

# Monitoring drying process of acrylic varnish with heterogeneous optical sensor

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## ABSTRACT

This work shows the application of a heterogeneous sensor system based on fiber Bragg gratings and optical coherence tomography to monitor the drying process of a commercial water-based acrylic varnish. The varnish mechanical deformation, thickness, and average refractive index developed in the process were monitored along 24 hours for a film with approximately 430  $\mu\text{m}$  of initial thickness, allowing identification of distinct time behaviors. Tomographic images exhibited refractive index discontinuities along the total film thickness indicating the development not uniform and concurrent through the whole material volume of the processes involved in the drying kinetics.

**Keywords:** Varnish drying process, fiber Bragg grating, optical coherence tomography.

## 1. INTRODUCTION

The major class of polymeric surface coating materials embraces paints and varnishes. Paint is a suspension of solid pigment particles dispersed in a liquid vehicle, which can be applied on a surface to form a solid film after drying. The dry film present pigments dispersed in a continuous polymer matrix that is invariant. In its simplest form, varnish is a non-pigmented paint. Considering the application, the polymeric film is selected by a variety of qualities including resistance, hardness and abrasion over a working range of temperature, weathering performance and resistance to chemical attack, as well as optical properties such as refractive index and transparency. Equally important are the properties of the liquid vehicle (solvent), which must be able to form stable dispersions with pigment, be of suitable viscosity and show a satisfactory drying rate<sup>1</sup>.

The drying process of film involves different phenomena and depends on the environmental conditions, film thickness, type of solvent, as well as the physicochemical properties of solution. In an attempt to explain the process several techniques were applied, although the involved drying mechanisms are still not fully understood<sup>2</sup>. Also, the development of such processes is not uniform through the whole material volume and they can be concurrent, increasing its complexity<sup>3</sup>. Despite these drawbacks, the proposed models to describe the drying mechanisms split up the process in three stages: evaporation followed by particle concentration and ordering; particle deformation; and polymer chain diffusion<sup>4</sup>. From the cited works, it becomes clear that to determine the features of varnish drying process it is convenient to use a sensing scheme able to measure multiple parameters at same time, providing information about mechanical and structural properties of the film. However, a single technique that fulfills such requirements is not readily available. Within this context, we propose an alternative method to study the time-behavior of a water-based acrylic varnish film that relies on the use of a heterogeneous sensor system (HSS). The sensor system employs two types of optical-fiber based sensors: fiber Bragg grating (FBG)<sup>5,6</sup> and a low coherence fiber interferometer in an optical coherence tomography (OCT)<sup>7,8</sup> configuration. By the employment of such composed system, multiple parameters as strain, temperature, thickness and refractive index changes can be simultaneously measured. For the FBG constituent, the main advantages are its high sensitivity, small size, electric and magnetic passivity<sup>9</sup>. This device was already used for monitoring the drying time of latex paints, but with the use of a rather complex encapsulated sensor head to increase the transducer efficiency<sup>10</sup>. For the OCT constituent, advantages comprise high spatial resolution and depth penetration what enables the possibility of tomographic measurements.

## 2. EXPERIMENTAL SET-UP

The first constituent of the HSS employs two FBGs, recorded in standard telecommunication optical fiber using a phase-mask illuminated by a KrF-excimer laser at 248 nm, to determine the stress and temperature evolution of varnish along drying. A coupler sends the light from a LED (Superlum PILOT 2) with wavelength range 1520 - 1570 nm to FBG 1 and FBG 2, with resonant wavelengths of approximately 1538 nm and 1540 nm respectively; the light back reflected is collected by a dynamic optical interrogator (Ibsen I-MON E 512D). The FBG 1 is covered with a coat of varnish and FBG 2 is positioned at approximately 1 cm above the painted surface for temperature monitoring. The FBG constituent of the heterogeneous sensor system is presented schematically in Figure 1a. FBG 1 is fixed on the sample holder in one side by a masking tape, while the other extremity passes by a pulley; a 5 g mass fixed in this tip provides a constant longitudinal tension, allowing its movement with the deformation of the varnish during the drying process. Once the FBG 1 is subjected to an initial longitudinal tension and is immersed in the coating film, the contraction and dilation of the varnish are indicated by changes in resonance Bragg wavelength. FBG 2 indicates only the thermal variations of the surroundings.

Commercial water-based varnish is applied with constant and controlled thickness, using a metallic guide (510  $\mu\text{m}$  height) and a glass plate to spread the paint over the fiber (125  $\mu\text{m}$  diameter). The formed varnish film adheres and covers the portion of the fiber with the acrylate jacket as well as the uncladded fiber portion where the grating is recorded. Mechanical deformations in the varnish during the drying process are monitored during 24 hours (5 Hz rate) with the dynamic interrogator. To minimize changes of temperature and humidity and airflow influence, the workbench is enclosed and the temperature is controlled to be  $(25.0 \pm 0.5)^\circ\text{C}$ . An additional electronic sensor was also employed to monitor the environment temperature (kept constant in the range from 24.5 to 25.5  $^\circ\text{C}$ ) and humidity variation (from 58.8 to 66.7 %).

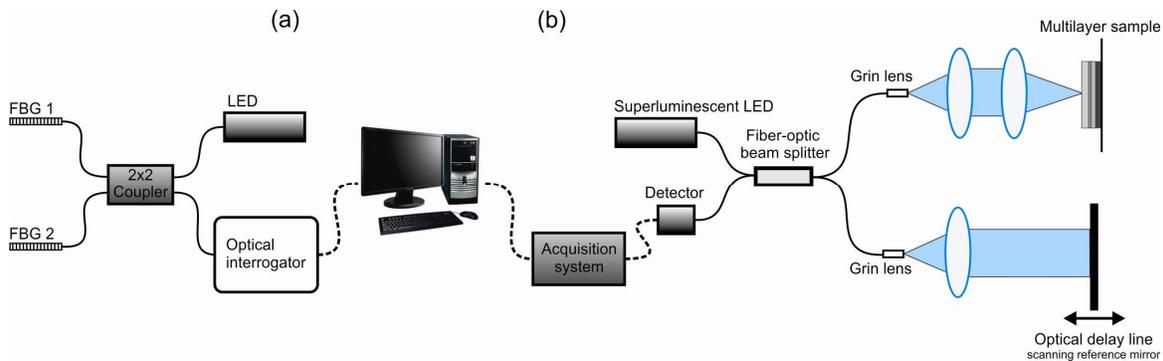


Figure 1. Heterogeneous sensor system: (a) FBG constituent and (b) OCT constituent.

The second HSS constituent, as shown in Figure 1b, characterizes the thickness and refractive index evolution of the varnish during the drying time. During the measurement, only the envelope of interference pattern function is considered and, using a previously-calibrated system, the interface position in the sample arm is determined by its center. Using a pre-established position of the sample holder, it is possible to determine the sample thickness<sup>11</sup>. Varnish refractive index is obtained from the measurement of the time delay between the two interfaces of the film: air-film and film-glass (sample holder). This time delay is an optical thickness  $\Delta s_{opt}$ , experimented by the light across the sample, and is related with the true geometrical thickness  $\Delta s$  and the refractive index  $n$  by relation  $\Delta s_{opt} = n\Delta s = n\nu\Delta t$ , where  $\nu$  is the reference arm scanning velocity and  $\Delta t$  is the time delay between these interfaces measured at the center of the envelope. So, if geometrical thickness is known, the group refractive index can be determined. In an inhomogeneous medium, like varnish in a drying process, the calculated index is an average of a gradient index inside the sample. The OCT was configured to take a transversal tomogram of the sample (3 mm depth, 10 mm width) in steps of 0.2 mm. The total time of each transversal image acquisition is about 5 minutes, and the characterization is carried out along 24 hours. Temporal central position of the interference envelope is related to the spatial position of the interface in the sample, whereas its amplitude is related to reflectivity by Fresnel's law.

### 3. RESULTS AND DISCUSSION

The strain effects associated with the varnish drying and measured by the FBG1, as well as temperature changes indicated by the FBG2, are shown in Figure 2a, while Figure 2b shows the time evolution of film thickness and average refractive index, measured by the OCT. Distinct time behaviors were identified in the drying process along 24 hours for a film with approximately 430  $\mu\text{m}$  of initial thickness. In the first 0.8 h (not depicted in the figure) the drying is characterized mainly by the water evaporation through the film surface. A poor film adhesion to the optical fiber surface prevents the observation of pure strain effects, and the FBG 1 response is probably associated to temperature and strain cross sensitivity. At this time interval, OCT images show the refractive index discontinuities along the film depth, mainly close the film surface. Water evaporation leads to a fast decrease of thickness with a  $-1.7 \mu\text{m}/\text{min}$  rate, as well as to an increase in the average refractive index with  $7 \times 10^{-4}$  RIU/min rate in the first 1.7 h. Both effects are associated with the water evaporation and the particle approximation, resulting in an increase of the material density. From 0.8 to 1.7 h a skin becomes to develop at the film surface resulting in lateral stress induced by capillary pressure and in the observed wavelength shifts associated with lateral expansion of the film. This time interval marks the beginning of the coalescence, nevertheless still remains a significant rate of water evaporation. The particle concentration is established from the upper film surface towards the lower one, reaching the whole volume at the end of this interval.

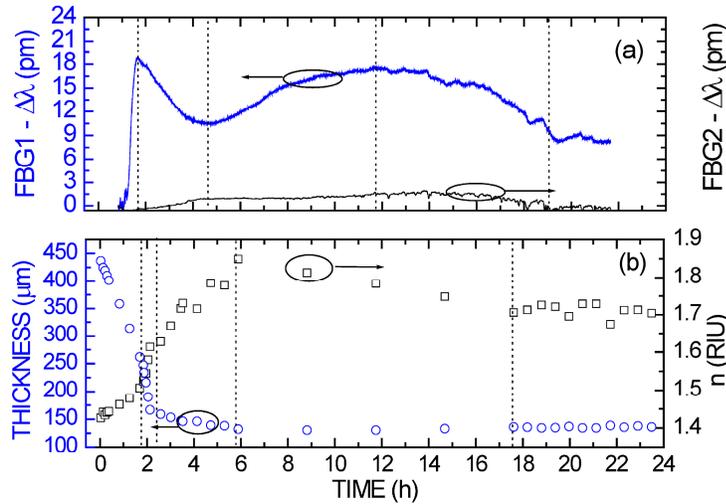


Figure 2. (a) Strain effects measured by FBG 1 and temperature changes indicated by FBG2, and (b) thickness and refractive index behavior measured with OCT throughout 24-hours drying process. Vertical dotted lines are used as a visual guide.

At 1.7 h, the tomographic image (Figure 3a) exhibits refractive index discontinuities along the total film thickness (depth), characterized by the high intensity of reflection measured by the OCT. The film thickness decreases to approximately 64 % of its initial value. At 1.7 h, an abrupt change in the mechanical deformation is observed and the film starts to contract up to 4.8 h (Figure 2a).

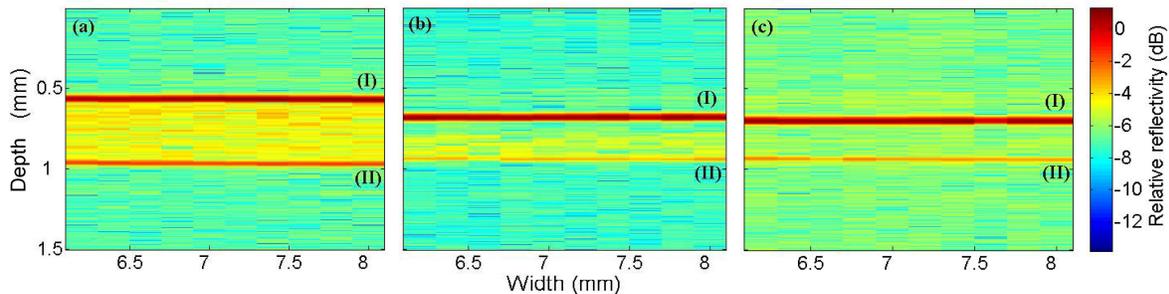


Figure 3. OCT images obtained at (a) 1.7, (b) 3.3 and (c) 10 hours. (I) air-film interface, (II) film-glass interface.

From 1.7 to 2.5 h, thickness decreases at a higher rate of  $-4.6 \mu\text{m}/\text{min}$  and the average refractive index increases with a non-linear behavior. Also, the refractive index discontinuities become less noticeable as the drying evolves. Such effects are compatible with the decrease in the evaporation rate and a more effective particle approximation. From 2.5 to 5.8 h thickness decreases at a lower rate, reaching an asymptotic value of  $133 \mu\text{m}$  (31 % of its initial value). The average refractive index increases at a  $9.6 \times 10^{-4}$  RIU/min constant rate and the tomographic images show the development of a uniform and thickness increasing upper layer surface from 3.3 h to 10 h (Figures 3b and 3c). Although after 5.8 h no important thickness changes are observed, important mechanical deformations still take place, characterized by dilation up to 11.7 h followed by contraction and further stabilization after 19 h. Refractive index decreases, stabilizing after 17.5 h. These observed changes even after the thickness stabilization are indicative of structural modifications still in course.

#### 4. CONCLUSION

The heterogeneous system based on FBG and OCT constituents reported in this work, demonstrated ability for monitoring the drying process of varnish films in a direct form. The results obtained showed that the strain sensor based on a fiber Bragg grating can be used for measuring mechanical deformation without the necessity of a special sensor package. While the FBG sensor can monitor strain and temperature changes, OCT has the ability to measure efficiently the film thickness and refractive index. Distinct time behaviors were identified to the varnish mechanical deformation, thickness and average refractive index during the drying process along 24 hours. Tomographic images exhibited refractive index discontinuities along the total film thickness indicating the development not uniform and concurrent through the whole material volume of the processes involved in the drying kinetics. The potential of the HSS application can be explored once the knowledge of kinetic process can lead to a better understanding of the various steps involved in drying of polymers.

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