



Study on thermoplastic-modified multifunctional epoxies: Influence of heating rate on cure behaviour and phase separation

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ABSTRACT

The effect of heating rate on the cure behaviour and phase separation of thermoplastic-modified epoxy systems was investigated. Polyethersulphone (PES) modified multifunctional epoxies, triglycidyl-aminophenol (TGAP) and tetraglycidyl-diaminodiphenylmethane (TGDDM), as well T300/914 prepreg were used. It was shown that heating rate had a significant influence on the cure kinetics and phase structures of investigated systems. Greater heating rate causes higher epoxy conversion. The domain size of the macrophases formed from phase separation increases with the increase of heating rate. A more complete phase separation is achieved by fast heated thermoplastic-modified epoxy blends.

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1. Introduction

Epoxy resins have been widely used in aerospace, construction and automotive industries owing to their excellent mechanical properties, thermal stability, solvent resistance and good processability [1]. Especially, the excellent adhesion to carbon fibres and the low shrinkage on curing provide epoxy resins advantages over other resin systems for being used as the matrix materials for advanced composites. However, the cross-linked structure results in brittleness of epoxies which limits their application in circumstances where higher toughness is required. A variety of approaches have been developed to solve this problem including the incorporation of inorganic fillers such as silica, barium titanite, dolomite, glass beads and aluminium hydroxide [2]; the use of rubber toughening agents, e.g. amino- and vinyl-terminated butadiene-acrylonitrile, carboxyl-terminated butadiene-acrylonitrile; the addition of thermoplastic toughening agents such as polyethersulphones [3,4], polyetherimides [5], polyester [6], polyaryletherketone [7], polyhydantoin [8], etc. While rubbers can be very effective in increasing the fracture toughness, they suffer from drawbacks of reducing the modulus and the glass transition temperature. Thermoplastic toughening has been used widely in industry because it enhances toughness without compromising other desirable properties significantly [7–11].

Reaction induced phase separation of an initially miscible thermoplastic-epoxy monomers mixture is a major mechanism of

thermoplastic toughening [12,13]. Understanding the phase behaviour of thermoplastic-modified epoxy systems is essential when the improvement of the mechanical property is the final purpose. By controlling various parameters such as thermoplastic concentration, molar mass, curing temperature and reaction rate, it is possible to generate various morphologies thus achieving different mechanical properties [14,15]. A few works were conducted to investigate the influence of cure rate on the phase separation mechanism of thermoplastic-modified epoxy systems [15–17]. Jo et al. applied a simulation approach to study the effect of reactivity on the phase separation behaviour [16]; Girard-Reydet et al. selected cure temperature as a parameter to change the cure rate [15]; and Peng changed the cure temperature and the amount of cure agent to modify the cure rate [17]. This work has used heating rate as a variable to investigate the cure rate effect on the cure kinetics and the final morphology of phase-separated blends.

Epoxy resins are the most important matrices used for aerospace grade carbon fibre reinforced polymer composites. The manufacturing of carbon/epoxy composites for high performance applications has been dominated by autoclave technique. Relatively slow heating rate (1–2 °C/min) is used by the autoclave processing to ensure uniform heating of the composite components and to reduce the risk of runaway exotherm of the epoxy matrix composites [18]. There has been an increasing need to develop out-of-autoclave composite manufacturing technologies aiming to reduce processing time and energy consumption whereas still produce similar quality to aerospace grade composites. Microwave [19,20] and radio frequency [21,22] heating provide faster cures of epoxies and composites with reduced energy consumption due to

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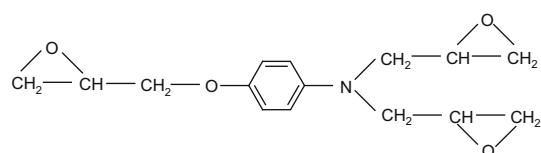
the intrinsic nature of the internal heating. However, poor process control and the non-uniform heating resulted from non-uniform distribution of electromagnetic field limit their applications in composites production industry. An alternative to achieve faster heating rate is using liquid as a heat transfer medium to conduct heat since liquid has a significantly higher coefficient of heat transfer than gas. Quickstep process was invented and has been developed based on this concept. It applies a fluid-filled, balanced pressure, heated floating mould technology for the curing, partial curing and joining of composite materials. Industrial heat transfer fluids such as polyalkylene glycols are used for transferring heat to a composite part which is trapped between a free floating rigid (or semi-rigid) mould. Heating rate as high as 10 °C/min can be achieved with limited exotherm by utilising this technology. The details of this technology can be found elsewhere [23,24].

Currently, the aerospace industry is urged to accelerate the production speed and to reduce the composite product costs due to the increasing demand of composite structures in aircrafts. The research in material design and manufacturing technologies is essential to meet this requirement. Under these circumstances, it is of great interest for industry how the rapid curing process influences the cure behaviour of composites comparing with the conventional slow production. PES modified TGAP and TGDDM systems are commonly used in the latest-generation thermoplastic toughened prepreg systems for the construction of primary aircraft structures. They provide improvements in impact resistance and less micro-cracking over earlier products. In this work, the heating rate effect on the cure behaviour and morphology of PES modified TGAP and TGDDM polymer blends and the fibre reinforced composite prepreg was investigated.

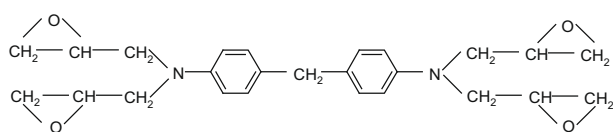
2. Experimental

2.1. Materials

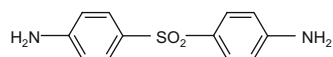
The epoxy resin TGAP (Araldite® MY0510, Huntsman) with epoxy equivalent 95–106, TGDDM (Araldite® MY720, Huntsman) with epoxy equivalent 117–134, and the hardener 4,4'-diaminodiphenyl sulfone (DDS, Sigma Aldrich) (purity > 97%) were used as received. The thermoplastic modifier PES (Ultrason® E1010 natural, BASF) was provided in granular form. The chemical formulae of individual component are illustrated as follows:



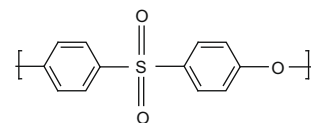
Triglycidylaminophenol (TGAP)



tetraglycidyl-4,4'-diaminodiphenylmethane (TGDDM)



4,4'-diaminodiphenyl sulfone (DDS)



polyether sulphone (PES)

Carbon fibre reinforced composite specimens were manufactured from T300/914 unidirectional prepreg which was obtained from HEXCEL Composites.

2.2. Sample preparation and characterizations

Thermoplastic/epoxy blends were prepared by solution casting from methylene chloride at room temperature. The blends were dried in a vacuum oven at 50 °C for 15 h to remove the trace sol-

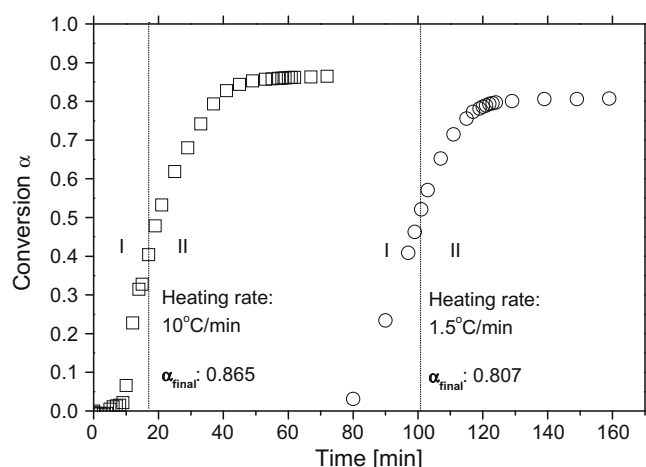


Fig. 1. Cure kinetics following with different cure schedules of the TGAP/PES/DDS (100/30/30) blend: I. heating stage (from 20 °C to 175 °C); II. isothermal curing stage (at 175 °C).

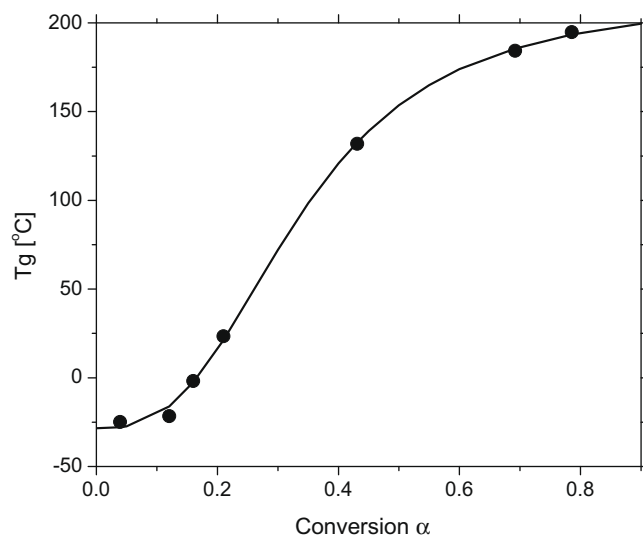


Fig. 2. T_g as a function of conversion for the TGAP/PES/DDS (100/30/30) blend cured at 175 °C for different times. The solid line is the curve simulated using Eq. (1). (The values of the fitting parameters are: $a = -28.45$, $b = 0.046$, $c = 213.77$, $d = 2.84$).

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