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Correlation between electrical conductivity and manufacturing processes of nanofilled carbon fiber reinforced composites

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ABSTRACT

This paper describes the difference on the electrical performance of carbon fiber reinforced composites (CFRCs) when two different *Resin Film Infusion* (RFI) manufacturing techniques are used. For the panel obtained by bulk infusion the measured in plane and out of plane electrical conductivities were 2.0×10^4 S/m and 3.9 S/m respectively and for the panel prepared using the traditional resin film infusion the values were 1.1×10^4 S/m and 1.7 S/m respectively. Morphological investigations on the sections of etched panels have highlighted that this difference in the electrical conductivity was strictly related to the different distribution of multiwall carbon nanotubes (MWCNTs) between the carbon fibers (CFs) of the plies.

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1. Introduction

Carbon fiber reinforced composites (CFRCs) are expected to contribute more than 50% of the structural mass of future aircrafts. Carbon fiber composites can be classified on the basis of the length (short or continuous) of the employed fibers. Continuous carbon fibers, aligned unidirectionally or forming a woven fabric have stronger effect on the mechanical, electrical, and thermal properties and give rise to composites characterized by higher anisotropy than that of found with short fibers [1]. In particular, CFRCs based on epoxy resins exhibit some rather inherent unsatisfactory characteristics, such as poor electrical conductivity. Epoxy resins are known, in fact, for their good or excellent properties covering an extensive range of applications [2–4], but at same time for their undesired electrical insulating behavior which limits their applicability as aeronautical materials. This drawback has raised concern over the performance of the composite structure during a lightning strike event due to the remarkable risk that a puncture of the structural part would cause a catastrophic failure of the aircraft.

In a traditional aircraft structure, the aluminium skin of the aircraft provides a highly conductive path for the lightning to flow around the structure without causing damage. Without a conductive path on the skin of a composite aircraft the lightning may pass through the airframe which could vaporize metal control cables, weld hinges on control surfaces and explode fuel vapors within fuel tanks. These *direct effects* also typically include vaporization of resin in the immediate strike area, with possible burn-through of the laminate. *Indirect effects* occur due to electromagnetic phenomena: high and steep-fronted transient over-voltages can damage and even destroy on board electronics or ignite potentially dangerous sparks. In addition electromagnetic interference may affect the proper functioning of the different on board electrical and electronic systems. Airplanes get struck by lightning frequently; obviously and fortunately, they are built to withstand such stresses. Modern composites are reinforced with conductive metal fibers or metal screen in order to dissipate lightning currents. But many of these solutions add additional weight and partially reduce the advantage of the composite applications. However, in the last decade, the availability of different nanofiller or nanostructured conductive materials has sensibly contributed to the continuous improvement of the engineering properties or abilities of the composites for aeronautic or automotive industries.

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By choosing the appropriate control of the material structure, as well as the appropriate fillers this critical point may be suitably overcome.

In particular, one strategy to increase the application range of epoxy resin impregnating layers of CFs is to incorporate nanoscale conductive fillers, such as carbon nanotubes (CNTs) [5–15], that are intrinsically characterized by high electrical conductivity. However, the incorporation of CNTs inside of epoxy matrices to manufacture CFRCs is not a trivial issue. In fact, even though CNTs hold much promise to impart tailored electrical conductivity to the composite panels, there are still several unresolved questions related to their distribution in the matrix and to the processability of the CNTs filled systems [16,17]. The existing liquid molding processes, such as resin transfer molding (RTM) and vacuum assisted resin transfer molding (VARTM) may be adapted to manufacture CFRCs impregnated with CNTs nanofilled resins. Unfortunately, loading percentages of CNTs larger than 0.3–0.5% by weight may lead to unacceptable high resin viscosities. In addition to the viscosity issues related to the high CNTs contents, filtration of the nanofillers by the fibrous medium may also lead to inadequate final component quality. Traditional liquid infusion process for CNTs nanofilled resin may lead to undesired filtration effects mainly due to “deep bed filtration” mechanisms causing a gradual reduction of the available flow channel dimensions. Clogging of the fibrous porous media and slowing down the resin flow front progression resulting in longer infusion cycles cause CNTs concentration gradients [18].

In this paper, in order to obtain high electrical conductivity, multi-wall carbon nanotubes (MWCNTs) were embedded inside an epoxy resin based on a mixture of tetraglycidylmethylenedianiline (TGMDA) and 1,4-butandioldiglycidylether (BDE). This particular epoxy formulation has proven to be very effective for improving nanofiller dispersion due to a decrease in the viscosity [19–22] and, in addition, it has been found to reduce the moisture content which is a very critical characteristic for aeronautic materials [19,23]. Furthermore, in order to minimize the filtration effects [24,25], the traditional liquid infusion process has been modified as described in the sequel.

The amount of MWCNTs inside the epoxy mixture used to impregnate plies of carbon fiber cloths was chosen by studying the electrical behavior of the nanofilled resin alone (without carbon fibers – CF). The electrical percolation threshold (EPT), i.e. the value of filler content ensuring the transition from insulating to conducting behavior of the composite, was found to be in the range [0.1–0.32% wt]. Also the ac measurements confirmed that the EPT ranges between 0.1 and 0.32% wt. An amount of 0.5% wt, beyond the EPT has been, then, adopted to prepare the nanofilled epoxy mixture used to manufacture the carbon-fiber reinforced panels through two different techniques: a traditional infusion approach and an unconventional process using inspired by Resin Film Infusion technique allowing to minimize the filtration effects *via* a better compaction and reduced resin flow paths. In this last case, a thick wet film of resin has been placed under the carbon preform (400 mm × 400 mm) made laminating 7 plies of carbon fiber cloths, in a vacuum bag, without any tube connection with external reservoirs, so that the resin is forced through the shortest possible path for infiltration, reducing at the minimum the necessary time and filtering problem. Using this last approach, the temperature can be better controlled during the infiltration, without any gradient along the resin path. Moreover, during the final phase of infiltration and during the curing it is possible to add autoclave pressure to better compact the preform, squeezing out the excess of resin, once the preform is full impregnated under vacuum. It is found that the reduced flow path strongly affect the nanotubes dispersion between the CF plies.

2. Experimental

2.1. Materials

2.1.1. Nanofilled resin

The epoxy matrix was prepared by mixing the epoxy precursor, tetraglycidylmethylenedianiline (TGMDA) (epoxy equivalent weight 117–133 g/eq), with an epoxy reactive monomer 1-4 butanedioldiglycidyl ether (BDE) that acts as a reactive diluent. The curing agent used for this study is 4,4-diaminodiphenyl sulfone (DDS). The epoxy mixture was obtained by mixing TGMDA with BDE monomer at a concentration of 80%:20% (by wt) epoxide to flexibilizer. The curing agent was added at a stoichiometric concentration with respect to all the epoxy rings (TGMDA and BDE), this mixture will be named hereunder T20BD formulation.

The MWCNTs (3100 Grade) were purchased from Nanocyl S.A. Transmission electron microscopy (TEM) investigation has shown for MWCNTs an outer diameter ranging from 10 to 30 nm. The length of MWCNTs is from hundreds of nanometers to some micrometer. The number of walls varies from 4 to 20 in most nanotubes. The specific surface area of MWCNTs determined with the Brunauer–Emmett–Teller (BET) method is around 250–300 m^2/g; the carbon purity is >95% with a metal oxide impurity <5% as it results by thermogravimetric analysis. Epoxy blend and DDS were mixed at 120 °C and the MWCNTs were added and incorporated into the matrix *via* ultrasonication for 20 min. An ultrasonic device, Hielscher model UP200S (200 W, 24 kHz) was used. The epoxy mixture T20BD was filled with MWCNTs at 0.5% concentration by wt. This nanofilled sample will be named hereunder T20BDCNTs formulations. This concentration was chosen because the curve of dc volume conductivity vs. MWCNTs concentration (the percolation curve shown in Fig. 1) highlighted that the electrical percolation threshold (EPT) is lower than 0.32%, therefore for this amount of MWCNTs the nanofilled formulation is beyond the EPT [23–26]. The value of the dc conductivity corresponding to the selected 0.5% wt concentration is 0.03 S/m. The formulation T20BDCNTs is also characterized by good dynamic mechanical properties. In fact, it is found that the incorporation of a small concentration of MWCNTs (0.32%) in the temperature range of −60 ÷ 180 °C causes an increase in the elastic modulus value with respect to the modulus of the unfilled epoxy matrix. Values between 3600 ÷ 1800 MPa are found in the temperature range of −60 ÷ 180 °C. The nanofilled formulation is characterized by values of glass transition temperature (Tg) higher than 180 °C [23,27].

2.2. CFRCs –manufacturing process

2.2.1. Infusion –panel manufactured using traditional approach

In the classical scheme of infusion the resin is heated in a reservoir connected to the vacuum bag by a small diameter nylon tube (8 mm) (see Fig. 2). Under the vacuum bag, on the preform, a distribution medium is used to facilitate the flow of the resin on the surface on the panel. In this way the resin has no difficulties to reach every point on the surface, while the vacuum in the preform pores attracts the resin along the shortest normal path (few mm).

The usual values of viscosity accepted in the typical infusion process is approximately 300 mPas, and the injection temperature does not exceed 80 °C.

Rheometric analysis of the nanofilled formulation T20BDCNTs shows that an acceptable value of viscosity is reached at quite high temperature (100 °C), which is unusual, but still feasible with disposable materials normally used for infusion.

This classical liquid infusion approach was followed using a sequence of different steps. First, a water based PTFE release agent

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