A STUDY ON FATIGUE BEHAVIOR OF NANOINTERLEAVED WOVEN CFRP

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Keywords: Fatigue, Delamination, Composite Material, Crack Grow Ratio, Nanofibers

Abstract

Interleaving nanofibrous mats between layers of composite laminates has been proved to be an effective method for improving composites delamination resistance. The present work aims to investigate the effect of interleaving a nanofibrous mat on fatigue delamination properties of mode I loaded carbon-epoxy composite woven laminates. Double Cantilever Beam (DCB) virgin and nanomodified specimens were fabricated, these latter by interleaving a 40 micron thick mat of Polyamide nanofibers in the mid interface, where the crack was initiated. Static and fatigue tests were performed in order to determine the delamination growth onset and the crack propagation rate vs maximum energy release rate respectively. Static tests showed an increase of delamination toughness for nanomodified specimens of 130%. Crack grow ratio charts for virgin and nanomodified specimens showed that for a given propagation speed nanomodified specimens can sustain a much higher load than the virgin ones; furthermore, nanomodified specimens presented a several times slower crack propagation speed compared to the virgin ones, for a given stress.

1. Introduction

Composites are now established as top materials for those applications requiring high performances and reduced weight, from sport cars to space shuttles, for both structural and aesthetic purposes.
Composites have been widely investigated in the last decades and many researches have been done to

overcome delamination problems [1]. Delamination is due to interlaminar stress and/or strain, which causes two consecutive plies to debond. A composite laminate delaminated can remain visibly unchanged while losing up to 60% of its stiffness [2]. Many efforts have been done in the past to solve or mitigate the problem of delamination [3–10]. The authors have already used polymers [11–13], and in particular Nylon 6,6 [14–17], to produce nanofibers to be interleaved between layers, obtaining very positive results. In particular, it has been found that the nanofibers are able to link the plies they are inserted in, giving additional strength when the matrix around is broken [18].
Despite the large amount of research on nanomodified composite, to date very few works have been found dealing with cycling loads [19–22]. Bortz et al. [20] fatigue-tested fully compressive, tensile and tensile dominated loading, showing 150-670% improvement in composites interleaved with carbon nanofibers due to the high interface density and damage shielding effect of the reinforce within the matrix. Arai et el. [21], investigated Mode I fatigue propagation and found out that with the application of multi walled nano tubes (MWNT), the number of cycles to failure and the fatigue fracture toughness increase of 150 and 300% respectively. Zhou et el. [22] obtained similar results in both static and fatigue tests. Shivakumar et al. presented a methodology to assess total fatigue life for mode I delaminated composite laminates [23] and then investigated nanomodified laminates [19]. They showed that the nanofibers increased the delamination onset life, and increased the fatigue threshold energy release rate by two-thirds. A Paris-law like for nanomodified composite laminates is a lack in the literature, and it is the void this work aims to fill.
Polymeric Nylon 6,6 nanofibers, produced by electrospinning, have been interleaved in epoxy based-carbon fiber woven composites: non nanomodified (V) and nanomodified (Ny) specimens have been then tested under Double Cantilever beam (DCB) fatigue test; the Strain Energy Release Rate (SERR) [24] method has been used to determine and compare the constant crack growth section of the Paris law diagram for the two configurations.
Results showed that the interleave significantly reduces the crack propagation speed, for a given stress, and that, on the other end, that for a given speed, nanomodified samples can carry on significantly higher load compared to static specimens.

2. Material and methods

2.1. Sample preparation

130 *mm* x 20 *mm* DCB specimens were manufactured according to the ASTM D5528 [25] stacking 14 plies of plain weave (PW) 220 *gcm* carbon-epoxy prepreg (GG204P-IMP503Z), supplied by Impregnatex Composite Srl (Milan, Italy); Nylon 6,6 Zytel E53NC010, provided by DuPont, were used for producing nanofibers by means of electrospinning (see Figure 1a) as presented in [13]. 400 to 650 *nm* diameter nanofibers were randomly aligned in 40 μm thick mats, with areal density of 18 g/m^2. Nanofibers were kept in oven at $40^o C$ overnight, before integrating it into the laminate, to remove residual solvents and the moisture absorbed by the Nylon. Nanomodified specimens were interleaved with the nanofibrous mat as presented in [13].
Laminates were 3.5±0.1 *mm* thick with no appreciable differences between configurations.
Virgin and nano-interleaved specimens were cut from two rectangular panels: a 30 μm thick PTFE film was inserted in the mid-interface during the lay-up to create an initial artificial crack which length a_0 was 45 *mm*. Both panels were vacuum bag moulded following an optimized cure cycle in order to ensure the perfect penetration of the resin into the nanofibrous mat, before the hardening occurs. In particular the maximum temperature was $130^o C$, below Nylon's melting temperature at $260^o C$. Loading steel blocks were glued to the specimens for the application of the load (see Figure 1b).

(a) Electrospinning equipment (b) DCB specimens

Figure 1: Electrospinning and testing equipment

2.2. Static Test

Static DCB tests have been done to determine the critical (G_{IC}) and the propagation (G_R) energy release rates. Three specimens were extracted from each panel, and results averaged. The tests have been carried out in a servo-hydraulic press machine (Instron 8033) equipped with a 250 N load cell, under displacement control condition, at a constant crosshead speed of 3 mm/min. During the test, the load-displacement curve was recorded and the crack propagation was visually determined by means of optical microscope. The Mode I energy release rate was determined according to the Modify Beam Theory (MBT), as suggested by the [25].

2.3. Fatigue tests

Samples for fatigue tests have been extracted from the same batch of those for static tests, therefore they had the very same dimensions and mechanical properties. Once manufactured, the specimens have been stressed until the crack started to propagate, in order to create a sharper and naturally developed crack within the specimens. The effective crack lengths were therefore 1 mm circa longer compared with the initial status.

The critical energy release rates calculated with the static tests have been used to determine the maximum displacement of the fatigue tests, which started at an initial $G = 0.85 \cdot G_{IC}$

Outcome of the dynamic experiments will be the $da/dN - dG_{max}$ curve for V and Ny configurations. da/dN is the propagation speed of the crack (a), and G_{max} the energy release rate of the material calculated at the peak load of the cycles.

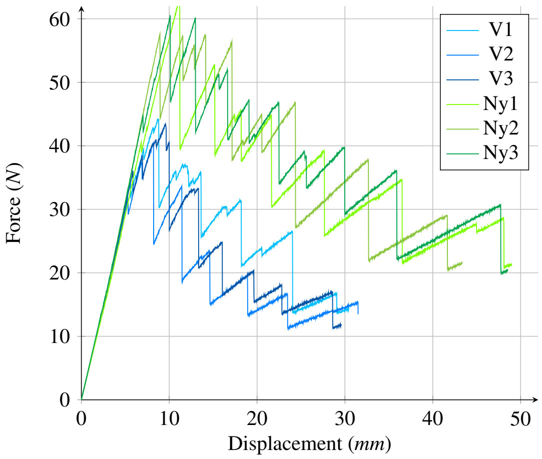
Fatigue tests were carried out following the guidelines provided in the ASTM D6115 [26] in the same Instron 8033 servo-hydraulic machine used for the static ones, equipped with a dedicated, in-house designed and manufactured 200 N load cell, under displacement control condition, at a load frequency of 3 Hz, with fixed loading ratio R ($\delta_{min}/\delta_{max}$) equal to 0.3, 500k cycles.

Peak-and-valley values of load and displacement have been recorded at each cycle. An high-resolution camera pointing the specimen's edge is used to visually measure the crack length during the tests.

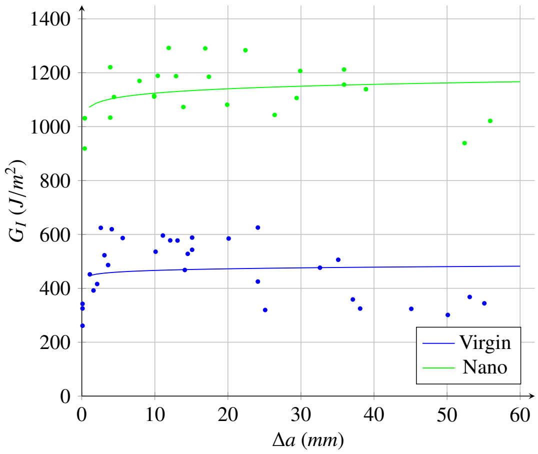
The calculus of the crack propagation rate followed the method proposed in [27]. $a(N)$ is determined through the compliance method and then discretely derived to obtain $da/dN(N)$. Load-displacement and crack length values are used to calculate G_{max}. Eventually the log-log plot for $da/dN(G_{max})$ is obtained.

3. Results

Figure 2 summarises the results of the static tests. Force-Displacement curves and R-Curves are shown in Figure 2a and 2b respectively



(a) Force-Displacement curves of static tests.



(b) R curve for static tests.

Figure 2: Static test results.

Figure 3 shows crack propagation speed vs. maximum energy release rate for each specimen.

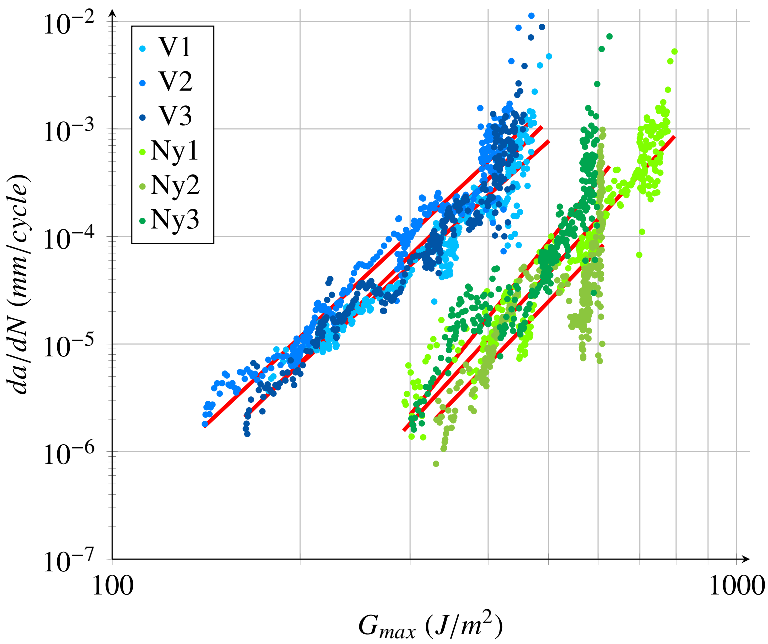


Figure 3: da/dN curves for V and Ny specimens

The two configurations are well clustered for both the tests, and the effect of the interleave will be discussed in the next section.

4. Discussion

The complete set of the results of static tests are published in [13]. It is worth mentioning here that that specimens with Nylon 6.6 nanofibrous interleave, showed a 137% and a 124% increase of G_{IC} and G_R respectively, compared to pristine ones.
For the fatigue experiments, the stable-growth propagation phase of the Paris law can be expressed with the power function1 [28].

$$\frac{da}{dN} = A \cdot {G_{max}}^n \tag{1}$$

where A and n are determined by the curve fitting on the experimental data. Drawing equation 1 in a log-log plot like that in Figure 3, results in a straight line, coloured in red in the figure. n is the slope of the line and represents the sensibility of the crack propagation speed to the energy release rate: it is equal to 5.25±0.22 and 6.32±0.62 for V and Ny specimens respectively.

On the other end, the plot shows that the ratio of the crack propagation speeds between V and Ny configuration span from 34.4 to 20.8, in the G_{max} range common at the two da/dN. It indicates that the crack in virgin specimens propagates much faster than that in Ny ones, for any given G_{max}. For example, at a given $G_{max} = 400\ J/m^2$, crack propagation speeds are $2.98 \cdot 10^{-4}\ mm/cycle$ and $1.15 \cdot 10^{-5}\ mm/cycle$ for V and Ny samples respectively, meaning that cracks in Ny interfaces propagate 26 times slower than those in virgin interfaces. If the curve would consider an extended domain ranging from the minimum value for the V specimens to the maximum of the Ny, then the ratio would span between 68 and 15.
In the same way it can be said that for a given propagation speed, Ny samples can carry on a much greater load than the virgin ones. For example, for a given crack propagation rate of $5 \cdot 10^{-5}\ mm/cycle$, V and Ny loads are 281 and 510 J/m^2 respectively.

5. Conclusion

Results of an experimental campaign have been here presented. Static and fatigue DCB tests on virgin and nanomodified composite laminate woven samples have been performed to assess the effectiveness of an electrospun mat interleaved into the delaminated interface.
Static tests registered an increase of 137 and 124% of critical and propagation energy release rate respectively when the nanofibers are used. Coherent results have been found from dynamic tests, which showed that for the reference G the crack in a nanomodified interface propagates several times slower than in a virgin one; it is also showed that for the given crack propagation rate, nanomodified samples can carry on much higher load.

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