EFFECT OF THICKNESS ON THE INTERFACIAL STRENGHT OF LAYER BY LAYER IN SITU UV CURING

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Abstract

Ultraviolet curing (UV) of composite materials is faster than the conventional thermal curing approaches. Because of the absorption of radiation passing through the composite, the thickness of laminates is limited to a maximum depth of between 8 mm and 13 mm in one shot. However, there are several manufacturing processes in which successive thin layers are placed and can be in situ cured by UV radiation. In the present paper the validity of the layer by layer in situ UV curing processing concept for glass reinforced polyester thick composite has been validated. A comparison of the mode II interlaminar fracture toughness, G_{IIC}, between a bulk UV cured composite and a two-step UV cured composite has been obtained using the four point end notched flexure (4ENF) specimen. The experimental results have shown that crack propagation takes place earlier at the two-step UV cured composites, and, no significant difference of G_{IIC} has been found varying the curing step thickness.

1 Introduction

The high operational costs involved in combination with the intricacy of the manufacturing techniques currently employed have restricted wider industrial use of composites. For these reasons, considerable effort has been made in the direction of finding and developing alternative cost-effective routes for manufacturing composite materials [1]. Some of such alternative routes are advanced resin transfer molding [2], fluid-heated and balanced pressure floating mould process [3], microwave curing [4], electron beam curing [5] and ultraviolet (UV) curing [6-9]. Thermoset resins such as vinylester [7], epoxy [8] and polyester [9], when formulated with photoinitiator, can be cured in minutes through exposure to UV light, and they can exhibit similar mechanical properties to their thermally cured counterparts. The increased speed of cure possible through UV curing and the fact that the curing starts on the component surface, can also reduce the emission of volatile organic compounds (VOCs) from the resins. Furthermore, as they have an indefinite pot life, the characteristics of the resin system allow complete impregnation of the fibre without premature gelation. Thus, the scrap rate is reduced and the excess resin can be collected and put back in the container for reuse. UV energy can only penetrate optically transparent materials, and because of the absorption of radiation passing through matter [10], the thickness of laminates for efficient applications of UV curing is limited to a maximum depth of between 8 mm and 13 mm in one shot [6].

However, there are several manufacturing processes (automated tape laying (ATL), automated fibre placement (AFP) and filament winding) in which the composite is not cured as a bulk, but successive thin layers are placed and can be in situ cured by UV radiation, overcoming the depth-of-cure constraints. The interlaminar shear strength and fracture characteristics of the UV cured secondary bonded composites are in some cases similar to those of thermally cured ones [7, 11]. However, to our knowledge, the mechanical functionality and the processing route of such layer by layer in situ UV cured composites has not been published yet in the literature. Therefore, the evaluation and the prediction of the failure modes and mechanical properties of UV cured layered structures is necessary to validate the material and also the fabrication process. Furthermore, the interlaminar fracture is one of the most commonly failure mode for composite materials, and most markedly in layered structures. Analyzing the standard test methods for the evaluation of the mode I interlaminar fracture toughness, the double cantilever beam (DCB) test method [12-15] is an international standard which permits the correct evaluation of mode I interlaminar fracture toughness. However, there is not an international consensus about the correct testing method to evaluate mode II fracture testing. The most widely used test is the end notched flexure (ENF) specimen. The most important disadvantage of that testing method is its crack propagation, which is very unstable [16-17]. Another test method to analyze the interlaminar strength is interlaminar shear strength (ILSS) method. The main disadvantage of ILSS is that many times the fracture doesn´t take place as an interlaminar failure. An alternative for mode II configuration was proposed by Martin and Davidson [18] which is called four point end notched flexure (4ENF). This method permits stable crack propagation and using the compliance calibration technique [19] the mode II fracture toughness can be determined. Some works has been published around mode II, most of them using carbon fibre [20, 21], but also around glass fibre reinforcements [22].

In the present paper the validity of the layer by layer in situ UV curing processing concept for glass reinforced polyester thick composite has been validated. In such manufacturing process the strong adhesion between successive layers is the key point for high performance composites. Thus, is important to evaluate the influence of the layer thickness on the interlaminar strength due to the absorption of the UV radiation trough the material. A comparison of the of the mode II interlaminar fracture toughness, G_{IIC}, between a bulk UV cured composite and a two-step UV cured composite has been obtained using the 4ENF specimen. Two composites with different thickness have been manufactured and characterised. The thinner one is a 10 layer laminate, whereas the thicker has 18 layers.

2 Experimental

2.1 Materials

The material used in this study is a UV cured glass/polyester composite. The reinforcement consists of 300 g/m^2 quasi unidirectional E-glass ribbon. The reinforcement is described as quasi unidirectional because of the small proportion of fibres of 90º which maintain the cohesion of the unidirectional fibres. The resin was UV curable unsaturated polyester supplied by Irurena S.A. The fabrication process used for the development of the test specimens was UV cured resin infusion. The lamp used was a flood type lamp provided by DYMAX with an intensity of 75 mW/cm^2. The pre-crack was made by inserting a 12.5 μm Teflon FEP 50A film, supplied by DuPont. This film was inserted at mid-thickness during the fabrication of the specimens, so in this way, all the specimens will have a starter crack to initiate the delamination.

Two composites with different thickness have been manufactured. The thinner one is a 10 layer laminate, whereas the thicker has 18 layers. In the same way, two different manufacturing methods have been used in the fabrication of those specimens: two step UV curing and bulk UV curing. Thus, 3 different types of specimen were manufactured (Table 1): two of them with 18 plies, one cured as bulk and the other one cured in two steps; and another one with 10 plies, cured in two steps also. Five specimens of each type were manufactured.

Specimen	Number of layers	Thickness	Curing method
9+9	18	4.61 ± 0.03	2 step (9+9) UV cured
Bulk 18	18	4.37 ± 0.15	1 step UV cured
5+5	10	2.63 ± 0.11	2 step (5+5) UV cured

Table 1. Manufactured specimens

2.2 Test geometry and procedures

As it is said before, the test used for the determination of the mode II fracture toughness is the 4ENF. A diagram of the 4ENF test is presented in the Figure 1. The mode II test fixture consists on an upper loading part with a central pin 24 mm above the two loading pins. This distance is less than the maximum value of 50 mm, according to the 4ENF protocol [19]. The specimen is supported at two points, with a separation of 2L=100 mm. The initial crack length (a_0) is 35 mm, measured from the left outer loading point.

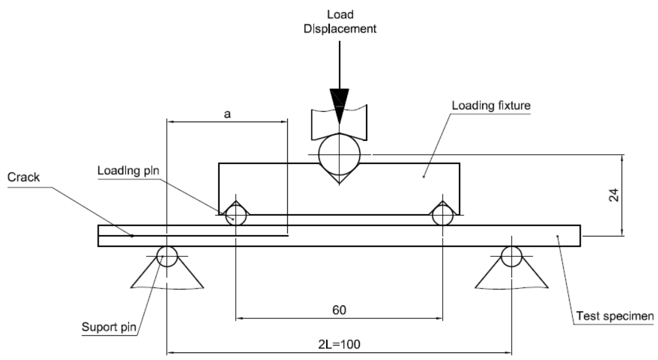


Figure 1. 4ENF test method for measuring mode II interlaminar fracture toughness (dimensions in mm)

The geometry of the manufactured specimens is shown in Figure 2. All specimens are rectangular, 140 mm long and 20 mm wide. The thickness of each specimen is directly proportional to the number of plies used in the manufacturing process.

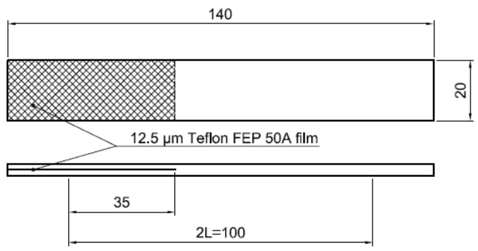


Figure 2. 4ENF specimen geometry in mm

All tests were performed at displacement rate of 1 mm/min and using a 5 kN capacity load cell. The followed testing procedure is the same that used in all previous studies carried out on 4ENF [19-22]. This consists on loading the specimen until crack advance starts, and as the crack advances, periodically determining the crack length form visual measurements until the crack approaches the right outer loading point. During the test, load and deflection values were recorded and continuously relating the deflection and crack length values in order to generate the compliance (*C*) *vs. a* curve, defining the compliance as the ratio of maximum displacement over the maximum load for each specimen. Figure 3 shows a deformed specimen during the mode II test.

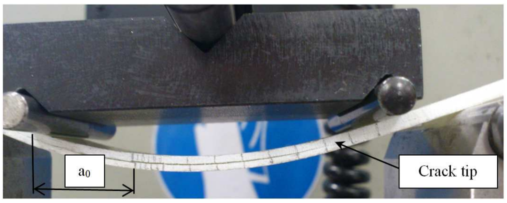


Figure 3. 4ENF specimen deformation during the test

2.3 Data reduction for G_{IIC}

The method for the determination of the mode II interlaminar fracture toughness, G_{IIC}, is the experimental compliance calibration method with the compliance expression [19]:

$$C = C_0 + ma \quad (1)$$

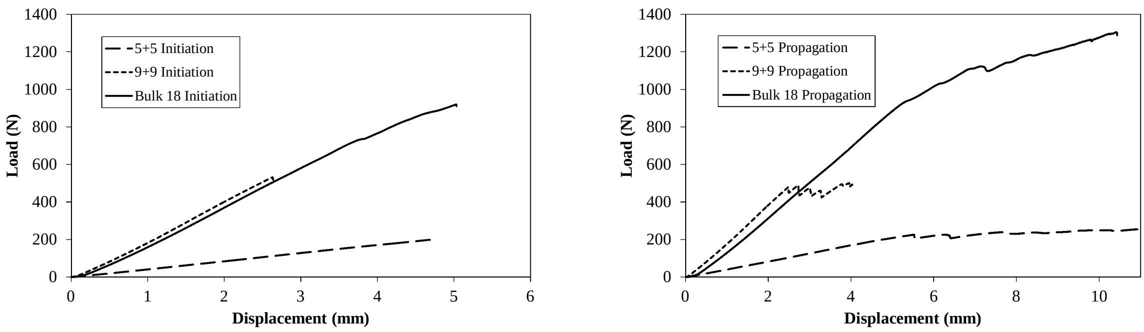
where *C* is the specimen compliance and a is the crack length. The fracture toughness, G_{IIC}, is:

$$G_{IIC} = \frac{P_C{}^2 m}{2B} \quad (2)$$

where P_C is the critical load, *m* is the slope of the compliance *vs.* crack length plot and *B* is the specimen width. For propagation, the critical load can be taken as the average of all P_{max} values to get an average fracture toughness for the specimen.

3 Results and discussion

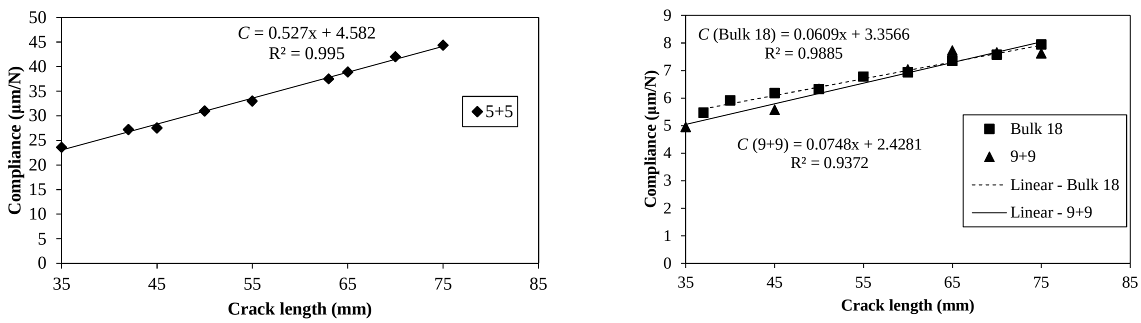
Three mode II specimens of each type described in Table 1 were tested. In Figure 4 an example of load-displacement plot of each type of specimen is shown. The initiation test (Figure 4a) consists on loading the specimen until the crack propagation starts, and the propagation (Figure 4b) test is a second loading to propagate the crack. Those plots indicate that all the specimens have linear behaviour until the crack initiation starts. Comparing bulk 18 and 9+9 specimens is remarkable that they have similar stiffness in both tests, whereas the two step cured specimens have lower critical load.



a. Initiation b. Propagation

Figure 4. Example of load-displacement plots for each specimen

Besides the difference of the maximum critical load, the most remarkable difference between behaviour in 4ENF test of one step and two step cured composites is how the crack grows during the propagation test. Whereas the crack propagation is slow and progressive in bulk cured composites, the two step cured composites present different behaviour: the crack propagation is more unstable and is given by leaps. This is because of the presence of an interface at mid-thickness of the two step cured composites that allows the easier propagation of the crack tip. Is important to note that the conversion factor in the first step of curing in two step curing composites is 100%, so, the adhesion conditions between curing first and second steps are the worst to reach G_{IIC} high values. Figure 5 presents the typical plots of compliance versus crack length obtained from the 4ENF test of each specimen type. The small deviations from the linear lines may be caused by the errors of defining the crack tip position. Figure 5b shows that the slopes of the compliance versus crack length plots of 9+9 and Bulk 18 specimens are nearly the same. Analyzing the 5+5 specimen (Figure 5a), as the stiffness is lower than the other specimen types, the slope of the compliance versus crack length plot is higher.

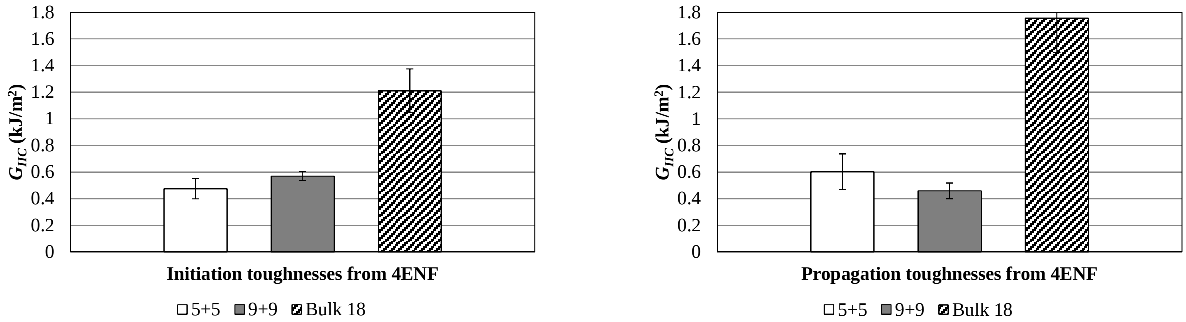


a. 5+5 specimen b. 9+9 and Bulk 18 specimens

Figure 5. Compliance (*C) vs.* crack length (*a)* curves from 4ENF specimens

Fracture toughness, G_{IIC}, determined from 4ENF test for 3 type of specimen manufactured is shown in Figure 6 (the initiation 5a and the propagation 5b). Interlaminar fracture toughness is substantially higher in bulk cured composites than in two step cured composites. This effect may be due to the adhesion interface at mid-thickness of two step cured composites. Remind that the conversion factor of the first curing step was 100%. Further investigations are needed to determine the impact of the first curing conversion factor in mode II the fracture toughness.

Analyzing the bulk cured composites, it is clear that the propagation toughness is notably higher than initiation value; this may be because of the absence of an adhesion interface at mid-thickness which hinders the growth of the crack. In the case of two step cured composites, fracture toughness is lower, but nearly similar for both specimen types. This would be because of the adhesion interface at mid-thickness and the 100% conversion factor in the first curing step. But, this behaviour can be an advantage in case of impact solicitations. The lower fracture toughness causes an easier growth of the crack, and in the case of an impact, this may result in a bigger delaminated area, and consequently, in a higher quantity of dissipated energy. Another conclusion is the fact that there is not notable difference of fracture toughness value between 5+5 and 9+9 specimens, so it can be said that the intensity of UV light that reaches the adhesion interface doesn´t affect in mechanical properties in this range. To determine this range of intensity, a study of transmittance has been made.



a. Initiation b. Propagation

Figure 6. Initiation (a) and propagation (b) toughnesses from 4ENF

The light absorbance trough a material is represented by Beer–Lambert equation [23]: the value of the light intensity that reaches the lower surface (I_{TRANS}) decreases exponentially with the thickness (t) of the material. For the study of transmittance, the light intensity of the directly non-emitted surface has been measured using Hamamatsu C6080-13 photometer. Figure 7 shows the transmittance of the material along tested thicknesses. It determines that for a transmitted intensity (I_{TRANS}) < 4 mW/cm^2 the fracture toughness is nearly constant.

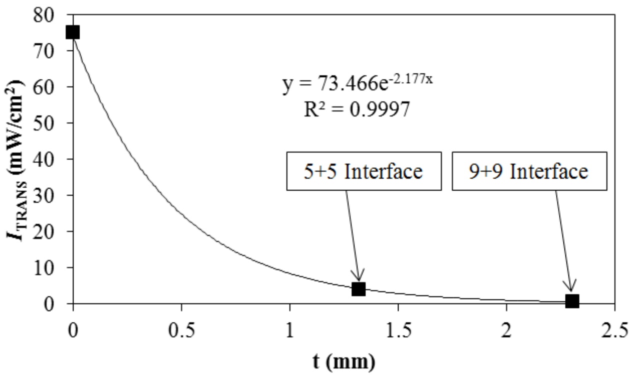


Figure 7. Transmittance

Examples of mode II interlaminar toughness versus crack length curves are shown in Figure 8. For the two step cured composites G_{IIC} value is nearly constant, with a small increase in case of the 5+5 specimen. However, bulk cured composites show higher increase of the G_{IIC} value during the crack propagation, apart from higher interlaminar toughness as it has been determined before.

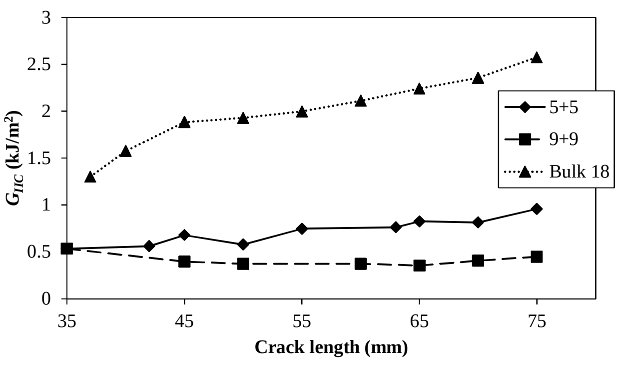


Figure 8. G_{IIC} *vs.* crack length for the specimens

4 Conclusions

In the present paper the validity of the layer by layer in situ UV curing processing concept for glass reinforced polyester thick composite has been validated. The influence of the layer thickness on the interlaminar strength due to the absorption of the UV radiation trough the material has been studied. A comparison of the of the mode II interlaminar fracture toughness, G_{IIC}, between a bulk UV cured composite and a two-step UV cured composite has been obtained using the 4ENF specimen. Following conclusions are reached.

- Interlaminar fracture toughness, G_{IIC}, is substantially higher in bulk cured composites than in two step cured composites. This effect may be due to the adhesion interface at mid-thickness of two step cured composites. This behaviour can be an advantage in case of impact solicitations. The lower fracture toughness causes an easier growth of the crack, and in the case of an impact, this may result in a bigger delaminated area, and consequently, in a higher quantity of dissipated energy.
- During the manufacturing of two step cured composites, the conversion factor of the first curing step was 100%, so the adhesion conditions were the worst to reach G_{IIC} high values. Further investigations are needed to determine the impact of the first curing conversion factor in mode II the fracture toughness.
- Analysing the light intensity that reaches the directly non-emitted surface, it has been demonstrated that the interlaminar fracture toughness, G_{IIC}, is nearly constant for a transmitted intensity (I_{TRANS}) < 4 mW/cm^2 (measured with Hamamatsu C6080-13 photometer).

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