NANOFIBER INFLUENCE ON LOW VELOCITY IMPACT AND ON VIBRATIONAL BEHAVIORS OF COMPOSITE LAMINATE PLATES

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Abstract *Low-velocity impact (LVI) and bump tests were performed on CFR-Epoxy composite laminates which plies were interleaved with electrospun nylon nanofibers. Bump tests are performed before and after the impact event. Nanosheets are placed in between plies interfaces during the hand lay-up process for specimen manufacturing. Two different configurations of the nanomodified laminate are produced. In the present paper the following three studies were developed: (i) the investigation about the vibration behavior of nano-interleaved composite laminates, (ii) the study the low velocity impact behavior of the nano-interleaved laminates, (iii) the investigation about post impact vibration behavior of the nano-interleaved laminates. All experimental tests were done by comparing virgin and nano-interleaved composite laminates. From the experiments a significant effect of nano-interleaves on the global behavior of laminates was found. Nanofibers are able to increase the absorbing energy capabilities of laminates and strengthen them after the LVI tests.*

1. Introduction

Carbon fiber reinforced plastic (CFRP) materials are widely used in many modern applications due to their high mechanical properties combined with low specific weight. These characteristics makes them ideal materials for aerospace, automotive, shipbuilding and many more applications. Because of their nature, laminate interlayers are one of the weak point. Physical discontinuities and mismatch in mechanical properties, such as stiffness, between adjacent layers are often principle of fails like delamination, buckling and so on. Over the years, many techniques are developed to mitigate the damage related to these phenomena. The most significant approaches proposed in literature to overcome the interlayers failure are based on the matrix toughening [1, 2], on the optimization of stacking sequence [3, 4], on the laminate stitching [5], on the braiding [6], on the edge cap reinforcement [7]. In 1999 Dzenis and Reneker [8] proposed and studied the usage of polymeric nanofibrous electrospun mats as an interleaving material to be placed between composite plies. After the pioneering idea of Dzenis and Reneker several studies have been devoted to the usage of polymeric electrospun nanfibrous mat to develop new composite material and to improve the mechanical performances of composite laminate [9–13]. From the literature survey it has been observed that the insertion of nanofibrous mats does not affect the total laminate thickness, but at the same time it may significantly affects the delamination resistance of laminate materials due to improved interlaminar strength. A nanofibrous sheet, placed on the ply-to-ply interfaces of a composite laminate, influences the distribution and the magnitude of shear stresses acting at the laminates intrefaces. Consequently it is expected that a nanofibrous sheet can also affect the vibrational behavior of the nano-interleaved laminates. In the present work we investigated the effect of interleaved electrospun nanofibrous mats to the vibrational behavior of the composite plate, to the response of the plate when subjected to low-velocity impact, and to the post-impact vibrational behavior of the same plates. Experimental campaign is performed on virgin and on nanomodified specimen subjected to low velocity impact (LVI) and bump test. LVI tests are performed to evaluate the effect of the nanofibrous mats to the laminate impact response in terms of energy absorbing capability, maximum load and rebound velocity. Bump tests are used to determine the vibrational behavior of the nano-interleaved laminates before and after the damage induced by the LVI.

2. Materials and methods

2.1 Specimen manufacturing

10-layers of *GG205IMP50* (kindly provided by Impregnatex Compositi S.r.l.) woven prepreg carbon fiber-epoxy resin composite plates were manufactured to perform the experimental campaign.

Nanofibers were manufacture by means of electrospinning process: polymer used was Nylon 6,6 $Zytel^{®}$ E53 NC010 kindly provided by DuPont and the solution was made of Formic Acid and Chloroform purchased by Sigma Aldrich, used without further purification. Nylon 6,6 was dissolved in a Formic Acid/Chloroform solvent (50:50 v/v) at a concentration of 14$\%$ w/v. Electrospun non-woven mats were fabricated by using an in-house made electrospinning machine designed for the mass production of electrospun mats. The electrospinning process was carried out under the following conditions: applied voltage 22-26 kV, feed rate 0.3 mL/h per nozzle, at room temperature and relative humidity RH = 40÷50$\%$. Electrospun non woven mats were 25±8 μm thick and kept under vacuum over P_2O_5 at room temperature overnight to remove residual solvents. Fiber diameter distribution (150±20 nm) was determined by measuring 200 fiber diameters with an image acquisition software (EDAX Genesis). Thermal properties of Nylon 6,6 electrospun mat were investigated by means of differential scanning calorimetry (DSC) using a TA Instruments Q100 DSC equipped with the LNCS low-temperature accessory.

Panels were fabricated by hand lay-up process, stacking ten plies with fiber directions parallel to the laminate borders. During such a process, nanofibers were placed into selected interfaces: two different nanomodified configurations were manufactured, as summarized in Figure 1. After lay-up panels were cured in autoclave according to specifications provided by the prepreg supplier. Final thickness was 2.27 ± 0.13 mm. The presence of nanofibers into laminates did not cause a measurable increase in plate thickness.

According to the scheme in Figure 1 specimens were impacted at the upper side. Configuration *nanomodified 1* is chosen because of symmetry. *Nanomodified 2* configuration presents all nanofibrous mats on the lower interfaces of the laminate. These choices have been done according to the results summarized in [14] where it si reported that, in case of low velocity impact of thin laminates the delamination cone starts from the inside of laminate and propagate up to the non-impacted side. For a better evaluation of the effect of the nanofibers, they are placed in the zone where the delamination is expected to start and to propagate. All the specimens, virgin and

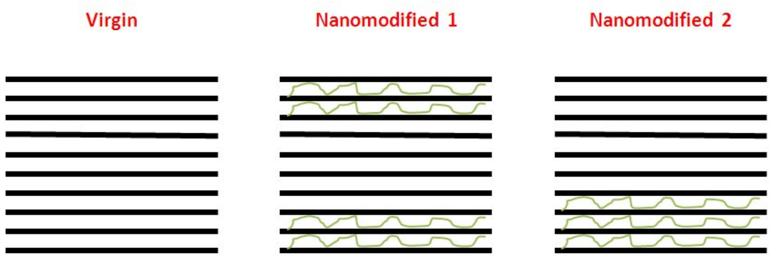


Figure 1: Virgin and nanomodified test conditions. Black lines represent specimen's layers, green lines individuate nanofibers.

nanomodified, are manufactured in a single autoclave process. For each laminates configuration in Figure 1 six specimens were manufactured. The number of specimens was determined by the two levels chosen for the impact tests. So, for each impact energy level three specimens were available.

2.2 Mechanical tests

Bump tests were performed to obtain the natural frequencies of the plates that were clamped in one-edge. LVI tests were performed following the ASTM D7136 [15].

2.2.1 Bump tests

Bump tests were carried out by an external solicitation that was provided by a steel hammer. Two strain gauges were placed at the upper surface of the plate. The strain gauges measured the strain of the external skin of the plates during the bump test and they enabled the calculation of the harmonic frequencies of the plates by a Discrete Fourier Transformation. Measure chain was made of strain gauge powered by a P-3550 Digital Strain Indicator; signal was then digitalized by an analogical to-digital acquisition device (NI9215). Signal was then acquired in a personal computer by a Labview executable program. Sampling frequency was 10 kHz and acquisition time was 6 seconds long. Two harmonic frequencies were extracted from the signal of each strain gauge.

2.2.2 LVI tests

LVI tests were carried out in a drop-weight machine equipped with a laser device able to determine position of the impactor; a piezoelectric load cell on the tip of the impactor measures contact force history. Load cell is provided by an hemispherical 12.7 mm diameter head. Multiple collisions were avoided by means of an electromagnetic braking system. A detailed description of the machine can be found in [16]. The impactor mass was 1.22 ± 0.01 kg; two drop heights of 0.25 and 0.5 m were chosen, corresponding to a nominal potential energy of 3 and 6 J, respectively. Laminates were placed in a clamping fixture which consisted of four rubber pins. Data acquisition was made using the same NI9215 acquisition devices used for bump tests. Load cell and laser signals were acquired at the sampling frequency oh 100 kHz without any filtering except the intrinsic one due to the measurement chain.

3. Experiment results

In the following sections all the mechanical experiments are presented.

3.1 Pre-impact bump test

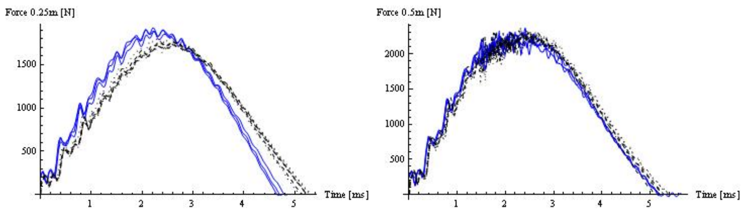
The first 2 harmonic frequencies for flexural modes are detected by Fourier Transform for each specimen. Results are shown in Table 1.

	Virgin		**Nanomodified 1**		**Nanomodified 2**	
	μ	σ	μ	σ	μ	σ
$f_1\ [Hz]$	127.1	0.6	108.2	1.6	108.4	4.2
$f_2\ [Hz]$	769.8	18.8	720.2	9.7	688.8	12.00

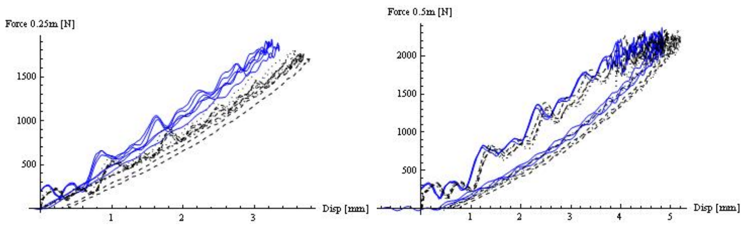
Table 1: Pre-impact harmonic frequencies. μ: mean value, σ: standard deviation.

3.2 LVI test

LVI experimental results are shown in figure 2. In particular the Force-Time and Force-Displacement diagrams are presented for all the drop heights and for all the 3 types of specimens: virgin, nanomodified 1 and nanomodified 2.



(a) Force vs Time diagrams.



(b) Force vs Displacement diagrams.

Figure 2: LVI results. Blue line: virgin specimens - Dashed lines: nanomodified 1 - Dotted lines: nanomodified 2.

From each tests energy absorbed, peak force and rebound velocity are investigated. Results are presented in Table 2.

3.3 Post-impact bump test

Harmonic frequencies are determined again after impact in the same way as stated in section 3. 1 Results are shown in Table 3 and 4.

4. Results and discussion

4.1 Impact tests

In the case of the lowest energy level of impact no significant changes in terms of rebound velocity and energy absorption can be found comparing the virgin and the nanomodified laminates. As it is clear from results in Table 2, data related to impact at 0.25 m present a big dispersion: it is not possible to lead to a conclusion for this case. This is due to the fact that 3 J of impact energy is not enough for induce a significant damage to the laminates, and only the elastic contribution is registered in terms of energy. For this low impact energy case the most interesting aspect to be highlighted regards the force history. From the fist diagrams presented in Figure 2(a) and 2(b) it is clear that nanofibers reduce the peak force during contact and increase the time of contact. Force reduction is around 8% for both the nanomodified configurations. From

	Virgin		**Nanomodified 1**		**Nanomodified 2**	
	Energy absorbed [J]					
	μ	σ	μ	σ	μ	σ
0.25 m	0.32	0.04	0.39	0.22	0.20	0.08
0.50 m	1.97	0.21	1.99	0.04	1.78	0.16
	Max Force [N]					
	μ	σ	μ	σ	μ	σ
0.25 m	1905.08	21.62	1756.77	30.46	1761.52	181.62
0.50 m	2337.52	24.93	2287.87	49.79	2329.27	68.16
	Rebound velocity [m/s]					
	μ	σ	μ	σ	μ	σ
0.25 m	2.076	0.016	2.009	0.060	2.088	0.011
0.50 m	2.483	0.044	2.504	0.033	2.550	0.003

Table 2: Impact results.

	Virgin		**Nanomodified 1**		**Nanomodified 2**	
	μ	σ	μ	σ	μ	σ
f_1 $[Hz]$	127.3	2.9	110.1	0.9	110.0	3.3
f_2 $[Hz]$	786.2	28.0	708.9	5.8	710.2	7.4

Table 3: Harmonic frequencies after 3 J impact.

	Virgin		**Nanomodified 1**		**Nanomodified 2**	
	μ	σ	μ	σ	μ	σ
f_1 $[Hz]$	126.0	0.6	121.4	0.6	122.5	4.1
f_2 $[Hz]$	757.4	3.7	750.6	7.5	754.3	25.8

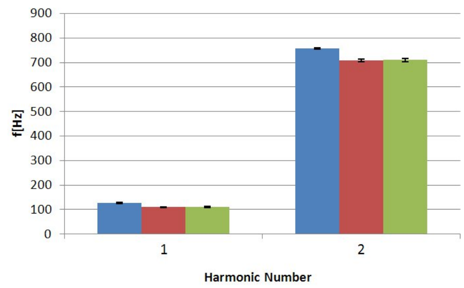
Table 4: Harmonic frequencies after 6 J impact.

force-displacement curve it also appears that virgin specimens are stiffer than the nanomodified ones. From Figure 2 it is not possible to mark the value of maximum force for highest energy impacts. For 6 J impacts, nanofibers lead to a reduction in absorbed energy and an increasing for rebound velocity at the same time: nano-interlayers are able to strength the interfaces and induce a smaller damage in the specimens. In nanomodified condition 2, a 10% less energy is absorbed by the laminate compared to those absorbed by the virgin specimens. Same effect is registered for the rebound velocity.

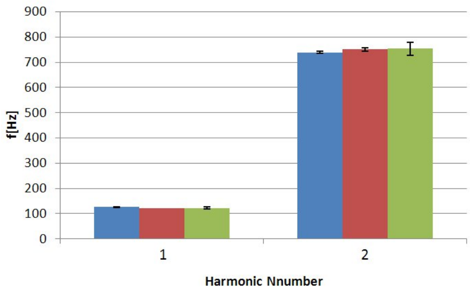
4.2 Bump tests

Bump tests results, on non-impacted laminates, showed that both nanomodified specimen types are less stiff than the virgin ones: the first harmonic frequency is proportional to flexural stiffness and it is lower for nanomodified specimen. This is confirmed in the first diagrams presented in Figure 2: the rising trend of nanomodified curves is clearly below the same part of the one of virgin specimens. Figure 3 presents the first three harmonic frequencies for all the configurations after the impacts.

For virgin specimens both first and second harmonics decrease after impacts. From tables 1 and 3 it is enhanced that there are no significant changes between the non-impacted and 3 J



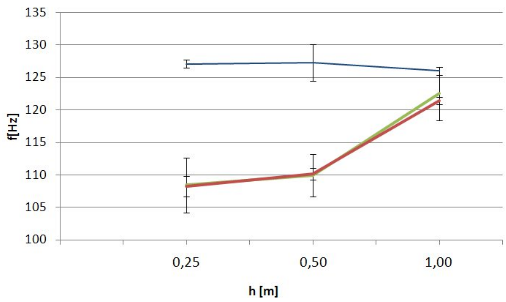
(a) Frequencies after 3J impact.



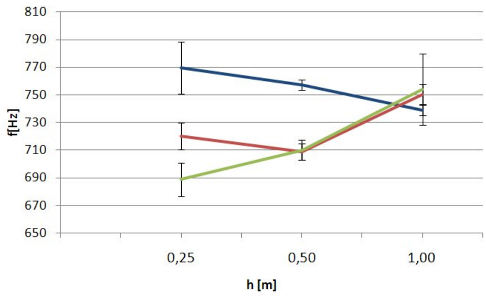
(b) Frequencies after 6J impact.

Figure 3: Post-impact harmonic frequencies. Blue bars: virgin specimens - Brown bars: nanomodified 1 - Green bars: nanomodified 2.

impacted specimens due to the fact that this impact energy level does not cause a significant damage to the laminates and consequently it does not affect the laminates vibratory behavior.
Figure 4 shows that for 6 J impacts the first two harmonic frequencies reach very close values for all the configurations. Experiments showed that virgin specimens present a decrease in first frequency, as expected, due to the damage induced by the impact. Opposite behavior is registered for nanomodified specimens, which present a rising trend in frequency, and thus in flexural stiffness. Both the first two harmonic frequencies are 12% increased after the 6 J impact.



(a) First harmonic frequency.



(b) Second harmonic frequency.

Figure 4: Comparison of post-impact harmonic frequencies. Blue lines: virgin specimens - Brown lines: nanomodified 1 - Green lines: nanomodified 2.

This fact is very unexpected and a flexural tests is performed to confirm the vibration results. So, to better investigate this results a bending test was performed on the impacted specimens cantilevered in one edge with the purpose to measure the laminate flexural behavior. An increasing load were applied while the strain gauges registered the deformation of the external skin of the plate. This test confirmed that specimen impacted with 6 J are stiffer than those impacted with the half amount of energy. From the Eulero-Bernoulli beam theory it is known that: $\omega_n = {a_n}^2 \cdot \sqrt{\frac{E \cdot I}{A \cdot \rho}}$. This equation leads to the fact that the ratio $\frac{\omega_n}{\sqrt{E}}$ must be constant and it is verified for all the tested specimens.
An explanation for the phenomena can be found in a previous work of the authors [12]: in this work the results of an experimental campaign about DCB end ENF tests on virgin and nanomodified specimens is presented. In figure **??** it is shown a Force vs Displacement curve for virgin and nanomodified specimens during a DCB test.
From figure 5 it is possible to see that the presence of nanofibers in the delaminated layer induce

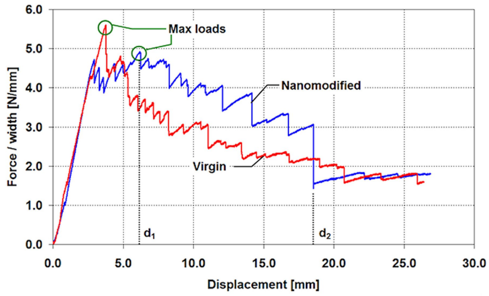


Figure 5: DCB force vs displacement curve for virgin (red line) and nanomodified (blue line) specimen

a reduction in maximum force capability and a strengthening after first fail at the same time. After maximum load, matrix starts to break and nanofibers are able to begin to work and to keep interfaces together. After first fail nanomodified interfaces, placed far away the neutral plane give the possibilities for the specimen to carry on higher loads respect to virgin ones and stiff the specimens. Consequently, after delaminations induced by impact at the highest energy level it can be expected that the presence of the nanofibers at the damaged interfaces can guarantee a significant residual load bearing capacity.

5. Conclusions

A deep experimental investigation was performed to investigate the effect of polymeric electrospun nanofibrous interlayer on CFRP composite laminate plates. Bump and impact test were performed in virgin and nanomodified configuration. Symmetric and non-symmetric lay-up configurations of nanomodified laminates were tested. Bump test are performed to investigate vibrational behavior before and after an impact event: first two harmonic frequencies are determined for each specimen. Low velocity impact tests are performed in a drop weight machine and three energy level are used. Results are collected in terms of force history, energy absorbed and rebound velocity. From impact test the effect of nanofibers in significant, and in particular for those non-symmetric configuration, in which nanofibers re concentrated in the lower interfaces respect to the impact. Increase in terms of energy absorbed and rebound velocity are assessed. An 8% lower peak force is also measured for the lowest energy impacts in nanomodified configurations. Results from bump test are very unexpected and are an interesting starting point for future insights. For nanomodified laminates it is measured an increasing of the first harmonic frequency of about 12% for both the nanomodified configuration, when impacted with 6 J of energy. Similar trend, but much lower is registered for 3 J impacts. A deeper study is required to validate such results, like a C-SCAN investigation, micrographs, more impact tests at intermediate levels. If confirmed, such results allow the introduction of electrospun nanofibers in a very wide range of applications in composite material fields.

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