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Radiation curing of carbon fibre composites

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HIGHLIGTHS

- Epoxy/carbon fibre composites were produced by means of e-beam irradiation.
- DMTA analysis pointed out a nonuniformity in the cross-linking degree of the material.
- An out-of-mould post irradiation thermal treatment allows a higher uniformity.
- Mechanical tests were interpreted on the basis of the cross-linking density and fibre/matrix interaction.

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ABSTRACT

Epoxy/carbon fibre reinforced composites were produced by means of e-beam irradiation through a pulsed 10 MeV electron beam accelerator. The matrix consisted of a difunctional epoxy monomer (DGEBA) and an initiator of cationic polymerisation, while the reinforcement was a unidirectional high modulus carbon fibre fabric. Dynamic mechanical thermal analysis was carried out in order to determine the cross-linking degree. The analysis pointed out a nonuniformity in the cross-linking degree of the e-beam cured panels, with the formation of clusters at low T_g (glass transition temperature) and clusters at high T_g. An out-of-mould post irradiation thermal treatment on e-beam cured samples provides a higher uniformity in the network although some slight degradation effects. Mode I delamination fracture toughness and Interlaminar Shear Strength (ISS) were also investigated by means of Double Cantilever Beam (DCB) and Short Beam Shear tests, respectively. Results from this mechanical characterisation allowed to correlate fracture toughness of the bulk matrix resin, cross-linking density and fibre/matrix interaction to the delamination fracture behaviour of the fibre reinforced material.

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1. Introduction

Radiation curing can be an alternative way to the conventional thermal curing processes to produce advanced carbon reinforced fibre (CFRP) composites (Crivello, 1999). The potential advantages are the short processing times, the possibility to carry out the process without the use of organic solvents and at moderate temperatures. These behaviours make radiation curing an environmentally friendly process. Furthermore the possibility to work at room temperature allows to produce materials with better mechanical properties due to the absence of thermal induced stresses (Lopata et al., 1999; Goodman and Palmese, 2002; Berejka and Eberle, 2002; Sui et al., 2009; Alessi et al., 2010).

Properties of carbon fibre composites depend on the properties of the polymer matrices, of the carbon fibres and on the quality of the interactions between the matrix and the fibres.

As for matrices, a fundamental role is played by their thermal and mechanical behaviour. In fact the polymer matrices need to exhibit high thermal resistance, high elastic modulus and high toughness. As a rule the thermal resistance is measured by the glass transition temperature of the cured materials, which for aeronautic/aerospace applications has to exceed 170 °C. The values accepted for the elastic modulus is higher than 3 GPa, while the toughness, measured through the critical stress intensity factor, has a target of 1.8 MPa $m^{1/2}$ (Janke et al. 2001).

Extensive studies carried out on thermal curing of polymer matrices allowed to obtain systems able to reach the above reported performances. In fact through the choice of apt formulations of epoxy/engineering thermoplastic blends it was possible to obtain at the same time an optimal cross-linking and morphology (Alessi et al., 2007a, 2007b; Kim et al., 1995; Mimura et al., 2000).

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In the last years similar studies have been carried out regarding the radiation curing process (Fengmei et al., 2002; Sui et al., 2003). For example, investigations recently carried out by some of the authors allowed to determine the influence of both the chemical formulation and the irradiation parameters on the structure, morphology and properties of radiation cured composites matrices (Alessi et al., 2007a, 2007b, 2012) and other studies were carried out regarding the effects of hydrothermal ageing on thermal and mechanical properties (Alessi et al., 2010). In particular it has been shown that it is possible to tune the processing conditions in order to optimise the molecular structure, the morphology and the thermal and mechanical properties of the cured polymer.

Unfortunately, together with the advantages above discussed, radiation curing of structural composite materials presents some significant drawbacks which still do not allow this technology to successfully compete with more traditional thermal treatments. Besides the noncommercial availability of specific formulations and carbon fibre sizing for radiation processing, and also considering the elevated implant costs, another inconvenience comes from the use of low processing temperatures, although this is considered one of the most important issues of radiation curing. In fact during irradiation at room temperature, as could be the case when working at moderate dose rates, the glass transition temperature of the polymerising system increases up to the processing temperature. In these conditions vitrification phenomena occur and the reaction kinetics is controlled by the diffusion of the reactive species, giving rise to a non-fully cured material. It has been shown (Alessi et al., 2007a, 2007b) that, in order to overcome this problem, the best processing conditions consist of a "dual cure" process; *i.e.* radiation curing at moderate temperature followed by a post-irradiation out-of-the-mould thermal curing at high temperature on the already radiation polymerised material.

As observed before, the final properties of a carbon fibre reinforced composite also depend on the properties of the fibres and on the quality of the fibre–matrix interactions. Fibre/matrix adhesion strength in particular has been found to play a strong influence on matrix dominated composite failure mechanisms (Drzal and Madhukar, 1993; Hoecker et al., 1995), and the Interlaminar Shear Strength (ISS) in particular has been identified as a macroscopic property exhibiting a strong correlation with the microscale fibre/matrix interfacial adhesion strength (Drzal and Madhukar, 1993). The Short Beam Shear (SBS) test (ASTMD2344/D2344M) is the most popular characterisation procedure to obtain the ISS (Eberle et al., 2005), due also to its simple set-up.

The ISS values for radiation cured composites have been found to be usually lower than the corresponding values for thermally cured systems (Eberle et al., 2005; Zhang et al., 2002). One reason for this has been identified in the lack of performance from sizing treatments used with commercial grade fibre reinforcements. In fact these have been optimised along the years to suit thermal curing processes, and there is a lack of knowledge and still few studies available on the fibre/matrix interactions with radiation curing (Zhang et al., 2002; Vautard et al., 2011). Moreover, as said above, the low processing temperature of radiation process may cause further constraints due to the high viscosity of the resin at low temperature, especially when high molecular weight toughening thermoplastic components are present. In these conditions in fact there is an inefficient fibre wetting by the resin blend.

In this work epoxy/carbon fibre composites have been synthesised by radiation curing and their thermal and mechanical characterizations have been carried out. Results indicate that the optimisation of the thermal behaviour can be achieved by a dual cure process, as already observed for the epoxy matrix (Alessi et al., 2007a, 2007b). Furthermore Mode I delamination and Interlaminar Shear tests have evidenced that the radiation cured materials suffer from bad fibre–matrix interactions and, also in this case, a beneficial effect of the post-irradiation thermal curing is evidenced.

2. Experimental

Flat laminate panels, with approximate dimension 20×25 cm^2, have been prepared by e-beam curing and some of them have been subjected to a post irradiation thermal curing described in the following. The epoxy monomer was 2,2-bis[4-(glycidyloxy) phenyl]propane (DGEBA) supplied by Aldrich and the initiator was an iodonium salt, cumyltolyliodonium tetra(pentafluorophenil) borate (Rh 2047), supplied by Rhodia Silicones.

A carbon fibre fabric, SikaWrap®-400C Mid Mod, supplied by SIKA Italia, is used where unidirectional fibre bundles are plain woven with a low number of weft threads. The fabric areal density is 400 g/m^2 and is made of high modulus carbon fibres (nominal Young's modulus $E_f = 390$ GPa).

The CFRP panel samples manufacture consisted of two main steps: hand lay-up impregnation and radiation curing by exposing the whole mould to the pulsed e-beam.

The laminates lay-up is $[0^\circ]_8$, and impregnation was carried out on an aluminium base plate previously treated with a liquid wax to act as a releasing agent. Another aluminium plate covered the laminate, and the two plates were pressed together with side clamps in order to guarantee obtain a uniform thickness and good surface finish. The light pressure applied by the side clamps was though just enough to obtain relatively low values of fibre volume fraction (V_f), about 34–40%, typical of low pressure consolidated composite laminates (Siddiqui et al., 2007). In order to obtain Double Cantilever Beam samples for Mode I delamination tests a thin rectangular aluminium patch was placed perpendicular to the fibres, between the two central layers, in order to obtain an initial interlaminar crack-like delamination.

E-beam irradiation of samples has been performed on the LAE 10 MeV linear, pulsed accelerator located in the laboratory of the ICHTJ (Institute of Nuclear Chemistry and Technology) in Warsaw. The aluminium mould containing the impregnated uncured composite laminate was fixed on a conveyor, in a horizontal position, under the pulsed electron beam gun. During irradiation the mould moved back and forward under the swiping electron beam. The temperature of the sample during irradiation was monitored through a thermo-resistor dipped into the resin.

The total irradiation dose was 80 kGy and the average dose rate was 100 kGy/h. Some radiation cured samples have been subjected to a post irradiation thermal curing at 140 °C for 2 h.

The cured materials have been characterised through dynamic mechanical thermal analysis (DMTA) and delamination fracture toughness tests.

DMTA tests have been carried out on small beam samples ($30 \times 8 \times 4$ mm^3) cut from panels using a Rheometrics DMTA V instrument with a single cantilever bending setup, in temperature swift mode between 25 and 250 °C and a heating rate of 2 °C/min. The frequency was set at 1.8 Hz and the strain was 0.02%. The glass transition temperature, T_g, for each system was defined as the temperature corresponding to the peak of the tan δ curve.

Two types of beams samples were considered the first with the unidirectional fibre direction aligned with the beam axis, longitudinal sample, and the second with the fibres direction oriented orthogonally to the beam axis, transversal sample. The curves reported here refer to transversal samples.

Interlaminar fracture toughness was studied through Double Cantilever Beam (DCB) tests. DCB samples were obtained from the four manufactured panels by cutting rectangular stripes of nominal dimensions $width \times length = 20 \times 140$ mm^2 along the reinforcement (warp) direction. Metallic hinges were glued at

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