Polymer Testing 29 (2010) 291–301

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Test Method

Measurement of (post-)curing strain development with fibre Bragg gratings

Patricia P. Parlevliet [1,*], Harald E.N. Bersee[**], Adriaan Beukers

Delft University of Technology, Faculty of Aerospace Engineering, Kluyverweg 1, 2629 HS Delft, The Netherlands

ARTICLE INFO

Article history:
Received 17 October 2009
Accepted 3 December 2009

Keywords:
Liquid composite moulding
Cure shrinkage
Fibre Bragg gratings
Strain relaxation
Thermosetting polyurethane

ABSTRACT

Curing shrinkage of polymer matrices is a significant source of residual strain formation in thick composite products manufactured with liquid resin infusion. The goal of this paper is to investigate the contributions of cure shrinkage and postcure to residual strain development in a thermosetting polyurethane system suitable for resin infusion of thick composites using fibre Bragg gratings. The results showed that around half of the total shrinkage that contributes to residual strain build-up is due to chemical shrinkage, whereas the other half comprises thermal contraction from the vitrification point. The postcure treatment was found to relax internal strains significantly. The strain-free temperature was found below the postcure temperature and, therefore, the postcure treatment did not induce additional chemical or thermal strains.

1. Introduction

Thick composite structures are gaining wider interest with the application of larger wind turbine blades and the replacement of thick metal aircraft parts with their lighter-weight composite counterpart in order to maximise weight reductions [1]. For thick polymer matrix composite structures, a cost effective manufacturing technology is presented by resin infusion technology, where a pressure difference forces the uncured liquid polymer to flow around the dry fibrous reinforcement in a closed mould and is then allowed to polymerise at elevated temperatures under pressure for a certain time.

During manufacture of thick composite laminates, significant through-the-thickness gradients in temperature can arise, which in turn can result in residual strains and stresses as well as inhomogeneous properties, compromising the performance of the composite structure [2]. Residual strains depend mostly on the shrinkage behaviour of the polymer matrix from the point where polymer shrinkage cannot be relaxed anymore, i.e. has sufficient stiffness for strain transfer [3,4]. In composites manufactured with a reactive polymer matrix, the polymer shrinkage contribution to residual strain formation consists of chemical shrinkage from the gel point onwards and thermal contraction during cooling from the cure temperature to the service temperature [3–5].

Experimental techniques for the monitoring of shrinkage of reactive polymers during cure are, due to the low viscosity, mainly based on volumetric dilatometry [6], such as the gravimetric-based method proposed by Li et al. [5]. A good overview of this type of experimental techniques is described by Schoch et al. [6]. However, these techniques do not provide information regarding the gel point or polymerisation shrinkage contribution to residual strain formation, meaning that more than one experiment is always necessary to establish the cure shrinkage behaviour responsible for residual strain formation [7]. For example, the gel point in thermosets can be established with several experimental techniques, of which Differential Scanning Calorimetry (DSC) and Dynamical Mechanical Thermal Analysis (DMTA) are used most frequently [8].

* Corresponding author. Tel.: +31 15 2788175; fax: +31 15 2781151.
** Corresponding author.
E-mail addresses: patricia.parlevliet@eurocopter.com (P.P. Parlevliet), h.e.n.bersee@tudelft.nl (H.E.N. Bersee).
[1] Currently employee of Eurocopter Deutschland GmbH.

0142-9418/$ – see front matter © 2009 Elsevier Ltd. All rights reserved.
doi:10.1016/j.polymertesting.2009.12.002

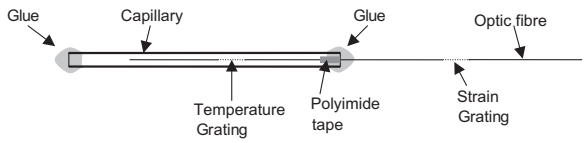

Fig. 1. Schematic view of Bragg grating temperature sensor.

Recent publications describe the use of optic fibres with Bragg gratings (FBGs) in 'single-fibre composites': a model composite with the optic fibre functioning as fibre reinforcement, while simultaneously detecting the strain development caused by the resin curing shrinkage and thermal contraction with the Bragg grating [9–11]. The gel point was found to be the point were strain build-up was first detected by the FBG [12–19]. The vitrification point could be identified where the FBG did not register any more curing shrinkage strains, since the chemical reaction has stopped [18,19]. After this point, the FBGs were found suitable for detection of thermal contraction and expansion coefficients by taking the slope of the strain versus temperature curves [12,15,19]. Another effect detected with FBGs by Giordano et al., was the mismatch in thermal expansion and contraction behaviour between the mould and the resin on strain development [12,13,15,19].

Therefore, optic fibre Bragg gratings seem a powerful experimental tool to establish the polymer shrinkage responsible for residual strain development in resin infused thick composites. The goal of this paper is to investigate the contributions of cure shrinkage and postcure to residual strain development in a polymer system suitable for resin infusion of thick composites with FBGs. The single mode[2] optical fibres that will be used in this study, are made of fused silica (SiO_2) with a Germanium (Ge) doped core for high photosensitivity, which is necessary for inducing the periodic refractive index pattern and, thus, creating the Bragg grating. We used 'draw tower' gratings (DTGs®) which are inscribed during the manufacturing process of the optic fibre by means of a lithographic process and are coated immediately after the inscription of the Bragg grating [20,21]. This is different from most other FBGs described in literature, where the fibre is made photosensitive by means of a hydrogen environment, usually carried out by stripping the cladding, application of the grating and renewal of cladding [22,23]. This latter type of grating is less stable and requires annealing above the maximum service temperature for several hours [22–25], whereas the DTGs® can be used immediately.

The room temperature curing thermosetting polyurethane (turane) is highly suitable as a matrix for thick composite manufacturing by means of the vacuum infusion process due to its low viscosity, and composites made thereof can be demoulded within 90 min [26]. The Daron® hybrid turane system (DSM Composite Resins, the Netherlands) is based on unsaturated polyester for its heat resistance and stiffness and polyurethane for its flexibility and toughness [26]. It is very suitable as a matrix for high performance automotive composite applications, such as the Nuna solar powered car, since it has high thermal stability (150–220 °C) and good mechanical properties. In addition, it is currently being certified in accordance with the CS23 European aviation regulations [27].

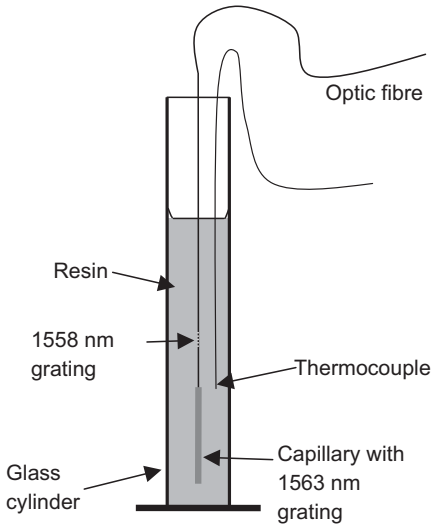

Fig. 2. Schematic view of matrix shrinkage test set-up.

A postcure cycle is recommended for this polymer system and, therefore, the cured specimens with FBG sensors were exposed to another temperature cycle for postcuring. A second thermal cycle was carried out to identify differences in polymer properties after the postcuring cycle. For comparison with the FBG results, differential scanning calorimetry (DSC) tests were done to confirm thermal transitions in the cured and postcured polymer. The thermal expansion and contraction of the polymer were determined with linear dilatometry using a thermal mechanical analyser (TMA), which can be compared with the FBG results to establish whether the strain transfer between the matrix and optic fibre is sufficient.

2. Experimental procedures

2.1. Materials

For the turane polymer two separate components were prepared.

- Component A, consisting of the Daron® ZW6154 hybrid system (DSM Composite Resins, Netherlands) mixed with peroxide (Lucidol® CH50X, Akzo Nobel, Netherlands) in a mass ratio of 100:2.
- Component B, consisting of Lupranate® M20R (DSM Composite Resins, Netherlands) mixed with Accelerator (NL64-10P, Akzo Nobel, Netherlands) in a mass ratio of 35:2 (relative to Daron® ZW6154) to form component B.

After mixing the two components, the liquid was degassed for 5 min under vacuum.

[2] Single mode fibres are most suitable for transmission of wavelengths over a long distance due to a narrow core through which the rays of light cannot escape [11].