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Polymer matrices for composite materials: monitoring of manufacturing process, mechanical properties and ageing using fiber-optic sensors

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Abstract. Composite materials have gained significant prominence in the field of aerospace engineering owing to their exceptional strength-to-weight ratio, making them well-suited for structural applications. However, these materials are susceptible to degradation due to exposure to environmental factors, such as humidity and temperature changes. Detecting and quantifying such damage presents considerable challenges, particularly in the case of cyclically loaded components. Fiber Bragg Grating (FBG) sensors provide a non-destructive means of monitoring composite material degradation by leveraging optical reflection to measure changes in strain and temperature. This research aims to assess and validate a methodology for employing FBG sensors to effectively monitor the degradation of composite material matrices. The investigation mainly consists in characterizing the correlation between FBG sensor wavelength shifts and the strains incurred due to the manufacturing process, moisture absorption, and thermal effects. The anticipated outcomes hold the potential to enhance the reliability and safety of composite structures employed within the aeronautical domain.

Introduction

The studies reported in this paper investigated the production process, conducted mechanical tests and evaluated the mechanical properties changes under different conditions in epoxy resin specimens. Understanding the behavior of epoxy resins is crucial due to their widespread usage in aircraft structures and their sensitivity to ageing caused by environmental conditions. Real-time monitoring is essential in aeronautic structures and two FBG sensors were used to monitor deformations and residual stresses during production and to monitor the strains developed during immersion in a hot-water environment. Tensile tests were performed to assess the mechanical properties, including the impact of moisture on resin performance and the recovery after drying. This work lays the foundation for further exploration of fiber optic sensors in moisture monitoring and highlights their potential for cost savings and enhanced aircraft safety [1].

FBG as humidity sensor

Several reference studies have demonstrated the feasibility of utilizing an FBG sensor as a humidity sensor by coating it with a moisture-sensitive coating. In this manner, when the sensor is exposed to a humid environment, the fiber detects a wavelength shift resulting from the strain induced by moisture absorption. The equation that relates the wavelength variations to the different contributions of relative humidity, mechanical deformations and temperature changes is reported here below:

$$\frac{\Delta\lambda_B}{\lambda_B} = (1-p_e)\varepsilon_M + (1-p_e)\frac{E_cA_c}{E_cA_c+E_fA_f}\beta_c\Delta RH + \left\{(1-p_e)\frac{E_cA_c\alpha_c+E_fA_f\alpha_f}{E_cA_c+E_fA_f}+\xi\right\}\Delta T$$

Where ε_M is the axial mechanical strain, $\Delta\mathrm{T}$ is the temperature change, $\mathrm{p_e}$ is the photo-elastic constant, and ξ is the thermo-optical coefficient. Where E is the Young's modulus, A the crosssectional areas, α the coefficient of thermal expansion, and β_c the constant coefficient of hygroscopic expansion of the polymer. The subscript c stands for the coating, while f stands for the bare fiber. Once decoupled the mechanical, thermal and humidity effects it becomes possible to determine each contribution and monitor the manufacturing and conditioning processes [2], [3], [4], [5].

Production of test specimens

Before facing the testing phase, it was necessary to produce the specimens in compliance with ASTM D638 Standard. Epoxy resin was adopted, since it is a commonly used matrix material in composites due to its excellent adhesive properties, low viscosity, and exceptional mechanical properties. It finds applications in aerospace, automotive, sports, and marine industries. The production process consists of different phases:

- Heat the resin in the oven to make it liquid.
- Place the resin in the vacuum oven performing a degassing process.
- Heat the resin again to reduce its viscosity.
- Cast the resin in the mold using a syringe.
- Cure the resin in the oven.
- Allow the specimen to cool down.

During this process, the strains developed in the specimen were monitored by the FBG sensor, obtaining the plot in Figure 1:

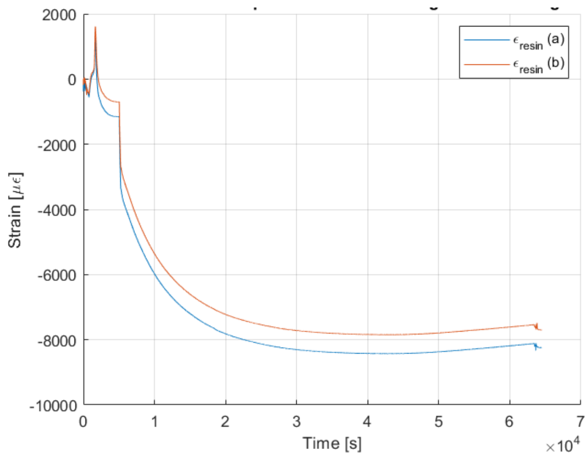

Figure 1: Total strain developed on the resin during manufacturing.

The entire production process caused a compression state-of-stress, leading to a total value of strain close to 8000 micro-strains and a residual internal stress of 25 MPa.

Conditioning process

To reproduce a hot-wet environment and accelerate the moisture absorption process, the test specimens were immersed in an 80°C water bath. The test lasted approximately 1500 hours, during which real-time monitoring of wavelength variations was carried out as shown in Figure 2. By extrapolating the collected data, it was possible to calculate the deformations of the specimens. The strains due to relative humidity shows a rapid increase in the first 100 hours, followed by a sharp decline until reaching a plateau. The non-monotonous behavior could be attributed to various factors, such as the slowdown of the hydrolysis process [6], [7]. The strains obtained in this phase are showed in the figure below:

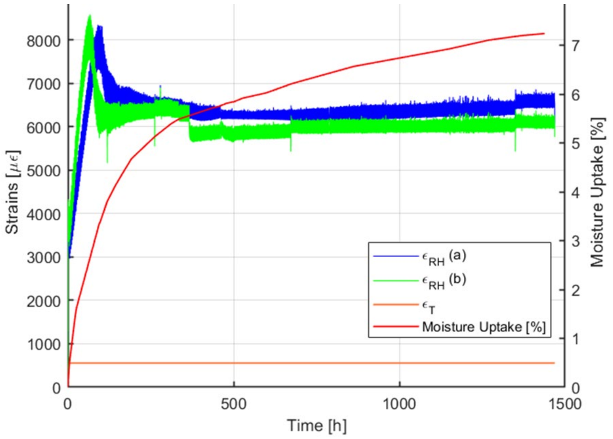

Figure 2: Strain comparison during conditioning.

Mechanical properties

Once having conditioned a series of specimens, their mechanical properties were compared to those characterizing unconditioned specimens and specimens dried after absorption. This was done to observe whether moisture absorption would degrade the material's performance and, if so, whether it could be recovered through a drying process. The figure 3 shows that the moisture absorption causes a degradation of the mechanical properties in terms of Young's modulus and resistance. After te drying process, the material is unable to regain its original properties. In fact, its performance worsens, exhibiting brittle behavior and being prone to premature failure. These tests were carried out using an electro-mechanical tensile machine in compliance with ASTM International Standards [8].

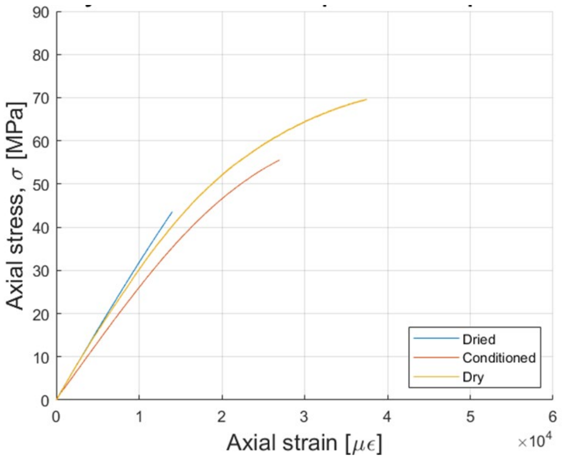

Figure 3: Comparison between dry, wet and dried specimens.

Conclusions

The research describes the technological challenges faced in studying the mechanical properties of epoxy resin. After developing production process to obtain defect-free specimens, tests were conducted to evauate the material's mechanical properties under dry, conditioned, and dried conditions. The specimens immersed in hot water showed a reduction in mechanical properties: -14.3% in Young's modulus, -24% in breakage stress, -31.5% in strain at break. The dried specimens were unable to fully recover their initial characteristics, and they show a further reduction of resistance properties: -37.7% in stress at break, -64.5% in breakage strain. Fiber optic sensors were used to monitor the strains during production and conditioning, showing a good response in detecting the humidity variations. After being immersed for 1500 hours, the fibers were not able anymore to detect deformations during a tensile test because they were damaged.

Further research should investigate the causes of sensor malfunction and study the effect of humid environments and temperature variations on complete composite materials used in the aerospace industry.

References

[1] V. Giurgiuntiu, Structural Health Monitoring of Aerospace Composite, Elsevier, 2016.

[2] P. Giaccari, H. G. Limberger e P. Kronenberg, «*Influence of humidity and temperature on polyimide-coated*» in Bragg Gratings, Photosensitivity, and Poling in Glass fibers and Waveguides: Applications and Fundamentals, Optical Society of America, p. BFB2, (2001). https://doi.org/10.1364/BGPP.2001.BFB2

[3] P. Aceti, «Sensing principles of FBG in humid environments,» Politecnico di Milano, 2023.

[4] Teck L. Yeo, Tong Sun, Kenneth T. V. Grattan, David Parry, Rob Lade, and Brian D. Powell, «Polymer-Coated Fiber Bragg Grating for Relative Humidity Sensing,» *IEEE Sensor Journal,* 2005.

[5] Pascal Kronenberg and Pramod K. Rastogi, Philippe Giaccari and Hans G. Limberger, «Relative humidity sensor with optical fiber Bragg gratings», *Optics Letters,* 2002. https://doi.org/10.1364/OL.27.001385

[6] Pietro Aceti, Luca Carminati, Paolo Bettini and Giuseppe Sala. Hygrothermal ageing of composite structures. Part 1: technical review. Composite Structures, 117076, 2023. https://doi.org/10.1016/j.compstruct.2023.117076

[7] Pietro Aceti, Luca Carminati, Paolo Bettini and Giuseppe Sala. Hygrothermal ageing of composite structures. Part 2: mitigation technique, detection and removal. Composite Structures, 117076, 2023. https://doi.org/10.1016/j.compstruct.2023.117076

[8] ASTM International. Standard Test Method tensile properties of plastics. D638