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An analytical model for B-stage joining and co-curing of carbon fibre epoxy composites



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1. Introduction

The aviation industry is employing evermore carbon fibre reinforced thermosetting polymer (CFRP) structures in order to minimise weight, and hence emissions during flight. However, much of the efficiency that can be gained by using such materials is lost due to inefficient joining procedures and strict certification standards that prevent adhesive joining of primary structures in civil aviation [1]. A solution may be to partially cure two subassemblies, combine them and complete the cure cycle for both; termed co-curing for this study. By better understanding the resin kinetics and developing procedures that allow co-curing, cost effective manufacturing steps may be utilised e.g. staged curing [2,3], to effectively cure and locally reinforce structural composite materials with thermoset (epoxy) matrices.

A melding approach has been reported by Griffiths and Corbett [4,5], whereby a small region of the part remains in a B-stage cured state by keeping said region at a lower temperature than the structure, thus at a low degree of cure, α , in the joining region whilst the rest of the part is fully cured. However, significant machine infrastructure would be required to produce such structures, also described by Bond [6], which may be expensive to implement. Co-cured joints also provide higher fracture toughness [7] and joint strength [8] than co-bonded joints due to mechanical intermin

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ABSTRACT

The objective of this paper was to develop cure kinetic models to describe the B-stage curing and cocuring assembly of carbon fibre reinforced thermosetting polymer (CFRP) composites. Starting from the analytical model, temperature cycles and experimental procedures are developed to join a B-stage CFRP part to a reinforcing B-stage CFRP patch for local reinforcement. Our results show that by using the analytical model, one may precisely describe the cure reaction and join the composites without any additional adhesive. The co-cured composites were successfully manufactured with stable fibre volume fractions and glass transition temperatures between the two sub-components. Additionally, merits of the process, such as modifying reinforcing areas locally, or formation of net shape detail are discussed. © 2016 Elsevier Ltd. All rights reserved.

gling and covalent bonding of the resins at the interface [9], adding value to the approach. Further, their mechanical properties have been modelled as they evolve during the curing process [10], hence important knowledge of how properties evolve with cure are available. Application of the co-curing approach for bigger structures requires dedicated tooling, in order to control temperature and resin flow locally [11,12]. Moreover, by producing the structure and local reinforcements separately, valuable freedom is achieved to enhance properties locally, e.g. in the amount of toughener [13,14] that may be introduced, or the reinforcing area or ply thickness that is chosen [15].

Cure kinetic modelling of thermoset resins is widely used and has been described in [16–24]. Most works focus on wide temperature ranges, model optimisation for a wide range of degree of cure or address approaches to control heat exotherm in modern formulations [25]. However, during the curing of a composite structure, the temperature varies in a few degrees around an optimum temperature. Until now such models have had limited possibility for Bstage curing and co-curing to be employed in a robust manner. Moosburger et al. [26] have demonstrated that partial cure of a tetrafunctional epoxy resin maintains its remaining chemical reactivity, the network formation is however slightly influenced by the cure history, hence careful study is needed to fully develop the epoxy network with an interrupted curing process. To our knowledge, no such study has addressed the use of cure modelling to join structures without adhesives.

This work demonstrates an approach for using the developed kinetic model to describe the B-stage curing and co-curing assem-





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bly of local reinforcement. Starting from the model, temperature cycles are developed to join a B-staged CFRP patch to a B-staged CFRP part by co-curing whilst carefully monitoring the evolution of degree of cure and glass transition temperature, $T_{\rm g}$. The example shown in this work explains a methodology to locally reinforce a bearing joint for enhanced bearing load, see Fig. 1.

A simple methodology is presented, that allows the combination of multiple manufacturing processes. The results presented may be directly employed to co-cure structures with various thermosetting resins in an efficient and cost effective manner.

2. Materials

The resin used is "HexFlow RTM6", Hexcel, UK. It is a monocomponent resin system consisting of a tetrafunctional epoxy component, tetraglycidyl 4-4′ diaminodiphenylmethane (TGDDM, Araldite MY 721), and two amine hardeners: 4,4′-Methylene-bis(2,6-diethylanaline) (MDEA, Lonzacure) and 4,4′-Methylene-bis(2, 6-diisopropylaniline) (MMIPA, Lonzacure). The resin is a hot curing system with a high T_g developed for infusion and RTM processes.

CFRP parts were manufactured by a RTM process from a biaxial stitched non-crimp fabric, "ECS6090-Series HTS 40", Saertex GmBH & Co., Germany, with an area weight of 256 g/m². Quasi-isotropic parts were made from 16 plies: $2[0/90, +45/-45, 90/0, -45/+45]_s$ to produce $150 \times 90 \times 3.8$ mm plates.

The reinforcing CFRP patches were manufactured from a thin ply material, 20 mm tape, 80 g/m^2 high tensile strength carbon fibre "TeXtreme Spread Tow", Oxeon AB, Sweden. Quasi-isotropic, 2 mm thick, 80 mm diameter, \varnothing 10 mm hole patches were made from 24 layers of the spread tow with the layup 3[45, 90, -45, 0]_s.

A nylon peel ply (Econostich, Aero Consultants AG, Nänikon, Switzerland) was used to prepare surfaces for co-curing.

3. Experimental

The differential scanning calorimetry (DSC) measurements were made using a "Q1000 Differential Scanning Calorimeter", TA Instruments, Delaware, USA. Typically, 2–3 mg samples and Nitrogen (flow rate of 50 ml/min) was used as a purge gas. The heat of reaction, ΔH , was calculated with a linear baseline for the integration and T_g was measured as the point of inflexion in the heat flow versus temperature curve.

3.1. Resin cure kinetics

Isothermal and dynamic DSC measurements of the uncured resin were made as reference data for the analytical modelling.

B-staging partially cure of part and patch Part Part Patch Co-curing Co-curing Co-curing completion of cure of part and patch Pressure Heat Co-curing Co-

The isothermal heat of reaction, $\Delta H_{\rm iso}$, was evaluated from the isothermal measurements as

$$\Delta H_{\rm iso} = \int_0^{t_{\rm c}} \frac{dH}{dt} \tag{1}$$

where $\frac{dH}{dt}$ is the heat flux and t_c is the curing time. A second dynamic measurement at 10 °C/min from 0 °C to 300 °C was made to obtain the residual heat of reaction, $\Delta H_{\rm res}$, of the same sample. By adding the two values, the total heat of reaction, $\Delta H_{\rm tot}$, was calculated.

$$\Delta H_{\rm tot} = \Delta H_{\rm iso} + \Delta H_{\rm res} \tag{2}$$

The dynamic heat of reaction, ΔH_{rate} , was obtained from the integration of the dynamic measurements, as

$$\Delta H_{\text{rate}} = \int_0^{t_c} \frac{dH}{dt} \tag{3}$$

Isothermal measurements were conducted at 120-, 140-, 160-, and 180 °C, and two measurements per temperature were made. Dynamic measurements were made with a constant heating rate of 1-, 2.5-, 5-, and 10 °C/min until the cross linking reaction was complete. The ultimate heat of reaction, ΔH_{ult} , corresponding to a degree of cure, α , of 1 was determined from the dynamic DSC measurements at 2.5 °C/min. The evaluation of α with time or heating rate was then calculated as follows:

$$\alpha = \frac{\int_0^t \frac{dH}{dt}}{\Delta H_{\text{ult}}} \tag{4}$$

3.2. B-stage degree of cure

A series of temperature modulated DSC (MDSC) measurements were used to measure α and $T_{\rm g}$ of the partially cured resin. This was especially useful as the residual reaction immediately followed the glass transition. The temperature was modulated with ±0.3 °C every 15 s with a heating rate of 10 °C/min up to 300 °C.

The exothermic peak in ΔH_{res} of the nonreversible heat flow was then linear integrated. With a known value of ΔH_{ult} (as measured using the dynamic DSC measurements) α was calculated as

$$\alpha = 1 - \frac{\Delta H_{\text{res}}}{\Delta H_{\text{ult}}} \tag{5}$$

The value of T_g of the partially cured samples was measured by taking the temperature at the point of inflexion in the reversible heat flow versus temperature curve using pure resin samples or resin samples taken from the produced composites after B-stage curing.

Fig. 1. Shows the concept of patch reinforcement of a structural part via co-curing of B-stage cured components. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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