in low flexibility due to low quantity of plasticizer. For a given hardness, high quantities of plasticizer are recommended to achieve more flexibility in the foil. Consequently, it is advisable to use high-molecular-weight plasticizers. Trioctyl trimellitate (TOTM) plasticizer meets this concern. However, this plasticizer is more expensive than other plasticizers. The second choice is diisodecyl phthalate (DIDP), which performance is relatively close to TOTM performance for a lower price.

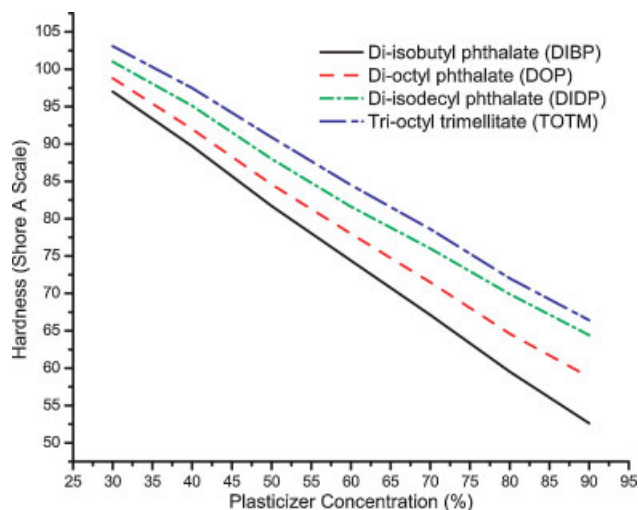| Table 2. Fabrication process conditions				
			Condition	
Step	Operation	Gap (μm)	Temperature (°C)	Heating time (sec)
1	Application of PVC-layer 1	200	—	—
2	Heat-curing of PVC-layer 1	—	200	60
3	Application of PVC-layer 2	300	—	—
4	Optical fibers insertion	—	—	—
5	Heat-curing of PVC-layer 2	—	200	60
6	Application of PVC-layer 3	400	—	—
7	Heat-curing of PVC-layer 3	—	200	60
8	Cooling + manual release			

Figure 3. Influence of plasticizer in the hardness flexible PVC foils. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

The migration of the tested plasticizer types was evaluated by weight loss. Samples of a reference PVC formulation with 35% ($\pm$0.5%) of plasticizer in pure micro-suspension polymerization PVC resin were exposed to high temperatures in a ventilated oven. The weight loss was measured with changing exposure time (Fig. 4).

The exposure to 165°C over the time shows that the highest volatility is registered with the plasticizers of low molecular weight. Figure 4 confirms that high molecular weight plasticizers, as DIDP and TOTM, have lower volatility than dioctyl phthalate (DOP) or DIBP. It is also interesting to notice that the performance of the DIDP is very close to the TOTM. From the obtained results, the plasticizer type more advisable for manufacturing of flexible optical sensing foils is DIDP.

The choice of the resin type was primarily focused on grades that are generally recommended for artificial leather foils. Several resin types were tested, regarding to their rheological behavior
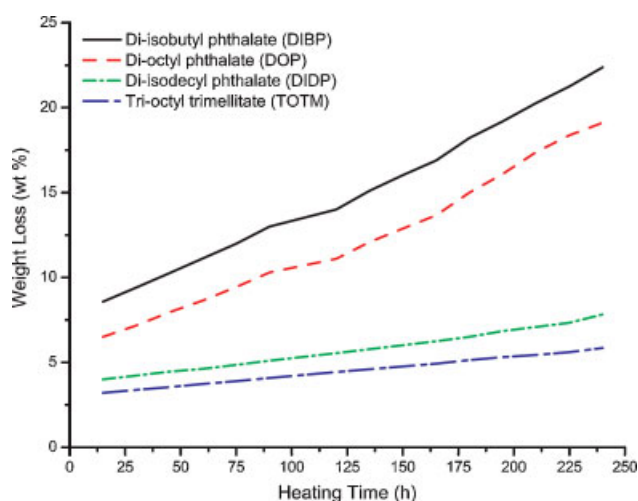
in plastisols. The plastisol samples were prepared with a concentration of 35% ($\pm$1%) of a high molecular and linear C10 + C12 phthalate plasticizer.

For process purposes, the viscosity characteristics of 8 different PVC suspensions (Table 3) were evaluated using a rotational viscosimeter with coaxial measuring system *Rheomat 115* (*Contraves*). Each plastisol formulation had its viscosity measured at 8 different shear rates at a constant temperature (Fig. 5). The viscosity readings were performed after 24 hr rest-time.

The PVC resin type is an important factor for the properties of the final product and it is also a major player in the foil manufacturing process. For industrial spread-coating, it is generally established that the ideal production speed should be in the range of 15–20 m/min. This production speed corresponds to the speed at which the release paper is pulled through the plastisol application unit and it is equivalent to an average shear rate of, approximately, 25 sec^{-1}.

Experimental

According to the characteristics that are desired for the integration layer formulation and industrial process, three different approaches were prepared (Table 4).

In the formulation #1, plastisol with low viscosities was targeted in order to avoid a bad spreading of the PVC paste over the release paper and also over the optical elements that should be covered by PVC. Attending to this requirement and according to Fig. 5 results, the suitableness of the grade PVC$_{RES}$6 was demonstrated. This resin exhibits the lowest viscosity in the shear rate range between 25 and 55 sec^{-1}, which constitutes a realistic shear value interval for ideal industrial spread-coating conditions.

Formulation #2 presented an intermediate viscosity level when compared to the other two. Its resins demonstrated thixotropicity behavior with falling viscosities as shear-rate is increased. Such formulations exhibited high viscosity at steady state but when shear forces are applied, for instance during the spread-coating process, the viscosity falls.

Figure 4. Plasticizer weight loss versus exposure time to high temperature. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

Table 3. PVC resins description

| Resin	Description	Rheological behavior
PVC$_{RESIN}$ 1	Emulsion polymer (MW: 104,000)	Dilatant
PVC$_{RESIN}$ 2	Emulsion polymerization high gloss	Dilatant
PVC$_{RESIN}$ 3	Emulsion polymerization low penetration	Mixed
PVC$_{RESIN}$ 4	Emulsion polymerization high molecular weight	Mixed
PVC$_{RESIN}$ 5	Micro-suspension homopolymer	Dilatant
PVC$_{RESIN}$ 6	Micro-suspension homopolymer low viscosity	Dilatant
PVC$_{RESIN}$ 7	Micro-suspension homopolymer high load	Complex
PVC$_{RESIN}$ 8	Emulsion polymerization foam	Thixotropic
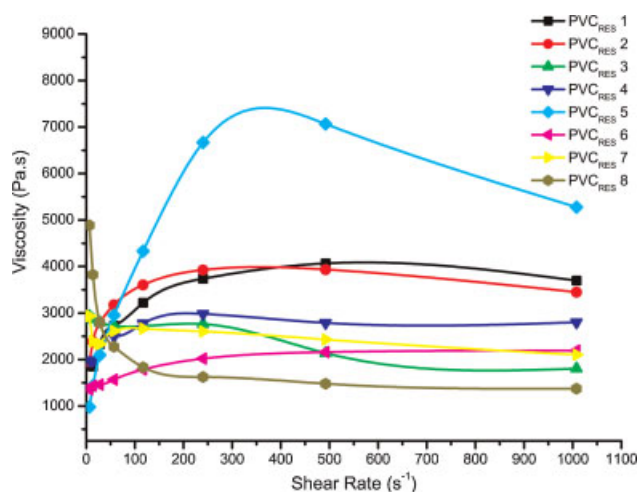
Figure 5. Viscosity response to shear rate for tested PVC resins. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

Formulation #3 was developed to provide a high viscosity. The starting formulation was the simple resin suspension PVC$_{RES}$8, which demonstrated thixotropic behavior and exhibited the highest initial viscosity. Formulation adjustments were carried out in order to raise the initial viscosity and to emphasize the fixing capability of the plastisol for deposited optical fibers. A strong increase in viscosity is achieved with the thickeners. Several thickeners were tested and finally the best result regarding homogeneity and viscosity of the suspension was obtained with a silica based thickener.

RESULTS AND ANALYSIS

Prototypes were produced with the three different formulations. All three proved to be perfectly suitable for spread-coating process. No difficulty was observed during the plastisol spreading through the gap between the knife and release paper, nor happened any damage to the carrier paper.

Surface analysis

The microscopic cross-section views of the flexible sensing foil (Fig. 6) demonstrate an optimal surrounding of the fiber by the mid-layer. Moreover, the pictures show that there are no irregularities on the foil, due to the fiber insertion. Therefore, the spread-coating process combined with the selected PVC

formulations constitutes a perfect method to cover, entirely and homogeneously, the optical fibers.

Immediately after releasing from the paper support, the foil presents an undulation on its surface, along the optical fiber path (Fig. 7 left). This effect is particularly intensified in the foils were the fiber is in straight line, since no undulation is detected in foils where the fiber shape was "S" layout (Fig. 7 right). The effect seemed to be related with internal stress at the interface fiber/matrix. This surface irregularity is the result of different heat expansion coefficients between optical fiber and PVC matrix. During the curing in the oven, the PVC matrix expanded by a much higher coefficient than the optical fiber. When the foil was cooled down, the PVC matrix contracted more than the optical fiber. This difference in the contraction mode was responsible for inducing internal stress in the interface of optical fiber with the PVC matrix. The structure in "S" shape allowed the fiber to follow, in a more effective manner, the dilatation and contraction of the PVC matrix during the cure and cooling stage.

Fiber unsteadiness (Fig. 8) due to its inner stress was detected in the prototypes made with formulations #1 and #2. The viscosity of these two formulations was not strong enough to avoid paste displacements. The surface of these two obtained prototypes was, therefore, still provided with few irregularities. The fibers unsteadiness problem was significantly reduced with high viscosity plastisol since, in formulation #3, no plastisol displacement is visible on the surface of the prototype.

Figure 9 shows a side-view of the folded prototype. It is visible that there is no irregularity on the surface. At the interface PVC/optical fiber, the surface is totally flat which proves that the optical fibers were perfectly fixed. Furthermore, the optical fiber is not felt when touching or rubbering the surface. This is also an indicator for an optimum integration level.

Mechanical properties

The mechanical properties were evaluated for the different PVC formulations. These tests were performed without the optical fiber element since the elongation is dependent on the fiber layout. The measurements were performed with a testing machine *Instron 4302*, enabling a controlled load to be applied. The load was measured in function of the resulting elongation. Loads were applied until the maximum tensile strength of the material was reached. The maximum elongation is representative of the formulation elasticity.

Formulations #1 and #2 sample foils reached almost the same ultimate elongation before breaking. However, for the formulation #1, the load needed to achieve the same elongation was

Formulation	#1 (%)		#2 (%)		#3 (%)	
Resin 1	PVC$_{RES}$6	55	PVC$_{RES}$3	40	PVC$_{RES}$8	42
Resin 1	—	—	PVC$_{RES}$8	13	—	—
Thickener	—	—	—	—	TH1	1
Plasticizer	DIDP	40	DIDP	37.5	DIDP	45
UV stabilizer	STAB1	1.5	STAB1	1.5	STAB1	1
Heat stabilizer	EZ1	1	EZ1	1	EZ1	1
Filler	CaCO$_3$	2.5	CaCO$_3$	7	CaCO$_3$	10

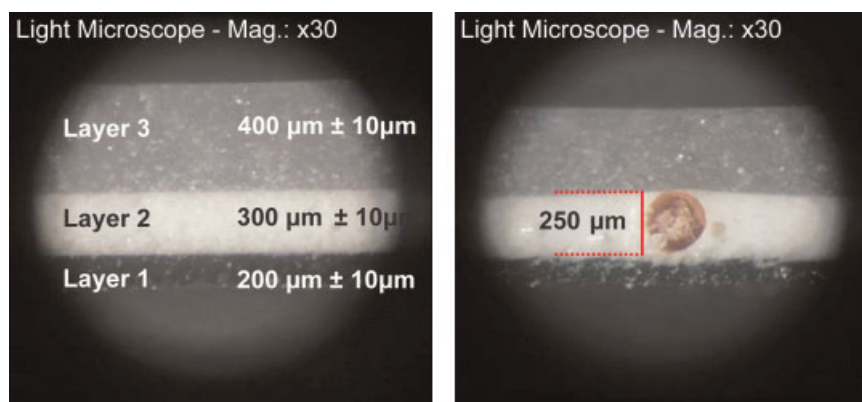**Table 4.** PVC formulations composition

Figure 6. Cross-section views of the multilayer prototype. The three layers on the left-side image and the optical fiber on the right-side image. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

Figure 7. Prototype with fiber in straight line layout (left); Prototype with fiber in "S" layout (right). This figure is available in color online at wileyonlinelibrary.com/journal/pat.

the lowest. This was due to the lower viscosity of formulation #1 when compared to formulation #2. Thus, the layer was more suitable to be stretched with less effort.

However, for the formulation #3, which had the highest viscosity layer, the prototype reached a lower ultimate elongation when compared to the other two (Fig. 10). Below 25% stretching,
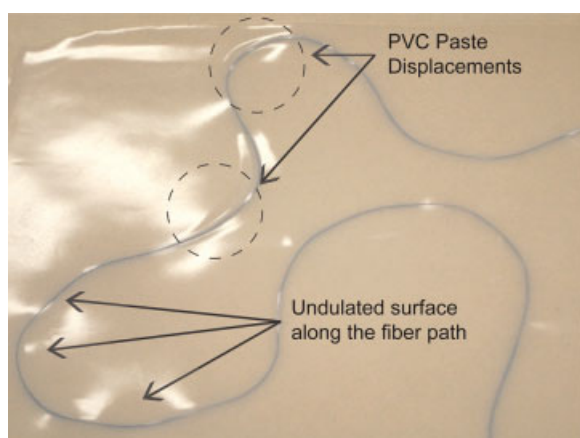

Figure 8. Visible irregularities in the prototype surface. This figure is available in color online at wileyonlinelibrary.com/journal/pat.
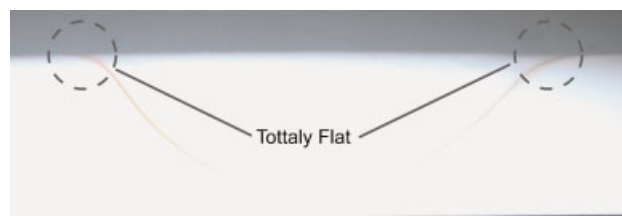

Figure 9. Side view of prototype. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

formulation #3 requires the highest load but, above 25%, it places itself between formulations #1 and #2.

The justification for this decrease of elongation capability and load behavior is presented in Fig. 11. The amplified cross-section showed that there were pores in the intermediate adhesive layer.

In fact, the used resin, in this formulation, is made of emulsion polymerization PVC especially designed for producing PVC foam. As no expansion agent is used in the formulation, a foam reaction was not expected. However, like it can be confirmed by a spectroscopic analysis, few pores were formed during the PVC curing period. This led to a PVC layer that was not monolithic or absolutely compact, like the layers formed by standard PVC foils. This foam-like structure was soft and easy compressible, like a sponge. The pores in this intermediate layer were in fact
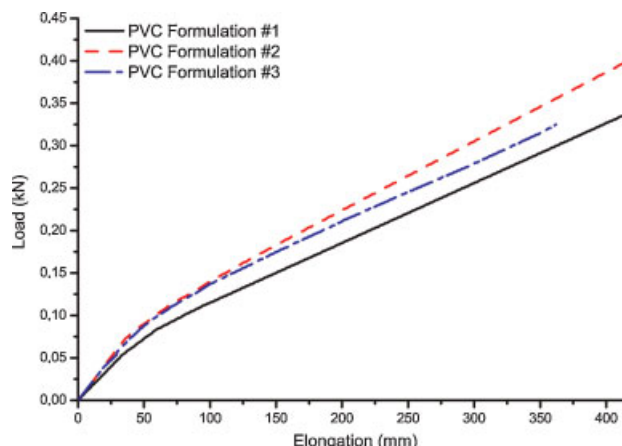

Figure 10. Elongation comparison between the formulations. This figure is available in color online at wileyonlinelibrary.com/journal/pat.
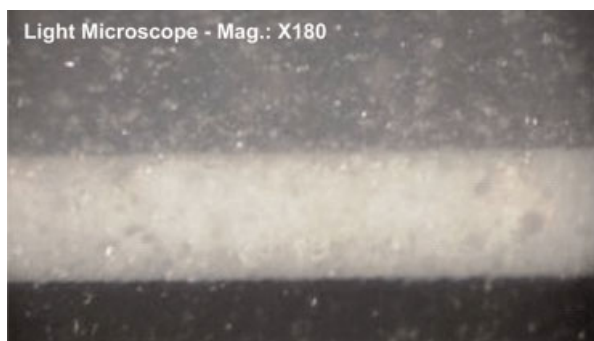
Figure 11. Amplified cross-section of the formulation #3 layer. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

discontinuities in the PVC matrix and constituted weak points, responsible for an earlier stretch–break in the elongation tests with low applied loads. Despite this loss in the elongation capacity of the obtained foils, the high elasticity modulus in the range of 4 MPa confirms more stiffness against deformation, while elasticity remained very high.

Sensing performance analysis

Although the formulation #3, from all developed formulations, has proven to have the best compromise between integration and mechanical properties, it was still necessary to evaluate its sensing properties. Fiber Bragg gratings (FBG) sensors were selected to be embedded by the developed formulation. As they are intrinsic optical fiber sensors with inherent sensitivity to strain and temperature, they become the best sensor solution for evaluate the performance of the developed smart structure.[23] The manufactured foil with embedded FBGs was glued to a metal plate in the same manner as it would be mounted in a host structure to monitor, and characterized as well at the mechanical and thermal level.

For the thermal evaluation, the metal plate was placed over a hotplate setup for cycles between 25 and 180°C. The sensor response to the performed test is plotted in Fig. 12. A fit was added according to the measured data from which was possible to establish a linear relationship, 0.01 nm/°C, between the sensor response and the temperature variation.
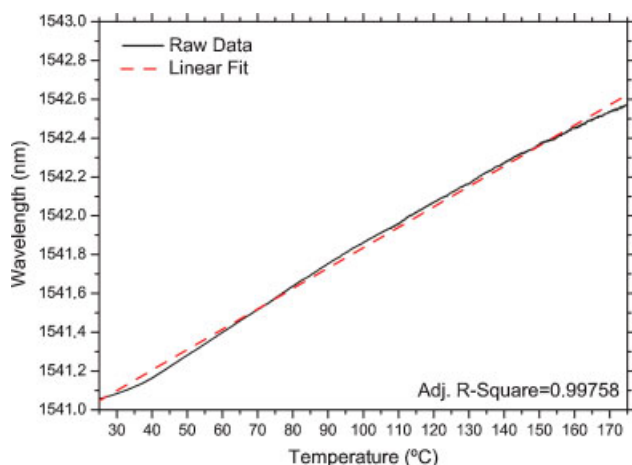

Figure 12. Temperature response of the smart structure. This figure is available in color online at wileyonlinelibrary.com/journal/pat.
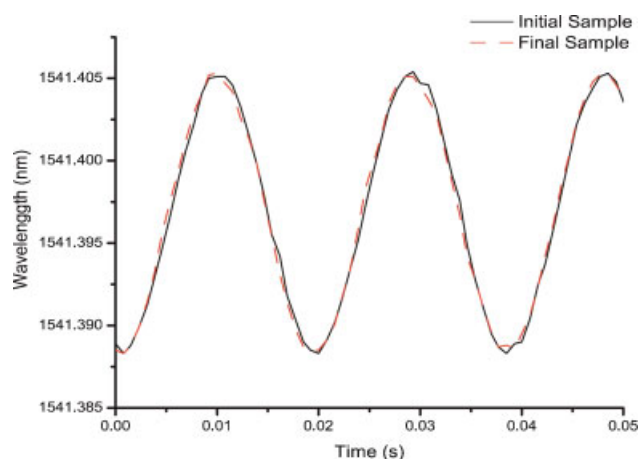

Figure 13. FBG response to strain cycles. This figure is available in color online at wileyonlinelibrary.com/journal/pat.

The mechanical test was performed with the structure in a cantilever approach setup. The metal plate was constrained at one end, while the other end was subject to 200 μm displacement cycles at a 50 Hz frequency in a total of more than 4 million cycles. The FBG response was monitored during the cycles, and the initial and final response are presented in Fig. 13. The FBG sensors have been able to follow with success the bending of the plate, resulting in a sinusoidal response with linear slopes between the cycle's limits. The sensitivity of the smart structure is 0.8 nm per 1% elongation. This value is lower than the free FBG sensitive, which is 1.2 nm for the same elongation.[24] The difference is due to the polymeric matrix that has a small constraining effect. Nevertheless, the obtained sensitivity is still compatible with many applications and commonly used interrogator devices.

Moreover, with time, the response did not suffer any change regarding its characteristics as the initial and final sample share the same response. Such behavior results from the successful bonding between the fiber and the PVC matrix, which could withstand the cycles without breaking the bonding.

CONCLUSION

The direct deposition of optical fibers during spread-coating needs a specific adjust of the PVC formulation in order to enable a stable deposition process. For this purpose, research was focused on the viscolestastic properties of the solid PVC matrix and on the rheological characteristics of the plastisol. The three presented formulations enabled, with success, the integration of optical fibers. However, the performance of each fabricated prototype was very different.

At the integration level, the formulations #1 and #2 did not have the correct characteristics to ensure the correct place of the optical fibers, not keeping it steady in place. By other hand, formulation #3 was able to fix the optical fiber after deposition. Also, with this formulation was possible to achieve a more homogeneous surface.

The mechanical behavior of formulation #3 is not the best of the three formulations set. It stretches less, 362% versus 425% of formulations #1 and #2. Also, for small elongation (below 25%) it is the formulation that requires the highest loads. However,

considering the elongation range of optical fiber, its performance is enough to avoid major sensitivity sensor loss.

From the overall results, the formulation #3 ensures the best integration of optical fiber technology in PVC foils, considering the spread-coating industrial process.

At the sensing performance level, the sensor demonstrated high sensitivity with an effective and durable bond between the fiber and the PVC matrix. The sensor response was linear to strain and temperature stimulus with minimum interference from the polymeric layers.

Furthermore, when comparing the developed structure with other integrated solutions, the prototypes show a robust structure, fully flexible, with reduced risk of fiber break and with minimum sensitive losses.

The smart structure performance as well as its inherent characteristics and customization capability ensure the competitive edge that polymeric substrates with embedded optical fiber sensors can have over other integration approaches.

Acknowledgements

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