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Carbon nanotubes grown on carbon fiber yarns by a low temperature CVD method: A significant enhancement of the interfacial adhesion between carbon fiber/epoxy matrix hierarchical composites

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ABSTRACT

In this work, we show that the interfacial adhesion between carbon fibers (CFs) and epoxy matrix in laminated composites can be significantly enhanced by the chemical vapor deposition (CVD) growth of multiwalled carbon nanotubes (MWCNTs) onto the fiber surfaces at low temperatures. The key process parameter was the deposition of catalytic nickel nanoparticles (NPs) onto the CFs at room temperature by a low energy double target DC sputtering system. This protocol enabled the growth of CNTs without any detrimental effect on the fiber properties, and enhanced effectively the adhesion between fibers and matrix. Fractographic investigations of single fiber/epoxy composites demonstrated an improved interfacial adhesion between the 'hierarchical' fibers (CF-CNT) with the epoxy matrix as compared to the bare carbon fibers. The developed protocol is versatile and it is envisioned to be easily scaled-up for volume production of CF-CNT, giving rise to high mechanical performance structural composites.

1. Introduction

Carbon fiber (CF) reinforced polymer composites are increasingly being employed in primary aircraft structures. Therefore, the necessity of fail-safety in these structures has motivated current research towards the solution of the well-known critical weakness of carbon fiber reinforced composite materials; "delamination". The recent delamination issue with the Boeing 787 Dreamliner [1,2] highlights the challenges in moving to different manufacturing methods with new materials. There are tremendous advantages for the use of composite materials, however, there is less experience in manufacturing with these materials and these types of problems are bound to occur. As aircraft materials evolve, finding and fixing delamination becomes an essential challenge.

Usually the structural damage due to delamination occurs at the matrix-fiber interphase [3–8]. Several ideas have been proposed to increase the interfacial bonding in this region. Chemical methods can be used to functionalize the carbon fibers with chemical groups that increase the adhesion between fibers and polymeric matrix [9,10]. Sometimes, nanoparticles and other nanostructures can be physically dispersed into the epoxy matrix, what would not only improve the mechanical properties across the interface of the plies but also reduce the crack propagation by bridging the two plies across the interface [3,4,11–13]. However, not always this simple approach works. Wang et al. [14] investigated the use of carbon nanotube buckypaper dispersed between the plies of carbon fibers composites. The obtained composite showed no increases in the mechanical properties compared to the control samples (fiber composites without the nanotubes buckypaper). In some cases the properties of the obtained samples were even lower than the control samples. As expected, the authors attribute this to the weak interface strength between fibers/carbon buckypaper/matrix.

The most interesting approach is the growing of multiwalled carbon nanotubes (MWCNTs) on the CF surfaces by chemical vapor deposition

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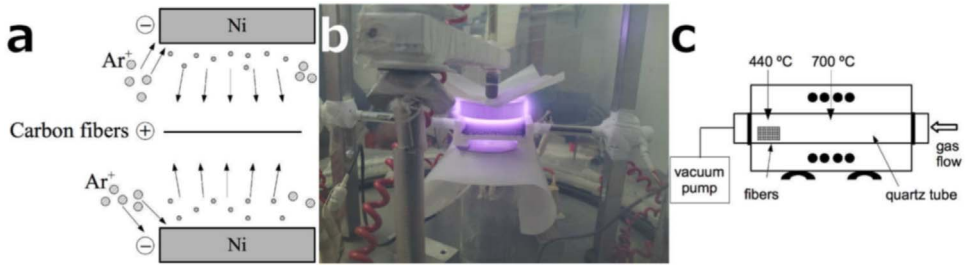

Fig. 1. (a) Schematic view of double target sputtering system; (b) photographs of the sputtering device during nanoparticles deposition; (c) strategy to synthesize MWCNTs, at low temperature, using a CVD system.

(CVD) [15]. Although it is a good and effective approach increasing the fiber surface area, inducing more contact points with the continuous matrix phase and introducing a mechanical interlocking mechanism, the CVD methodology followed could be detrimental. One of the pioneer works describing the growth of MWCNTs onto CFs was the one of Thostenson et. al. [15]. For the deposition of the catalyst on the carbon fibers' surface, they proposed to cover the fibers with a continuous film of the catalyst metal using magnetron sputtering, following by a thermal treatment in hydrogen atmosphere to convert the applied film in catalyst particles. This method becomes a standard procedure for the deposition of the catalyst on the CFs, and posterior growth of MWCNTs. However, the thermal treatment used to convert the continuous film into nanoparticles demands high temperatures that causes structural damage on the CFs and degradation of their mechanical properties [16].

Following the work of Thostenson, several researchers have studied the CVD growth of MWCNTs on CFs. Although applying different methodologies to the deposition and type of the used catalyst, high temperatures are always applied in some part of the process. For instance, H Qian et al. [17] and Feng An et al. [18] uses impregnation method to introduce iron catalyst precursor onto the carbon fibers. The immersion of the fibers in iron nitrate is carried out at room temperature, but the CVD synthesis was performed at 750 °C. In previous works of our group we shown that commercial available iron oxide nanoparticles could be used as catalyst, however, a high temperature reduction step it mandatory prior the CNTs synthesis [19,20]. High temperatures not only introduce defects on CFs structure, but facilitate the diffusion of the metallic catalysts into the fibers inhibiting their catalytic activity and decreasing thus the synthesis yield [21]. MWCNTs can be chemically grafted to the CFs surface at low temperatures [22,23], however with very poor recovery quality and effectiveness in the interfacial properties.

Many successful approaches for the synthesis of catalyst nanoparticles and carbon nanostructures at low temperature are reported in the literature [24–27]. These approaches yield mostly helical and straight nanofibers or carbon nanocoils and only a small fraction corresponds to carbon nanotubes. This phenomenon might be consequence of the low temperature used for the cracking of acetylene. The classical route of thermal decomposition of C_2H_2 is limited at low temperatures. Below 600 °C only amorphous carbon it's obtained instead of CNTs [28]. There are two recent works which report methods where all the steps necessary for CNT synthesis on carbon fibers had been realized at temperatures lower than 500 °C. In the oldest one [29] the catalyst for CNT growth is originated from mono- and bi-metallic salt solutions and the MWCNTs were synthesized on catalyst particles by means of an ambient pressure CVD reactor using an oxidative dehydrogenation reaction between C_2H_2 and CO_2. With this growth process, the CNT growth rate was rather moderate at 500 °C and had a maximum at 650 °C. In the new one [30] a nickel chloride solution was employed as the catalyst precursor bath and an ethanol flame was used as the carbon source. To grow the CNTs, the CF fabric wetted with the NiCl2 catalyst precursor was placed on a metal frame that was inserted into the flame at a temperature about 450–520 °C. The CNT growth rate obtained with this method was rather high.

In this work, we report a new methodology for the CNT grafting onto CF reinforcements, where catalyst nanoparticles are deposited on the CFs at room temperature and MWCNTs are grown by CVD at low temperatures (450 °C). The catalyst nanoparticles were deposited on CFs using a low energy double target DC sputtering system. The MWCNTs growth takes advantage of the temperature gradient inside the CVD oven. The carbon pyrolysis occurs at 700 °C while the fibers are positioned at a lower temperature zone avoiding its thermal degradation. CFs fully covered with MWCNTs were obtained (CF-CNT). We also show through cryogenic failure tests of single fiber/epoxy model composites that hierarchical composites with a CF-CNT exhibit an improved adhesion with the epoxy matrix compared to bare CFs.

2. Materials and methods

As received CFs (Hexcel, Hexforce282) were cleaned initially by immersion in an ultrasonic bath in a two-step process: 15 min in acetone followed by 15 minutes in ethanol. The CFs were dried then in a vacuum chamber at 120 °C for 2 h (called as “bare fibers”). Onto the surface of bare CFs, catalytic nanoparticles were deposited using a dual DC sputtering equipment schematically shown in Fig. 1a. In this process, the CFs were positioned as anode between two nickel cathodes. DC sputtering process was carried out with a current of 10 mA during 1.5 h, in argon atmosphere at 1.0 Torr at room temperature. The electrical field accelerates Ar^+ into the nickel targets and the impact causes the ejection of nickel atoms that, due to the system parameters, nucleate in nanoparticles that are deposited onto the fiber surfaces [31]. Fig. 1b shows a photograph of the device during nanoparticles deposition. After this step, MWCNTs were grown onto the carbon fibers by the modified CVD device depicted in Fig. 1c. Acetylene pyrolysis occurred in the oven region set at 700 °C while the nanoparticle coated CFs were placed in the 420–460 °C region. The gas flow carried the carbon species from the high to the low temperature region where the MWCNTs grew on the CFs. Prior to the MWCNTs growth, the temperature profile was obtained under argon flow. Then, the MWCNTs growth was carried out with 100 sccm of N_2:H_2 (90/10) and 2 sccm of acetylene at a pressure of 180 Torr for 50 min.

3. Results and discussion

In Fig. 2, it is shown the field emission scanning electron microscopy (FE-SEM) images of CFs at the different preparation stages in order to obtain the CFs cover by MWCNTs (CF-CNT). The surface morphology of CFs after the cleaning process is shown in Fig. 2a. As can be seen in Fig. 2b, the nickel nanoparticles were homogeneously distributed on the CFs. Fig. 2c and d correspond to two different magnifications of the CF-CNT, showing clearly that CFs were completely covered by MWCNTs. Fig. 2d also shows that carbon nanotubes of different morphologies, straight and helical, are grown on the fibers. We can expect that the helical morphology increases the superficial area of the MWCNTs and would lead to better adhesion between the

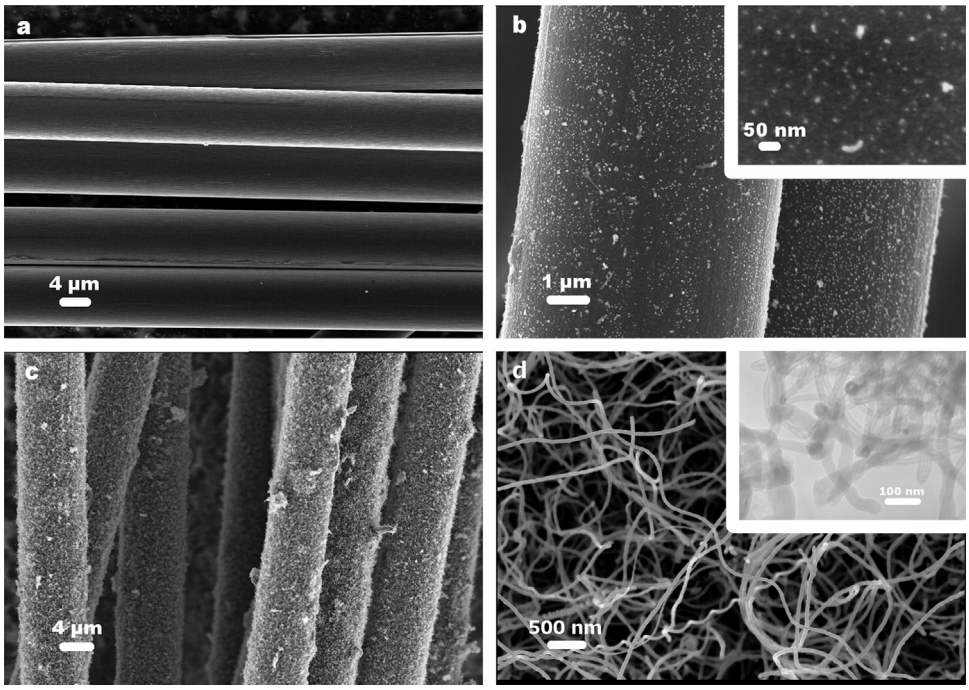

Fig. 2. FE-SEM images of (a) bare fibers, (b) nickel nanoparticles covered fibers, (c-d) carbon nanotubes grown onto the carbon fibers. In (c) can be seen as the synthesis gives origin to straight and helical nanotubes. The inset in Fig. 2(b) shows a higher magnification taken from the same sample. The inset in Fig. 2(d) shows a TEM image of MWCNTs mechanically detached from the CF-CNT.

nanotube and the polymer matrix. The inset of Fig. 2d depicts a transmission electron microscopy (TEM) image of MWCNTs mechanically detached from the CF-CNT. It's important to note that the TEM image shows the tube characteristic of the synthesized nanostructures. Measurements performed from this image, shows that the MWCNTs have outer and inner diameter between 30–40 and 6–10 nm, respectively. The broad diameter distribution is a typical characteristic of CNTs grown by catalytic CVD. As the acetylene pyrolysis occurs in the high temperature zone of the oven (700 °C) we can assure that the corrected carbon species reaches the sample and that the synthesis generates carbon nanotubes even with the relatively low temperature of the sample. Other papers in the literature that attempt the synthesis of MWCNTs by pyrolysis of acetylene at low temperatures (470 °C) gets mostly nanofibers and a few nanotubes. [27].

The Raman spectra of CF and CF-CNTs, Fig. 3a, shows the characteristic peaks of graphic carbon materials, the D peak at ~1348 cm^{-1} and the G peak at ~1581 cm^{-1}. The ratio between the intensities of the D and G peaks, $I_{D/G}$, was found to be 0,94 to the bare fibers and 0,83 to the CF-CNT. A low $I_{D/G}$ value is attributed to the presence of less structural defects, what is consistent with the presence of carbon nanotubes on the surface of the fibers. The Raman spectra shows that our methodology generates carbon nanotubes of better quality than the recent work of Du et al. [30] which also synthesize MWCNTs on carbon fibers at low temperatures.

De Greef and coworkers have studied the strength and strain-to-failure of the CFs with single-fiber tests carried out after the CVD process using Ni catalyst under Ar/H_2 atmosphere [29]. They reported a strongly decreased of these mechanical properties as the growth temperature increases above 600 °C but was negligible below it. They claimed that the decrease in mechanical properties is consequence of

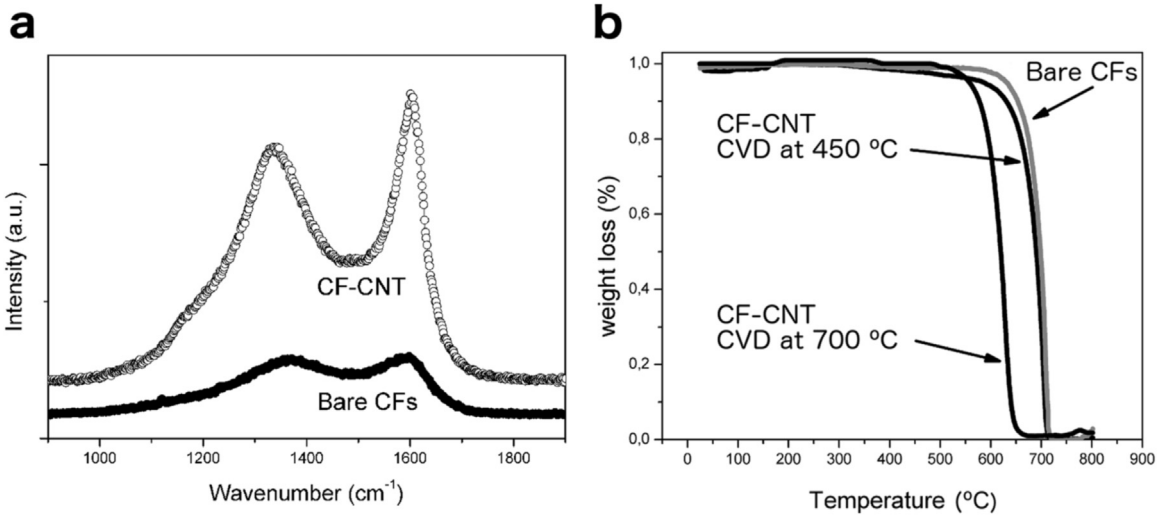

Fig. 3. (a) Raman spectra of the bare CFs and the CF-CNT. (b) Thermogravimetric analysis curves under air atmosphere of bare CFs and CF-CNT with the CNT-growth occurring at two different temperatures;.

fiber surface damages. Furthermore, by analyzing fiber strength data with Weibull statistics, they show that the defects/flaws size had increased after CNT growth at 700 °C. As it is well-known the Thermogravimetric analysis (TGA) under air atmosphere is very sensitive to the increase of the surface area because assists the oxidation of the material. Fig. 3b shows the thermogravimetric curves obtained with the bare CFs and with the CF-CNT obtained with our CVD growth process carried out at two different temperatures, one of them over the threshold temperature established by De Greef and the other below it. Our thermogravimetric analysis shows the same trend, the most significant deterioration of the CFs thermal stability occurs with subsequent annealing at the highest temperature (700 °C) for grafting CNT to the carbon fibers. Recently, using the same material for tensile test and TGA, Pozegic and coworkers [32] has shown that this correlation between tensile strength and thermal stability is valid. Then, our indirect experimental evidence indicates that the CVD process subsequent to our catalyst deposition method based on low energy DC sputtering at room temperature would not cause any noticeable degradation of tensile strength of the CNT-grafted carbon fibers.

The influence of the CNTs on interfacial bonding of the CF with epoxy resin is usually assessed measuring the interfacial shear strength through single fiber fragmentation [33,34] or pull-out test [18,30,35]. Sometimes the results of this test are also compared against fractographic analysis of fracture surface as can be seen, for example, in the work of Q. Li et al. [34]. There is clearly shown that a low value of the interfacial shear strength correlates with the pull-out of the fiber while a high value corresponds with no pull-out and a low splitting between fiber and matrix in the fracture surface. In this study, we compared the degree of interfacial bonding between the CFs, with and without grafted CNTs, and the epoxy resin through a fractographic analysis of fractured composite surfaces. Flexural failure was carried out at cryogenic temperature looking for a demanding condition over the fiber-matrix interface bonding due to the large residual stress developed from the mismatch in thermal expansion between the fibers and the resin [36]. No significant effect on the toughness of the matrix is expected due to the very low testing temperature [37]. The flexural beam samples were prepared with a single filament of “as-received” carbon fiber, or CNT-grafted carbon fiber, along the axial direction and the DGEBA (diglycidyl ether of bisphenol A) epoxy resin was chosen as the matrix. Fig. 4 shows typical micrographs of the fracture morphology observed in the single fiber beam. Large longitudinal splitting is clearly observed in the fractograph of the “as received” CF composite depicted in Fig. 4a. An obvious debonding area appears at the joint between the matrix and the “as-received” CF indicating the fracture planes initiated at the interface between fiber and matrix (Fig. 4b). The fracture surface of the composite prepared with CF-CNT (Fig. 4c and d) presented completely different morphologies with that of the “as-received” CF. The interface region follows the nanostructure of the MWCNT forests deposited onto the CFs and its size is approximately in the range of 1 μm. A relative short longitudinal splitting is also evident. Small and shallow debonding areas appeared at the joint between the fiber and the CNT nanocomposite interphase indicating that the CNTs have a higher adhesion force with the matrix and the fracture planes initiated at the interface between CNT nanocomposite and fiber. Besides offering effective bonding, CNTs next to the CF surface can further prevent fiber slipping in the matrix under the applied tensile load due to high interfacial frictional resistance.

4. Conclusions

It is reported for the first time a facile and up-scalable method for the preparation of CFs entirely covered with MWCNTs, in which all the stages of the process are performed at low temperatures. In particular, the deposition of catalytic nanoparticles was performed at room temperature. The double target DC sputtering has a key role to generate a homogeneous distribution of nickel nanoparticles onto the CFs. The growing of MWCNTs on the CFs at 420–460 °C was possible thanks to the strategy designed for this synthesis taking the advantage of the typical temperature gradient inside the conventional CVD device. The low temperature of the process leads to no significant degradation of the CF properties as revealed by the thermogravimetric stability investigations, remaining their structural integrity and inherent properties. SEM analysis of the cryo-fractured surfaces of single fiber model composites demonstrated the enhanced interfacial adhesion strength for CF-CNT/epoxy compared to bare CF/epoxy composites.

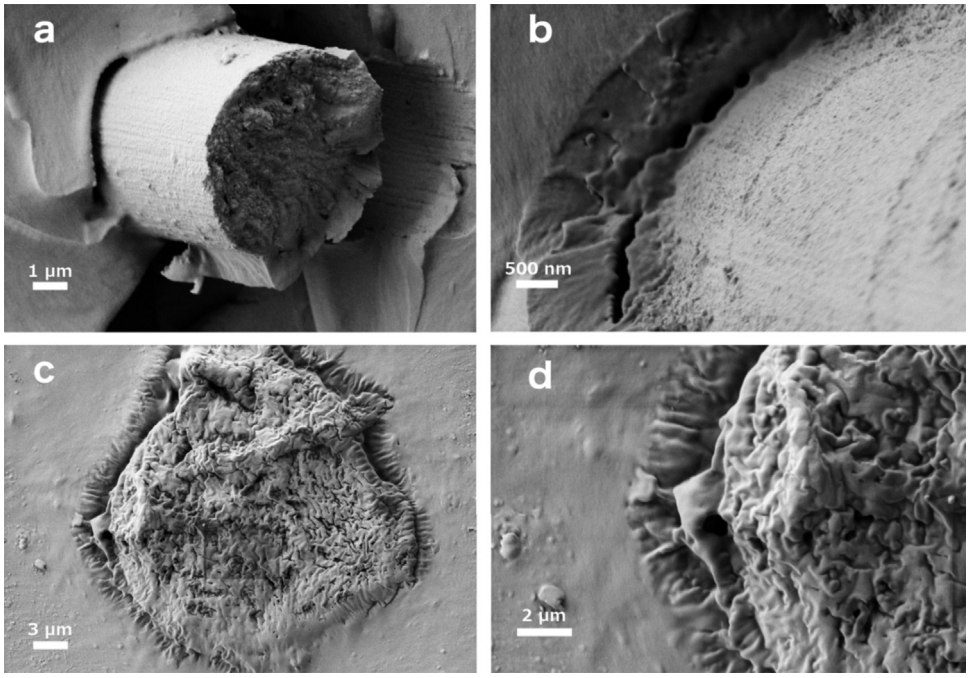

Fig. 4. Fracture morphology of the single fiber beam; (a) and (b) bare CF/epoxy composite; (c) and (d) CF-CNT/epoxy composite.

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