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Letter

Effect of neat and reinforced polyacrylonitrile nanofibers incorporation on interlaminar fracture toughness of carbon/epoxy composite

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HIGHLIGHTS

- Al_2O_3 nanoparticles were incorporated in PAN nanofiber to be used in reinforcement of carbon/epoxy composites..
- The carbon/epoxy composite panels were reinforced with Al_2O_3 -PAN and PAN nanofibers.
- Fracture tests were conducted on DCB specimens to study the interlaminar fracture behavior of reinforced composites.
- Al_2O_3 -PAN nanofiber reinforced composites had higher fracture energy improvements compared to the PAN nanofiber reinforced composites.

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ABSTRACT

This paper presents an experimental investigation on fracture behavior of epoxy resin-carbon fibers composites interleaved with both neat polyacrylonitrile (PAN) nanofibers and Al_2O_3-PAN nanofibers. In particular, the paper focuses on the effect of adding Al_2O_3 nanopartiles in PAN nanofibers, which were incorporated in unidirectional (UD) laminates. The effectiveness of adding a thin film made of Al_2O_3-PAN on the fracture behavior of the carbon fiber reinforced polymer (CFRP) has been addressed by comparing the energy release rates, obtained by testing double cantilever beam (DCB) samples under mode I loading condition. A general improvement in interlaminar fracture energy of the CFRP is observed when the both neat PAN nanofibers and Al_2O_3-PAN nanofibers are interleaved. However, higher interlaminar strength has been observed for the samples with a thin film of Al_2O_3-PAN nanofibers, suggesting a better stress distribution and stress transformation from resin-rich area to reinforcement phase of hybrid composites.

Laminated polymer matrix composites (PMCs) reinforced with carbon, glass, aramids etc fibers have been extensively employed in variety of applications due to their superior properties as well as facile and economical methods of fabrication [1-3]. The in-plane mechanical properties of these types of materials were dictated by the reinforcement phase (fibers) mechanical properties subsequently, the in-plane properties of PCMs are powerful enough for many applications including aerospace, ground vehicles and wind turbines. In contrast, the out-of-plane mechanical properties of the laminated composite were dominated by mechanical properties of the matrix [4, 5]. It is worth mentioning that the matrix mechanical properties are significantly less than of the mechanical properties of reinforcement phase resulting inadequate out-of-plane properties. Moreover, ply-by-ply temperament of the laminated PMCs makes them susceptible to delamination owning to creation of microcracks and subsequently, microcracks propagation through the weak

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resin-rich layer. Due to, this significant disadvantage laminated PMCs have not been used in critical applications [4]. Therefore, a series of researches have been carried out for enhancing the matrix mechanical properties by incorporation of a second phase such as carbon nanotubes [6, 7], micro/nanofillers [8-12] in the matrix or even improving the curing system [13, 14]. However, the above mentioned methods have not been employed in fabrication of composite materials due to some limitations such as troubles due to significant increase in the viscosity of resins which adversely affect the manufacturing process and subsequently, decreases the mechanical properties of final fabricated composites [15].

A method of composite strengthening was proposed by Dzenis and Reneker with the use of nanothechnology and has gathered much research interest. In this method un-oriented electrospun nanofiber mats were incorporated between layers of the reinforcement phase leading to significant improvement in the mechanical properties of the weak resin-rich area [16, 17]. The method provides an inexpensive and facile technique for enhancing mechanical properties of resin-rich area with minimum influences on the fabrication processes [4]. The improvement in mechanical properties of resin-rich area, via incorporation polymeric nanofibers, has been extensively studied during recent years [18-21] as well as improvement in fracture toughness of epoxy adhesives [22-28] and neat resins [29, 30]. The researchers reported that the nanofiber breakage, nanofiber pull out, stress distribution by the nanofibers, and stress transform from weak phase to reinforcing phase are the mechanisms of mechanical properties improvement [20, 21]. Therefore, the nanofibers diameter, the type of nanofiber (mechanical properties of the nanofibers), thickness of the nanofiber mat, and the nanofibers and matrix interaction have a significant impact on the amount of mechanical properties improvement [5].

In the present research, the effect of reinforcing nanofiber, via using of the nanoparticles, on the improvement of fracture toughness of a carbon/epoxy composite was investigated. Improving mechanical properties of nanofibers by incorporating nanofillers was reported in previous works [31, 32]. Consequently, the approach is reinforcing the polyacrylonitrile (PAN) nanofibers by adding Al_2O_3 nanoparticles [33]. Therefore, the neat PAN and PAN-Al_2O_3 nanofibers were deposited on carbon fabrics. Three types of the carbon/epoxy composites namely control samples, PAN reinforced the carbon fiber reinforced polymer (CFRP) and PAN-Al_2O_3 reinforced CFRP panels were fabricates. The composite panels were then evaluated via mode I fracture energy assessment tests to investigate the influence of reinforcing nanofibers on the fracture toughness of a conventional carbon/epoxy composite.

PAN (M_w=150000 g/gmol) and N, N-dimethylformamide (DMF, 99.8%) were provided from Sigma-Aldrich. The epoxy resin (EPON^{TM} Resin 828), and its curing agent (EPIKURE^{TM} Curing Agent F205) were supplied by Hexion Inc. The conventional carbon fiber fabric (300 g·m^{-2}) was purchased from Jinsor-Tech Industrial Co. Al_2O_3 nanoparticles (99%, diameter average=20 nm) were purchased from nanosany Co.

The 10 wt% of PAN and 10 wt% of PAN containing 1 wt% of Al_2O_3 nanoparticles were dissolved in adequate DMF by employing magnetic stirrer for 24 h at room temperature. In order to make sure excellent dispersion of Al_2O_3 nanoparticles, the solution (containing Al_2O_3 nanoparticles) was solicited for 20 min using high intensity ultrasonic liquid processor (UP 400S, 40 KHz Sonics Vibra Cell, Hielscher, Inc). The obtained solutions were filled in the syringes with needle gauge 21 (inner diameter=0.51 mm). Before the electrospinning, the conventional unidirectional carbon fiber fabrics were placed around a 15 cm diameter grounded drum. The nanofibers were directly electrospun on the carbon fabric surfaces. The electrospinning process parameters were set the same as our previous work for both neat PAN nanofibers and Al_2O_3-PAN nanofibers [5] leading to a stable electrospinning process for preparation of the uniform nanofibers without any beads. Electrospinning process was hold until 1 g of nanofibers was gathered on a square meter of carbon fiber fabrics. Figure 1 schematically shows the nanofiber deposition on the surfaces of carbon fiber fabrics.

Carbon/epoxy composite panels were prepared using hand lay-up method proceed by the vacuum assist resin transfer molding (VARTM) method. The epoxy resin and its hardener were mixed in the mass ratio 100:58 in accordance with the manufacturer's recommendation. Two composite panels were fabricated containing 12 layers of uniaxial carbon fabric (parallel aligned) and epoxy matrix. The nanofiber reinforced panels compromised 11 layers of the neat PAN and Al_2O_3-PAN nanofibers in the resin-rich area, whereas the control composite did not contain any nanofibers. A 30 μm thick polyethylene film was also inserted in the mid-interface of all the specimens during the lay-up to create an initial artificial crack. Vacuum pressure was hold for 18 h in order to complete cure of epoxy resin at room temperature. Subsequently, three composite panels were separated from vacuum system and post cured in an oven at 60 °C for 30 min.

The morphology of Al_2O_3 nanofiber and the fractured surfaces of the composites were analyzed by employing a field emission scanning electron microscope (FE-SEM), Hitachi S-4300, Japan. Before morphological investigations, the surfaces of the nanofibers and fractured composites were coated with a fine layer of gold. As mentioned in our previous work the average diameter of the neat PAN nanofiber and its standard deviation were obtained 380 and 70 nm, respectively [5]. Figure 2 shows the FE-SEM micrograph of Al_2O_3-PAN nanofibers and diameter distribution of the nanofibers for 50 diameter measurement using Image J software. From Fig. 2, it can be observed that the nanofibers containing Al_2O_3 nanoparticles were fabricated uniformly without any signs of beads. The average nanofiber diameter and its standard deviation were measured 417 and 112 nm, respectively. Therefore, the average of Al_2O_3-PAN nanofibers is 10% more than average of neat PAN nanofibers diameter. This increasing in nanofiber diameter can be attributed to growing of solution viscosity due to addition of the Al_2O_3 nanoparticles.

The double cantilever beam (DCB) samples have been prepared with 130 mm length, 25 mm width, initial crack of 45 mm. DCB samples were cut from composite panels using water jet. Steel hinges were glued on both tips of the samples to apply the load. DCB fracture tests have been performed to calculate the mode I energy release rate G_{Ic}, using Eq. (1), from the modified beam theory (MBT) according to ASTM D 5528 [34]:

$$G_{Ic} = \frac{3P\delta}{2ba'}, \tag{1}$$