

UV-CURED FIBER REINFORCED COMPOSITE LAMINATES : A DETAILED STUDY OF PHOTONIC AND CHEMICAL PARAMETERS FOR OPTIMIZED MECHANICAL PROPERTIES

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Abstract

In this work, 6 layers laminates made out of glass/polyester prepregs were manufactured using an UV LED irradiation process and their mechanical properties were determined by measuring ILSS values. Influence of PI concentration has been thoroughly investigated in order to obtain laminates with enhanced mechanical behavior. It has been shown that the TPO concentration does impact the conversion levels of the final composites. There is an optimum concentration value that allows both the generation of enough reactive species to polymerize the sample and which also allows the transmission of light within its thickness to obtain a homogeneously UV-cured composite.

1. Introduction

The emergence of composites in the industrial world has pushed back the technical boundaries of materials science. Light-weighted and though exhibiting interesting chemical and mechanical properties those materials have proven to be of high performances [1]. Their field of applications extends from aeronautic to high-end sports equipment so that the only limitation when seeking for a material with specific requirements appears to be its manufacturing process which implies high costs in a broad sense. Indeed, they are mainly achieved under thermal curing either at elevated temperature, involving high energy consumption, or, at ambient temperature meaning a limited control of the reaction, slow processes and thus high manufacturing costs. Today's economic and environmental issues force us therefore to rethink our way to design composite materials.

Despite many advantages the use of light to manufacture composites parts is very recent and still marginal. UV-curing could overcome many of the technological issues the thermal curing is struggling with as it is a solvent-free technology working at ambient temperature with reaction times lasting minutes rather than hours. The formulations exhibit also an infinite pot life allowing a « cure-on-demand » of the composites [2]. The main limitation of the use of light to manufacture composites is that the photons need to penetrate the material to generate reactive species. It is thus a well-mastered technology when it comes to coatings or thin films but which needs adjustments to be geared to the curing of thick materials. Some works successfully describe the manufacturing of glass-fibers reinforced polymers using UV-curing [3-4] but only a few have been interested in the mechanical properties of the obtained materials [5-7] and even less in a comparison with their thermal equivalents. TPO has been proven to be a suitable photoinitiator for photocomposites in the above-mentioned

works but the influence of its concentration on the conversion levels reached by the resin and on the mechanical properties of the final parts was never investigated. The aim of this work was to determine the influence of the amount of photoinitiator in a 6 layers glass fibers reinforced composite.

2. Materials and methods

2.1. Manufacture protocole

The materials used in this work were an unsaturated polyester resin diluted with styrene acting as matrix for our laminates and unidirectional glass fibers for the reinforcing phase. TPO was used as photoinitiator in our system because of its high absorbtion at 395 nm. The prepregs were manually impregnated with resin in order to respect a ratio of 40% resin and 60% fibers and systematically stored for one night to ensure a good impregnation of the reinforcements by the polyester resin. Each composite is made up of 6 layers of prepregs and put under vacuum for 30 minutes to ensure a good compaction between the layers as shown in Figure 1.

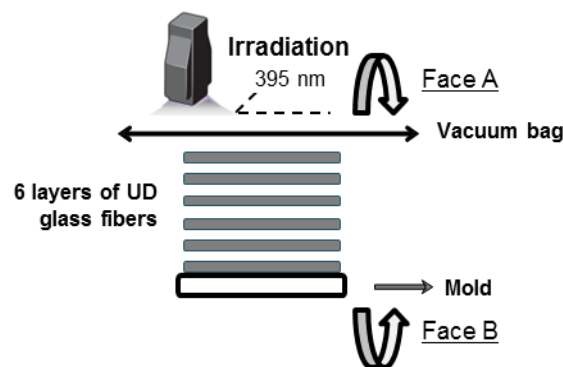


Figure 1: Fabrication of the laminates.

The irradiation source is a LED lamp mounted on a robot arm that scan the composite so that each point of the plate receives the same amount of light. In such conditions irradiation lasts for about 20 seconds and the light dose is 1350 mJ/cm². In what follow we will consider the Face A of the composite laminates which was directly exposed to the light and the Face B that were in contact with the mould.

2.2. Characterization

2.2.1. Conversion values

Conversion values of the laminates were determined using an infrared spectroscopy method: Attenuated Total Reflectance (ATR). Monitoring the evolution of well-chosen peak areas gives us an idea of the conversion value of a composite laminate both on sides A and B. The absorbance peak at 1646 cm⁻¹ is regularly used in the literature as characteristic of the polyester and hence used to measure the extent of a reaction. In our case, however, this particular vibrational band can't be followed because of its very low intensity. Based on the work of Huang and Leu [8], another calculation method had to be set up involving some others vibrational bands. This method allows to determine the polyester conversion and the total conversion of a system from the follow-up of a band which is characteristic of styrene.

2.2.2. ILSS

In order to compare the properties of the different UV-cured laminates that were manufactured in the lab, mechanical tests were to be performed. Interlaminar Shear Strength (ILSS) was chosen as criterion as it is often used in industry for quality control and for comparing material. Mechanical tests were carried out on an Instron 250 kN device under standard lab conditions and according to NF EN ISO 14130 [9].

3. Results and discussion

8 different photoinitiator concentrations ranging from 0.2 to 5% were investigated in this study. The results on the conversion values of the Face A (directly exposed to light) show that increasing the amount of photoinitiator induces higher conversion values. In contrast, the conversion values of the Face B tend to diminish as the concentration of TPO raises. If we consider the absolute difference, for the same plate, between the conversion of the faces A and B we can define a criterion for the uniformity of a plate. The closest to 0 this gap gets, the more homogeneous the plate is. We plotted in Figure 2 these absolute differences at different TPO concentration levels. We add then to this graph the ILSS results.

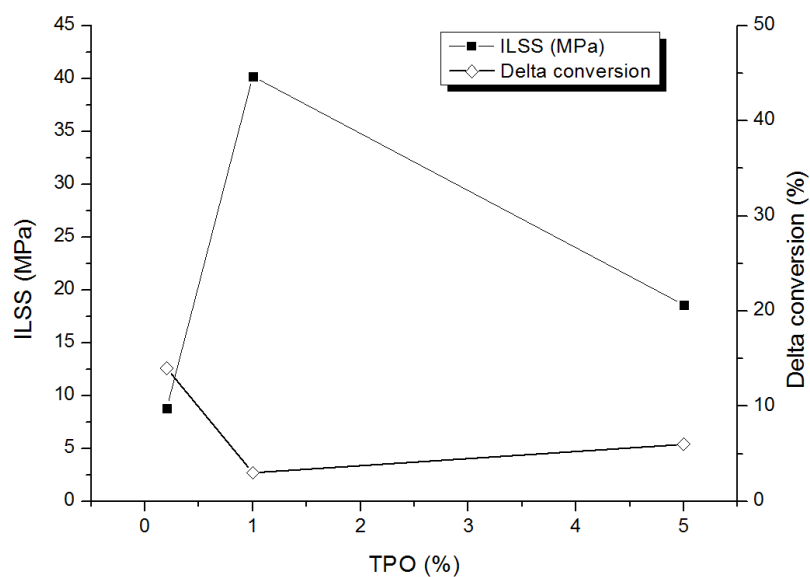


Figure 2: Evolution of both the ILSS values and the conversion gradients of the laminates as a function of TPO concentration

Figure 2 shows that the ILSS results have a Gaussian distribution. At the lowest photoinitiator's concentrations, the mechanical properties are poor. Same occurs at the highest concentrations. In contrast, there is an optimal range of values for which the mechanical properties of the composite laminates are enhanced. On the contrary, the distribution of the absolute differences of the conversion has the opposite shape, which means that the better composites in terms of ILSS are those with the lowest gradient of conversion within their thicknesses.

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References

- [1] Shaffer, J.; Philippidis, T. P.; Vassilopoulos, A. P., Polymer Composites. In Handbook of Advanced Materials, John Wiley & Sons, Inc.: 2004; pp 1-63
- [2] Endruweit A, Johnson MS, Long AC. Curing of composite components by ultraviolet radiation: A review. *Polym. Compos.* 2006; 27(2): 119-128.
- [3] Hussein MA. Photo-fabricated unsaturated polyester resin composites reinforced by kenaf fibers, synthesis and characterization. *J. Appl. Polym. Sci* 2012; 123 (2): 968–976.
- [4] Shi, Wenfang, et Bengt Rånby. « UV Curing of Composites Based on Modified Unsaturated Polyester ». *Journal of Applied Polymer Science* 51, no 6 (s. d.): 1129-39.
- [5] Crivello JV, Narayan R, Sternstein SS. Fabrication and Mechanical Characterization of Glass Fiber Reinforced UV-Cured Composites from epoxidized Vegetable Oils. *J. Appl. Polym. Sci* 1997; 64 (11): 2073–2087
- [6] Compston P, Schiemer J, Cvetanovska A. Mechanical properties and styrene emission levels of a UV-cured glass-fibre/vinylester composite. *Compos.Struct.* 2008; 86 (1-3): 22-26.
- [7] Zhang X, Duan Y, Zhao X, Li D. UV stepwise cured fabrication of glass fiber/acrylate composites: Effects of exposure dose on curing uniformity and interlaminar shear strength. *J. Compos. Mat.* 2015.
- [8] Huang, Yan-Jyi, et Jiing-Shing Leu. « Curing of unsaturated polyester resins. Effects of temperature and initiator: 1. Low temperature reactions ». *Polymer* 34, n° 2 (1 janvier 1993): 295-304.
- [9] ISO14130