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Low temperature growth of carbon nanotubes on carbon fibre to create a highly networked fuzzy fibre reinforced composite with superior electrical conductivity

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ABSTRACT

We report a method for the growth of carbon nanotubes on carbon fibre using a low temperature growth technique which is infused using a standard industrial process, to create a fuzzy fibre composite with enhanced electrical characteristics. Conductivity tests reveal improvements of 510% in the out-of-plane and 330% in the in-plane direction for the nanocomposite compared to the reference composite. Further analysis of current–voltage (*I–V*) curves confirm a transformation in the electron transport mechanism from charge – hopping in the conventional material, to an Ohmic diffusive mechanism for the carbon nanotube modified composite. Single fibre tensile tests reveal a tensile performance decrease of only 9.7% after subjecting it to our low temperature carbon nanotube growth process, which is significantly smaller than previous reports. Our low-temperature growth process uses substrate water-cooling to maintain the bulk of the fibre material at lower temperatures, whilst the catalyst on the surface of the carbon fibre is at optimally higher temperatures required for carbon nanotube growth. The process is large-area production compatible with bulk-manufacturing of carbon fibre polymer composites.
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1. Introduction

Carbon fibre reinforced polymers (CFRP) have revolutionised industries that demand mechanically strong materials without the potential weight penalty [1–3]. However, in the aerospace industry, metals are still being incorporated into structures to impart electrical conductivity to avoid charge build up gained through air friction and lightning strikes. Charge accumulation could lead to discharge sparks and/or material failure. The addition of metals, however, adds weight, cost, leads to corrosion issues and has proven troublesome to consolidate with the carbon fibre composite. Hence, there is interest in enhancing the electrical conductivity without relying on metals. With the current advancements in nanotechnology there exists opportunity to not only improve mechanical properties, [4–11] but to substantially enhance the electrical [12–14] and thermal [15–17] properties; introducing the composite material into new applications. Fabricating

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CFRP with the most promising nanomaterial, carbon nanotubes (CNTs), has proven challenging with issues such as dispersion, degradation of the CNTs and matrix viscosity. The general trend has recently been focused on *in situ*-growth of CNTs [18–22] or attaching CNTs to the carbon fibre, [23–28] creating a ‘fuzzy’ carbon fibre reinforced polymer (FCFRP) composite. These methods are preferred over dispersing the CNTs in the polymer matrix [29]. Despite advances in the dispersion techniques of CNTs in the polymer matrix [19,30–32] using techniques such as: covalent and non-covalent modification, surfactant addition and by mechanical processing methods, the technology is still in its infancy with issues such as: poor nanotube alignment, limitation to small weight percentage additions, poor CNT graphitisation, CNT agglomeration, lack of morphology control, and poor matrix infusion capability when subjected to infusion techniques such as resin transfer moulding (RTM) and vacuum assisted resin transfer moulding (VARTM) [9,11,27,33,34].

Using carbon fibre as a substrate for CNT growth allows for well dispersed and high density of CNTs to be incorporated in the composite. The CNTs can bridge adjacent carbon fibres, creating electrical and thermal percolation pathways throughout the composite as well as enhancing the mechanical characteristics of CFRPs. These include the out-of-plane characteristics, as well as enhancing the interfacial adhesion between carbon fibres and resin-dominated processes such as the interlaminar shear strength or longitudinal fibre compression [27,35]. This is nontrivial considering that the most common failure mechanism of conventional composites is fibre bending and breaking due to the lack of support by the matrix. These parameters are factors that determine the limits of the mechanical performance of CFRP composites [36]. It should also be stated that the challenge of dispersing the CNTs is removed, as is the potential damaging methods currently used to disperse the CNTs. Thus, fewer defective CNTs are incorporated into the composite resulting in improvements in electrical and thermal properties.

From the onset of the early experiments [37] of carbon nanofibre growth on carbon fibre it has been emphasised the importance of preventing mechanical degradation of the carbon fibres by the intense heating required for CNT growth using standard chemical vapour deposition (CVD) techniques. Thostenson et al. [38] used this growth technique for surface addition of CNTs on pitch-based carbon fibres. This spurred many studies to investigate the influence of CNTs on the fibres by either CVD growth processing or attaching the CNTs on the carbon fibre. Parameters such as pre-treatment, [20] temperature, [9,35,39–41] catalyst [9,20,41–45] (metallic and non-metallic) and hydrocarbon source [41,43] were reported. The majority of the work in the area considered the mechanical improvements from interlaminar toughness to tensile strength, however, very few studied the implications on the electrical and thermal properties.

At present, growing CNTs on the surface of carbon fibre has the advantage over attaching CNTs in terms of the quantity, length, controllability of size of CNTs [45] and orientation [46] that can be incorporated into the composite. All of which are crucial if the nanophase is to compliment the CFRP composite. However, with growth temperatures usually above 700 °C and reactive atmospheres present within the standard CVD chamber, significant degradation of the fibre has been observed [35,39,47–49]. If FCFRP is to replace CFRP in industries, it is imperative that the mechanical properties which make CFRP useful are retained. We report a process that allows the growth of CNTs with minimum degradation to the underlying carbon fibre substrate by maintaining the substrate at lower temperatures than those normally used in conventional CVD techniques for CNT growth without compromising on quality, yield or production time of CNTs. Our Surrey Nanosystems 1000n photo-thermal chemical vapour deposition (PTCVD) growth system features a water-cooled substrate table (cooled to 5 °C) and uses optical radiation for thermal heating [50,51]. This arrangement enables part of the carbon fibre fabric to remain at low temperature, whilst the catalyst on the carbon fibre (that is directly exposed to the optical radiation) is allowed to reach a high temperature necessary for the growth of carbon nanotubes of high crystalline quality [35,39,41,44,49,52–59]. After CNT growth, the thermal radiation (sourced by an array of halogen lamps) can be turned-off quickly, enabling the PTCVD to cool down in a short time, ~5 min, which greatly improves the sample throughput yield (up to 8 times as many samples compared to conventional CVD systems) and reduces the amount of gas used for cooling. As it employs optical heating instead of resistive heating, where there is a much larger thermal resistance between it and the catalyst sites, the system reduces the amount of energy required for growth. Additionally, this method is large-area compatible and directly transferable to industrial manufacturing companies. A detailed description of the design of the commercially available PTCVD and its operation principles for CNT growth at low substrate temperature is available for the case of other types of substrates (temperature-sensitive semiconductor substrates) elsewhere [50,60,61]. These studies report successful growth of CNTs of high crystalline quality, at low substrate temperatures.

Plasma Enhanced Chemical Vapour Deposition (PECVD) has the potential to lower the growth temperature but with the drawback of growing poor quality nanotubes or the growth of only carbon nanofibres [50]. There is also the possibility of the plasma energy needlessly increasing the temperature of the substrate [62,63].

This paper shows improvements in the electrical properties of the composite whilst minimising degradation through using a PTCVD system. An industrially-relevant VARTM method is used to demonstrate a good infusion of the nanocomposite through the capillary action of wetting the fibres out instead of using a polymer sizing to improve the wet out [4].

2. Experimental section

Sized bi-directional 2/2 twill carbon fibre (Grafil Pyrofil TR30S) was used as the reinforcement in this study (unless otherwise stated) with a low viscosity, room temperature curing two-part DGEBA epoxy resin (IN-2 epoxy infusion, Easy Composites™). CFRP samples were prepared using the VARTM to infuse four, 100 mm × 100 mm plies of 2/2 twill carbon fibre with the lay-up $[0,90]_4$. A nylon peel ply was cut and placed over the lay-up with an infusion mesh on top, which was subsequently sealed to the metal mould using vacuum bagging

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