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Fatigue delamination of a carbon fabric/epoxy laminate with carbon nanotubes

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ABSTRACT

This paper describes the results related to the crack growth rate measurement on double cantilever beam (DCB) specimens made of a carbon-fibre fabric-reinforced multifunctional epoxy composite. Two plates were evaluated where the resin was enhanced using a combination of 0.5% carbon nanotubes (CNTs) and Glycidyl POSS (GPOSS) flame retardant for one of the plates. Loading in mode I was displacement-controlled with $R = 0.1$, $f = 4$ Hz and a constant maximum strain energy release rate $(G_I)_{max}$ during the cycle. The value of $(G_I)_{max}$ was defined to be from 20% up to 60% of the mode I interlaminar fracture toughness. The specimens enhanced by CNTs and GPOSS highlighted a significant decrease in the fatigue crack growth rate of approximately 80%. The crack growth rate was also observed to be significantly related to the interface of the weft and warp tows of the plain weave.

1. Introduction

The advent of carbon-fibre composite passenger aircraft has been driven primarily by the need to reduce structural weight and hence environmental impact [1]. Indeed a reduction of 1 kg of saved fuel equates to a reduction of 3.15 kg of CO_2 emissions. In this regard, the European Union has set ambitious aircraft emission reduction targets by 2050 as the level of commercial air traffic is set to continue doubling every fifteen years. The very specific strength and stiffness, plus corrosion- and fatigue-resistance of carbon-fibre composite materials make them highly suitable for lightweight aerostructures. Carbon-fibre composites also have low electrical conductivity which necessitates the need for additional measures to ensure adequate lightning strike protection. The industry has adopted the use of a fine metallic mesh incorporated into the aerodynamic surfaces. This approach adds necessary weight to the structure, as well as increasing manufacture and maintenance complexity. Composite materials also have low thermal conductivity which impacts on the design of anti-icing systems. In recent years, a number of research groups have explored the unique properties of nanoparticles dispersed in resin, or introduced between-lamina interfaces, to address these limitations.

Another critical point related to the use of composites in aeronautics is due to the poor flame-resistance of epoxy resin. Unfortunately, the epoxy resin between the carbon-fibre layers can burn under accidental aircraft fire conditions. For this reason, Federal Aviation Administration (FAA) certification for the Boeing 787 required demonstrating that the level of fire safety in the B-787 was equivalent to a conventional transport (aluminium) aircraft. This regulation has been extended to all other structural aeronautic materials. It is evident that no new material can be developed in this field without considering its behaviour in flame conditions.

Effective solutions can be found in nanotechnology as a result of the fascinating possibility of transferring some of the interesting nanostructure properties to specific composites. Several authors have observed that nanofillers in the resin improved both the electrical and flame-resistance properties [2,3]. Carbon nanotubes (CNTs) embedded inside the epoxy resin to enhance electrical conductivity are found to significantly affect the fire properties of epoxy systems by preventing the epoxy systems from forming intumescent charring [4]. Incorporation of flame retardants is also possible. The authors in Ref. [4] also highlighted that the incorporation of 5 wt% glycidyl polyoctahedral silsesquioxanes (GPOSS) into the epoxy matrix resulted in a limiting oxygen index (LOI) value of 33, compared to an LOI value of 27 for the pure epoxy mixture.

The addition of CNTs to polymers reinforced with continuous

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microfibres only has an effect on those composite qualities that are controlled by the properties of their matrix. The stiffness, strength, and strain to failure in the fibre direction are only slightly affected. Some authors have introduced CNTs into the composite and have improved the shear strength [5], impact resistance [6], fracture toughness [7], and fatigue characteristics [8]. These attributes support the reliability and effective use of the composite in a new generation of aerospace structures. When the nanotubes are stirred correctly and are not agglomerated, a polymer matrix has better adhesion to the fibres, which could be the primary reason for the improvement of the previously listed attributes. The CNTs create a new component in the fibre: a matrix system that makes the synergic effect more apparent. However, the nanotube walls have an extremely small surface energy, which creates problems for the creation of bonds with the polymer. The creation of nanotube agglomerations, which is associated with Van der Walls forces and the high length to diameter ratio of the nanotubes, is a problem that makes the demanding effect difficult to attain [9]. A possible solution is the functionalization of nanotubes, which means a chemical change in the CNT surface. The presence of butanedioldiglycidyl ether (BDE) in the epoxy mixture has proven to be very effective for improving nanofiller dispersion, owing to a decrease in the viscosity [10,11], and in addition, it has been found to reduce the moisture content, which is a critical characteristic of aeronautic materials [12].

This study attempts to investigate fatigue crack growth behaviour of carbon fibre–reinforced polymer (CFRP) plates that are typically used in aerospace structures, considering CNT and flame-retardant additives. The crack growth was observed on double cantilever beam (DCB) specimens loaded in mode I using sinusoidal cycles. The modified compliance calibration (MCC) method was used for detailed crack length evaluation. Fracture surface analysis was used to support the investigation. The response of the crack growth rate on the fracture interface was also investigated. The understanding of crack growth rate in carbon fabric reinforced composites is crucial, especially when adopting the damage tolerance philosophy for use in an aircraft structure.

2. Material and methods

2.1. Material properties

Two CFRP plates were provided for the experiments. The resin was enhanced using 0.5% of multi-walled carbon nanotubes (MWCNTs) and a flame retardant for one of the plates. The epoxy matrix used to manufacture the panels was prepared by mixing an epoxy precursor, tetraglycidylmethylenedianiline (TGMDA), with an epoxy reactive flexibilizer 1–4 butanedioldiglycidyl ether (BDE) at a ratio of 80:20 (by wt.).

The flame-resistance of the epoxy mixture was improved by using 5 wt% of GPOSS, which is a viscous liquid that is functionalized with oxirane rings and belongs to the class of polyhedral oligomeric silsesquioxane compounds [4]. Epoxy blend and diphenyl diaminosulfon (DDS) were mixed at 120 °C and MWCNTs were added and incorporated into the matrix using ultrasonication for 20 min. The epoxy mixture used to manufacture the panels was filled with 0.5 wt% of MWCNTs, for which the mixture is beyond the electrical percolation threshold [13]. This formulation was used to manufacture CFRCs using resin film infusion (RFI) with an unusual technique to infuse a nanofilled resin into a dry carbon-fibre preform. This new technique allows us to overcome drawbacks related to non-optimal values of the viscosity for the RFI process.

The laminate was made with 24 plies of a plain weave carbon-fibre fabric (SIGMATEX (UK) LDT 193GSM/PW/HTA40 E13 3K), with a sequence of lamination (0/90°) such that the fabric was quasi-isotropic and balanced. A non-adhesive Polyether ether ketone (PEEK) foil insert 10 μm thick was inserted at the midplane during lay-up at a distance of 60 mm from the specimen edge. Table 1 shows selected measured properties of the material used. The fibre, resin and void contents were measured using ASTM D3171 [14] on each plate, where one sample was extracted from the middle of each plate. After the specimen extraction from the plates using a diamond blade, two aluminium loading blocks were bonded to each specimen end on the insert side.

2.2. Specimens and test procedure

Testing was performed on DCB specimens with a width B of 20 mm and a length of 185 mm. Both edges of the specimen were painted using a thin white colour and were marked using lines with an interval of 5 mm. The specimens were clamped using bonded aluminium blocks into a INOVA ZUZ 100 loading machine, with a LTC-116-0.1 load cell, which has a capacity of 1 kN and is appropriate for dynamic loading (Fig. 1). The end of the specimen was supported by a flexible wire to maintain the specimen in the horizontal direction, and to reduce any additional forces that could originate from the specimen weight.

A pair of video cameras each having a resolution of 2048 × 1536 pixels, and measuring at 12.5 frames per second, focused on the edges to cover approximately 15 mm of the specimen length. The theoretical resolution was 7 μm/px. However, the precision of the crack measurement from the front was affected by partial fibre bridging in certain areas. The time of the cameras and the loading machine was synchronized manually before measurement.

The test was performed at room temperature. The ASTM D6115 [15] is the only released standard considering fatigue delamination in CFRP, and it focuses only on the growth onset. Therefore, the fatigue test procedure was based on a draft standard for fatigue delamination crack growth that is being prepared for ASTM International. This procedure is well described in Ref. [16]. The initial load was applied with a constant crosshead displacement of 5 mm/min until δ_{max}, according to Table 2, was reached. Next, the specimen was unloaded. Using a simple data fit of the linear part of the load-displacement curve, the displacement was zeroed at a new value that corresponded with the fitted zero force. This step was performed because the displacement-controlled cycling could cause negative force values for very low displacements when the displacement was not zeroed properly. The crack propagation during the following loading was monitored using video cameras (Fig. 1). Sinusoidal loading with displacement control set to δ_{max} and δ_{min}, according to Table 2, and a frequency of 4 Hz was applied. Load P and displacement δ were recorded by the INOVA machine, at a frequency of 100 Hz. The recorded files typically had 10^7 rows of data and were processed using a MATLAB script that evaluated maximum force in each cycle, and then averaged the maximum force over approximately 100 cycles to reduce the number of data points.

The crack propagation length for the individual specimens used for the compliance calibration was determined from the snapshots of the video recording. The length was measured from the picture using a custom-made program with a virtual gauge. ASTM D5528 standard [17] was used to evaluate the results. The modified compliance calibration (MCC) described in the standard was used for data reduction. This method was selected because it considers a specimen thickness that was slightly different for the two series. The constants, A_1 and k from Eq. (1), were determined for individual specimens using a least-square fit applied to the plot of the optically observed delamination lengths a versus the cube root of the corresponding compliance C. For each specimen, the constants were used specifically for one specimen. The relationship between