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ORIGINAL ARTICLE

Load bearing enhancement of pin joined composite laminates using electrospun polyacrylonitrile nanofiber mats

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Abstract Polyacrylonitrile (PAN) nanofibers were produced by an electrospinning technique and directly deposited onto carbon fabric to improve the load bearing strength of pin joined composite laminates. Two types of specimens, virgin laminates and nano-modified laminates, were prepared. A modified carbon fiber reinforced polymer (CFRP) laminate was fabricated by interleaving electrospun nanofibers at all of the interlayers of an eight-ply woven carbon fiber fabric. The load bearing test results of the pin joined laminates indicated the electrospun PAN nanofibers increased the load bearing strength by 18.9%. In addition, three point bending tests were also conducted to investigate the flexural modulus and flexural strength of both types of laminates. The flexural modulus and flexural strength also increased by 20.9% and 55.91%, respectively.

1. Introduction

Carbon fiber reinforced polymers (CFRPs) have been widely used in many structural components. In particular, in airplane structures, such as the Boeing 787 and the Airbus A350 XWB, over 50% of the weight consists of CFRPs. This rapidly growing use of composite structures requires sophisticated joining methods because the joining area is the critical points that have to be designed carefully. Currently, three types of joints are commonly used in composite structures, namely mechanical fastened joints, adhesive joints, and hybrid mechanically fastened-adhesive joints (Thoppul et al., 2009). Bolted joints and hybrid joints are the most widely used for the joining of composites. Bolted joints offer an advantage because they can be removed without destroying the structure. Several investigations in this type of composite joining approach are simplified by using a pin joint, where the out-of-plane load due to the bolt clamping is avoided and only pure in-plane load is considered. The load bearing strength of a pinned joint

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of CFRPs depends on several parameters, such as the linear densities of the fiber or fabric and the geometry of the joint (Asi, 2009; Liu et al., 1999). The linear densities of the woven fabric have a dramatic effect on the bearing strength of the pinned joints of the composites. The bearing strength of glass-fiber reinforced epoxy composites first increases with the increase of the linear density of the woven fabric and then decreases with further increasing in the linear densities, due to higher void content and crimp levels of the composite specimens (Asi, 2009). The geometric parameters of the end distance from the hole-center (E), the width of the plate (W), and the hole/pin diameter (D) remarkably affect the load bearing performances of the composite materials. For the woven glass-fiber reinforced composite laminates, the optimum bearing strength could be achieved by using the parameters of $W/D = 4$ and $E/D = 4$ (Asi, 2009). The failure modes of specimens with $W/D > 2$ and $E/D > 2$ are bearing, while those for $E/D = 2$ are mixed modes of bearing and shear out, and those for $W/D = 2$ are mixed modes of bearing and net-tension (Asi, 2009). The diameter of the pin is strongly dependent on the thickness of the laminates used to obtain the best joining. A report indicated that the square root of the laminate thickness over the pin diameter should be below the unity to reach the bearing mode of failure (Liu et al., 1999). In the case of a bolted joint of CFRP laminates, the bearing strength is also highly affected by clamping forces (Rosales-Iriarte et al., 2011). In addition, the clearance between the bolt and laminate holes reduced the joint strength at bearing dominant loads due to high stress in the contact area (Rosales-Iriarte et al., 2011). Similarly, at the pin joint of GFRP laminates, the bearing strength was found to be reduced by the clearance, and an interference-fit was recommended for use in a pin joint (Kiral, 2010). Certain service conditions of composite materials are exposed to severe environments, such as hot-wet environments, which also have been studied. The changes in strength and failure mechanisms of bolted joints of carbon-fiber reinforced polyphenylene sulfide (PPS), a type of thermoplastic polymer, have been reported (Vieille et al., 2011). The load bearing test has been conducted at room temperature (RT)/Dry and 120 °C/hygrothermal aging conditions. The environmental conditions enhance the ductile properties of the PPS matrix, but degrade the fiber/matrix interface. For double-lap joints, the combined action of temperature and hygrothermal aging delays the onset of damage due to the enhanced plasticization mechanism of the matrix above the bolt/hole contact area. This enhanced plasticization is the reason why damage stress increases to such an extent with increasing temperature and moisture content. However, in single lap joints, the secondary bending of the specimen causes more out-of-plane plastic deformation than in-plane plastic deformation of the matrix above the bolt/hole contact area. Hence, under severe conditions, the single lap joint strength was decreased. Another experimental investigation on the bearing damage evolution of a pinned joint under static and repeated tensile loading concluded that the bearing failure occurs by a process of damage accumulation (Satoshi et al., 2010). At the early stage of damage, 0^0 layer in-plane bending damage occurred, followed by delamination, fiber kinking, and finally shear matrix cracking. Based on the major feature of bearing failures described above, the mechanical properties of the matrix have a significant effect on the load bearing strength. To increase the mechanical properties of the matrix, the presence of nano-fillers in the matrix of the laminate has been investigated (Asi, 2010). Several specimens with different weight percent of Al_2O_3 particles as the filler in GFRP composites have been investigated in single-hole pin loaded specimens tested in tension. The highest bearing strength was obtained for a composite specimen with 10 wt.% Al_2O_3 particle content, which exhibited a bearing strength 20% higher than that of the unmodified epoxy matrix. A further increase in the Al_2O_3 particle content resulted in a decrease of the bearing strength, but the strength remains above that of the unfilled GFRP laminates. In the laminate composites, delamination along the interlaminar planes is a serious problem that can cause structural failure. Several methods, such as laminate stitching, matrix toughening, and optimization of the stacking sequence, have been used to improve the delamination resistance. Furthermore, secondary reinforcement of the laminate with nanofibers has been reported to be a promising method to enhance delamination resistance. The pioneering idea of such delamination resistance was proposed by Dzenis and Reneker in their patent (Dzeniz and Reneker, 2001). The existence of polybenzimidazole (PBI) nanofibers as an interleaving reinforcement of CFRP improved the Mode I and Mode II fracture toughness values by 13.5% and 128.2%, respectively. By applying polycarbonate nanofibers at the interlayer of CFRPs, the first ply failure (FPF), damage initiation stress, and number of micro-cracks at the delamination area also have been improved by 8.5%, 8.1%, and 21.6%, respectively (Sihn et al., 2008). The nanofibers were produced by electrospinning with and without CNT as a filler material. The polycarbonate nanofibers were introduced at each interlayer of six-ply CFRP laminates, and the different of total thicknesses of the laminate with and without nanofiber interlayer was less than 0.001 mm, which was a negligible thickness. Another polymer nanofiber, such as epoxy, has been investigated (Liu et al., 2008a, 2008b). GFRP laminates with epoxy nanofiber used as a secondary reinforcement were evaluated for their Mode I and Mode II fracture toughness values. No significant effect was found on G_{IC} (Liu et al., 2008b), while G_{IIC} was enhanced significantly when the thickness of the nanofiber mat was 130 μm for a single mat or not more than 60 μm for multi-sheet of mats incorporated (Liu et al., 2008a). The pristine laminate failure was found to be dominated by resin cracking and fiber-breaking, while the nano-interleaved laminate developed with an initial resin deformation occurring nearby the nanofiber layer, and crack propagation was hindered by nano-fiber bridging. The use of Nylon 6.6 as nanofiber to reinforce CFRP laminates also attracted the attention of many researchers. The effect of Nylon 6.6 on the impact damage resistance (Akangah et al., 2010) and interlaminar properties (Palazzetti et al., 2012) of CFRP laminates have been evaluated. The increase of threshold impact force by 60% has been reported (Akangah et al., 2010). The results of the Double Cantilever Beam (DCB) tests conducted on virgin and nano-modified laminates at the mid-plane interface of the laminates indicated an increase by 23.2% of the mechanical energy absorbed and an increase of approximately 5% of the G_{IC} for the nano-modified laminates. The end notched flexure (ENF) test results revealed that the nanofibrous mats improve the maximum stress before material crisis by 6.5%, and the maximum mechanical energy that can be absorbed by the material during the crack propagation was improved by 8.1% (Palazzetti et al., 2012). The present work investigates the use of an electrospun PAN nanofibrous mat as the interleaved material to enhance the mechanical performances of composite laminates. PAN electrospun mats are fabricated and placed in composite laminates based on an epoxy-matrix/carbon-fibers that is tested to investigate the effect of the use of polyacrylonitrile (PAN) nanofiber mats on the load bearing strength of pin joined composite laminates.

2. Experimental details

2.1. Materials

2.1.1. CFRP laminates

CFRP laminates were prepared using woven carbon fiber with epoxy resin as the matrix. The carbon fabric, 0–90 plain weave 3 K, was manufactured by "jb martin ltée", Canada, and the epoxy resin 105 was received from West System, USA.

2.1.2. Electrospun nanofiber mats

The PAN nanofibers were prepared by dissolving (5 wt.%) polyacrylonitrile powders in N–N Dimethylformamide (DMF), and then stirred at room temperature until the mixture became homogenous. Subsequently, the solution was filled in a 20-mL NORM-JECT Luer Lok tip plastic syringe having an 18-gauge stainless-steel needle with a 90° blunt end. The electrospinning setup included a high-voltage power supply, purchased from the NanoNC, Inc. (S. Korea), and a nanofiber collector of aluminum foil that covered a laboratory produced roller with the diameter of 12 cm, as shown in Fig. 1.

The collector was placed at a 20-cm tip-to-collector distance (TCD). During electrospinning, a positive high voltage of 20 kV was applied to the needle; and the solution feed rate of 0.5 mL/h was maintained using a KDS 200 syringe pump purchased from the KD Scientific Inc. (Holliston, MA). The electrospinning setup is shown in Fig. 1. The electrospinning was performed at room temperature.

2.1.2.1. Preparation of specimens. Composite laminates were fabricated by hand lay-up of eight-plies of woven carbon fiber fabric and subsequently cured using a hot press machine at 100 °C for 1 h at a given pressure of 0.6 MPa. The modified laminates were fabricated by embedding the woven carbon fiber at the rotating drum collector for 48 h during the electrospinning process. The woven carbon fiber with embedded PAN nanofiber mats was dried in an oven at 40 °C during a night prior to the hand lay-up. In total, there are seven nanofiber layers within the modified laminates. The same hot press conditions were applied to the nano-modified laminates. Next, the specimens were cut using a diamond blade cutter to the dimensions shown in Fig. 2, with the width (W) and total length ($E + L$) of 24 mm and 104 mm, respectively. Five specimens were prepared for each of the virgin and modified laminates. A hole of diameter of 6 mm was drilled by a diamond coated drill from Sandvik Coromant. The position of the holes is in

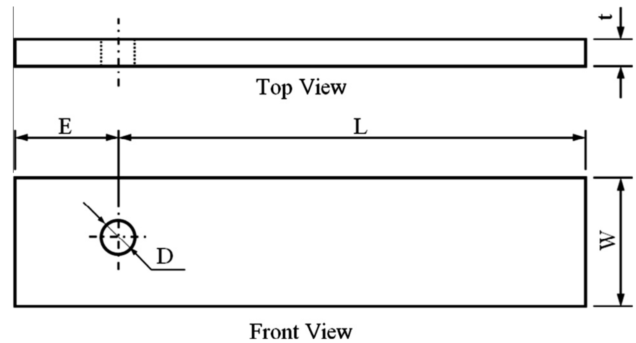

Figure 2 Load bearing test specimen.

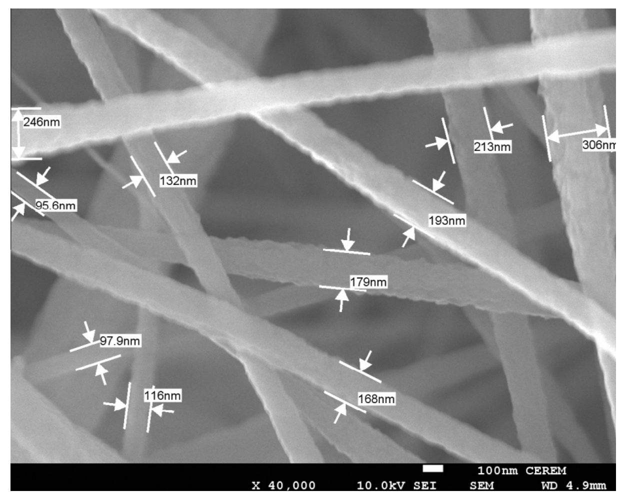

Figure 3 FESEM image of PAN electrospun nanofiber.

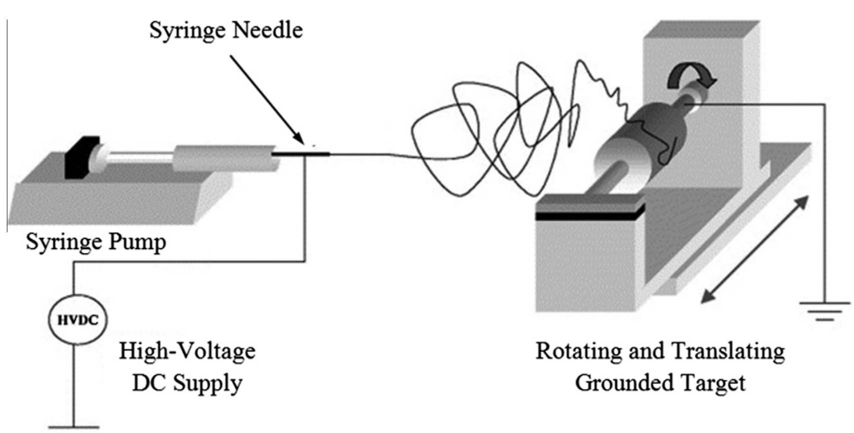

Figure 1 Schematic for electrospinning system.

the middle of the width and 24 mm from one end (symbol-*E* in Fig. 2).

2.2. Testing procedures

2.2.1. Scanning electron microscope image

The microstructure observations of polished and coated surfaces of the nanofibers were investigated using a Field Emission Scanning Electron Microscope (JEOL FESEM 7600 F). The surfaces were coated with platinum to avoid charging during the FE-SEM observation.

2.2.2. Load bearing test

Load bearing tests were conducted using an Instron 3385H series tensile test machine, with a 1 mm/min crosshead speed. A small load cell with maximum of 5 kN load was used to ensure data accuracy. To investigate flexural modulus and flexural strength of both types of specimens, three point bending tests also were performed, following the ASTM standard D790-10 (ASTM, 2010). Subsequently, the tested specimens were cut according to Fig. 2 to conduct microscopic observation of the cross section using an optical microscope.

3. Result and discussion

The morphology of electrospun fibers is influenced by various parameters, such as applied voltage, solution flow rate, distance between the capillary and collector, and especially the properties of the polymer solutions (including concentration, surface tension, and the nature of the solvent). From the

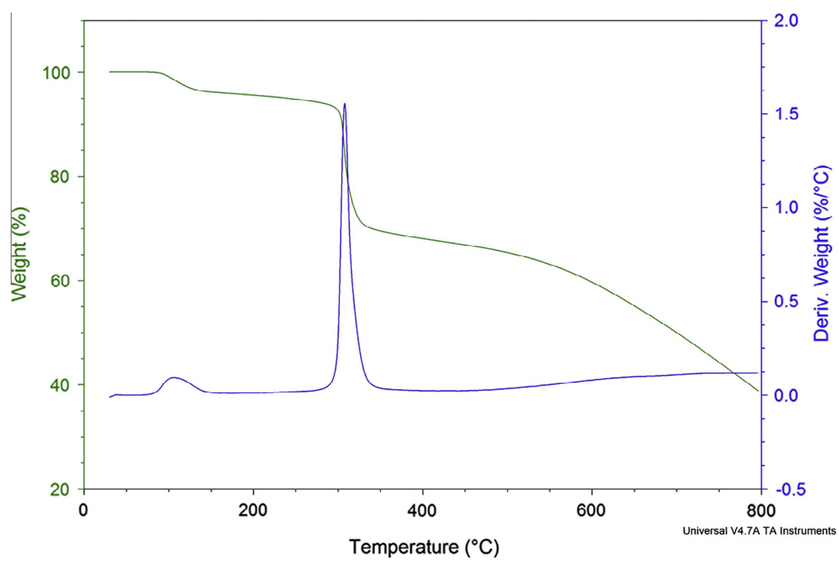

Figure 4 TGA of the PAN nanofibers.

Table 1 Mechanical properties of nanofibers (as a single fiber or a fibers mat).

Type of nanofiber	Modulus elasticity (GPa)		Ultimate strength (MPa)		Elongation at break (%)	Note	Ref.
	Single fiber	Mat	Single fiber	Mat			
Polyacrylonitrile (PAN)	7.6		30–70		150–220		Naraghi et al. (2007)
		1.1 (2.8)		51.8 (80.5)	22.1 (11.6)	As spun (as hot stretched)	Hou et al. (2010)
		4.5		265	17.8	Hot pressed	Ge et al. (2004)
		2.2 (3.6)		71.9 (216)	12 (10)	Normal PAN Terpolymer (Hot stretched)	Ji et al. (2009)
Polyimide (PI)	76		1700		2.8		Chen et al. (2008)
		2.1		308	201.9	In terms of 6F-PI	Cheng et al. (2010)
		1.3		174	57.2	PI film	Chen et al. (2011)
		15		664	5	BPDA-PDA PI	Huang et al. (2006)
Nylon 6.6	0.45–0.95		110–150		66		Zussman et al. (2006)
	0.9	0.019	304	10.5	40 (250)	As single fiber (as a mat)	Bazbouz and Stylios (2010)
Poly(vinylidene fluoride) – (PVDF)		0.023 (0.39)		4.4 (44.6)	105 (122)	As membrane (as twisted nanofibers)	Nakashima et al. (2011)

Figure 5 Load bearing test set up.

SEM micrograph of the PAN fibers electrospun from a 5% PAN solution in DMF, as shown in Fig. 3, the physical morphology of the electrospun samples was examined using the scanning electron microscope. All samples could be produced without a bead and as uniform nanofibers. The addition of a different concentration of PAN has a large effect on the electrospun fiber morphology. Good nanofiber conditions were produced without any beads, and the average diameter of the nanofiber is 150 nm.

In this study, TGA was conducted in N_2 atmosphere as shown in Fig 4. There is one sharp exothermic peak at 295 °C for electrospun fibers. It has been reported that an exothermic reaction ranging between 200 and 350 °C in an inert atmosphere is typical of PAN.

Many investigations on the mechanical properties of nanofibers were conducted, with the results summarized in Table 1. Based on these available data, polyacrylonitrile (PAN) nanofibers appear to possess high potential for the application as secondary reinforcement of the composite laminates.

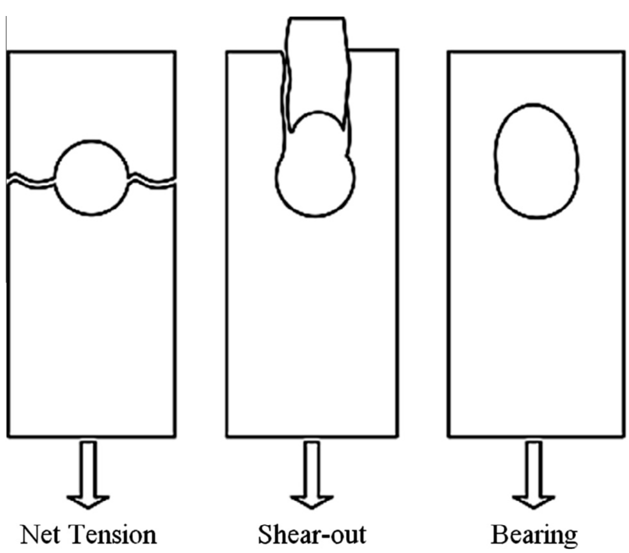

Figure 6 Failure modes of bearing test.

3.1. Load bearing and flexural properties

The experimental details of the load bearing test set up are shown in Fig. 5. The types of load bearing failure are briefly described in Fig. 6. Three point bending tests also were performed, following ASTM standard D790-10 (ASTM, 2010) using the same Instron machine as used for the load bearing tests, as shown in Fig. 7. Subsequently, the tested specimens were cut according to Fig. 8 to conduct microscopic observation of the cross section using an optical microscope.

The mechanical results for the load bearing and flexural properties tests are presented in Fig. 9 and Table 2; the force–displacement curves for both virgin and nano-modified configurations are shown. The force values were normalized to the specimen's width. In Fig. 8, examples of the

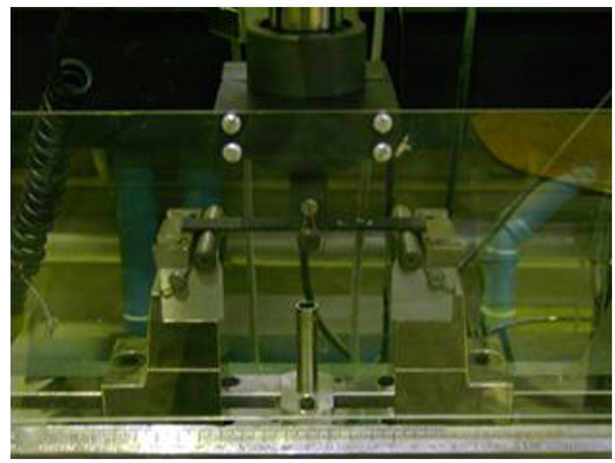

Figure 7 Three point bending test.

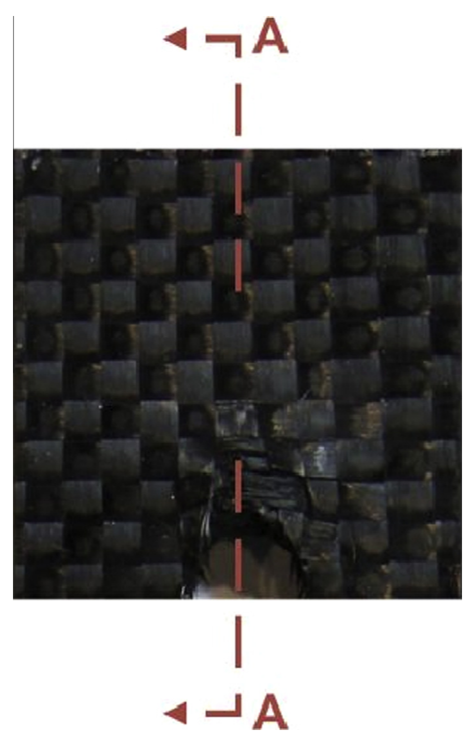

Figure 8 Sectioning at bearing plane.

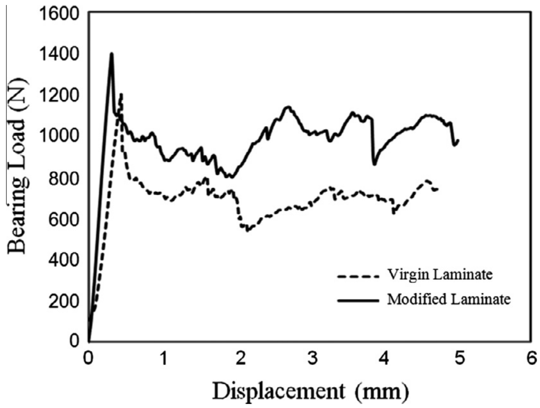

Figure 9 Load bearing test results.

force–displacement curves are reported for the two types of specimens: the virgin and the nano-modified specimens. This diagram highlights the higher maximum load of the nano-modified specimens with respect to the virgin ones. Nevertheless, the load values of the nano-modified laminate for higher displacements exhibit a higher magnitude with respect to the virgin laminate. From the force–displacement diagrams, the displacement values discriminating the three phases of the test were identified. Such a behavior can be related to the presence of the nanofibrous layer that tends to hinder the crack propagation in the opening. The above reported summary of load bearing data is presented in Table 2, and the median data of each type are plotted in Fig. 9. The load bearing strength was increased by 18.93% due to the existing PAN nanofiber mats at the inter-layers. A negligible difference in the thickness was observed, which could be considered as the hot pressure machine tolerance.

It is interesting to understand the load bearing strengthening mechanism of a pin joint using a PAN nanofiber interlayer. Because the first damage mechanism is the 0° layer in-plane bending damage and delamination, the matrix properties play the most important role in damage onset. Although this work did not evaluate specifically the effect of nano-fiber on the properties of the epoxy, the flexural test results of nano-modified laminates indicate the increment of the flexural modulus and strength by 20.9% and 55.91%, respectively. The results indicate the improved stiffness and strength of the composites, which are mostly affected by improved matrix properties. These enhancements seem to play the main role in the reduction of in-plane bending damage. Furthermore, Fig. 9 shows the effect of matrix enhancement on the bearing strength, which clearly shows that the slope and peak of nano-modified specimens is higher than those of the virgin ones. The increased values of the flexural modulus and strength appear higher compared to another set of reported data of the enhancements of 0.9% in the flexural modulus and 6.4% in the flexural strength (Palazzetti et al., 2012). The source of this difference could come from the number of nanofiber layers and the type of nanofiber materials.

In addition to these increments, the use of PAN nanofiber mats at the interlayer also has a significant effect on the bearing load after failure. Fig. 9 also shows that the modified laminates can carry a greater load after local failure of the joint. The tests were run until 5 mm of crosshead displacement; the curve of modified laminate was observed to always lie above the virgin one after the local damage (the peak load). This phenomenon occurs due to the improved delamination properties of the nano-modified laminates. Although the work did not evaluate the delamination toughness itself, many investigations have reported that the use of a nanofiber layer increased the delamination toughness (Dzeniz and Reneker, 2001; Liu et al., 2008a, 2008b; Palazzetti et al., 2012). In addition to delamination toughness, the microscopic fracture surface in Fig. 10 indicates that nano-modified laminate could reduce the fiber kink-kink and fiber failure, thereby improving the load bearing capability after failure.

Table 2 Load bearing and three point bending test result.

Specimen	Thickness (mm)	Load bearing strength		Flexural modulus		Flexural strength	
		M.V. (N)	Δ%	M.V. (GPa)	Δ%	M.V. (MPa)	Δ%
Virgin	1.685 ± 0.016	1200.92 ± 173.0		45.04 ± 4.12		245.89 ± 20.66	
Nano-modified	1.664 ± 0.082	1428.26 ± 168.86	18.93	54.45 ± 2.19	20.9	383.37 ± 35.48	55.91

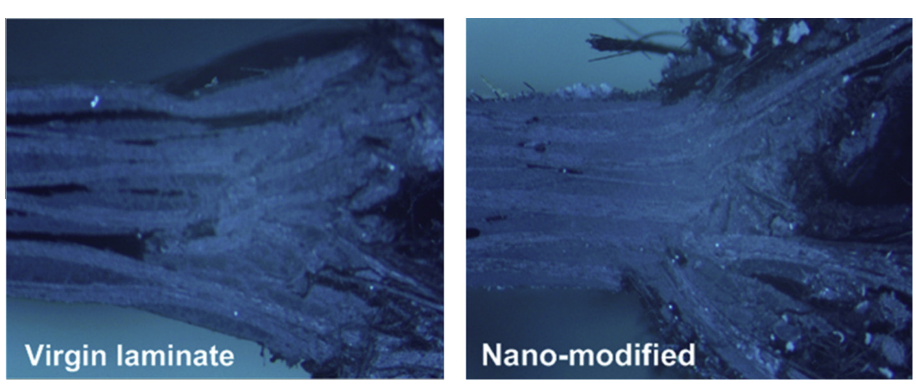

Figure 10 Fracture surface of the cross-section bearing plane.

4. Conclusion

In this work, the effect of PAN nanofiber mats on the load bearing strength of pin joint CFRP laminates was initially investigated. Two types of specimens, virgin laminates and nano-modified laminates, were tested. The laminates were fabricated by hand lay-up of eight-ply woven carbon fiber followed by hot pressing. The nanofiber mats were introduced at each interlayer by embedding the woven carbon fiber onto the rotating drum collector of the electrospinning machine prior to the hand lay-up process. In addition to load bearing tests, the flexural properties were also investigated. The results can be summarized as follows:

1. PAN nanofiber mats increase by 18.93% the load bearing strength of pin joint CFRP laminates.
2. The flexural modulus and the flexural strength of CFRP laminates also increased by 20.9% and 55.91%, respectively, due to the presence of nanofiber mats.
3. The change in the mechanical properties of the matrix can delay in-plane bending damage and delamination; this delay in the damage onset increases the load bearing strength.
4. The obtained results could provide benefits to many applications of composite structures, especially where the use of joints is required. The use of electrospun nanofiber at the interlayer enhanced the load bearing strength of the pin joint without changing the thickness of the laminates; hence, the application could be more practically simple by embedding the nanofiber only in the vicinity of the joining area.

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