Composites: Part A 46 (2013) 117–121

Contents lists available at SciVerse ScienceDirect

Composites: Part A

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

Short communication

Embedded single carbon fibre to sense the thermomechanical behavior of an epoxy during the cure process

Kuang-Ting Hsiao *

Department of Mechanical Engineering, University of South Alabama, Mobile, AL 36688, USA

ARTICLE INFO

Article history:
Received 19 March 2012
Received in revised form 11 November 2012
Accepted 14 November 2012
Available online 28 November 2012

Keywords:
A. Smart materials
B. Thermomechanical
D. Process monitoring
E. Cure

ABSTRACT

A new approach that uses a single carbon fibre for sensing the thermomechanical behavior of an epoxy during the cure cycle is presented. By recording and analyzing the electrical resistance and temperature history of a carbon fibre embedded inside an epoxy specimen during the cure cycle, the interaction between the carbon fibre and the surrounding polymer can be revealed. Compared with reported TMA and DMA results, this embedded carbon fibre sensor approach successfully detects the glass transition zone covering the *final transition temperature*, the *main transition temperature*, and the *starting transition temperature* that respectively have similar values as: (i) T_g *corresponding to the abrupt change in CTE*, (ii) T_g *by storage modulus* (E') *onset*, and (iii) the *upper temperature limit for the linear relationship between* E' *and the temperature*. The future applications for this sensor method are also discussed.

1. Introduction

In a polymer matrix composite (PMC), the resin plays an important role in transferring and distributing the mechanical load among the reinforcement fibres. Typically, the elastic modulus of a resin decreases as the operating temperature increases and drops dramatically after the temperature passes a critical point such as the crystalline melting point (T_m) for a thermoplastic resin or the glass transition temperature (T_g) for a thermoset resin. Therefore, it is necessary to know the maximum operating temperature of a resin. To evaluate the thermomechanical behaviors of polymers, Dynamic Mechanical Analysis (DMA) [1,2] is a popular method. In a DMA test, a small resin specimen is placed inside a thermal chamber and is subjected to a cyclic dynamic mechanical loading (e.g., three-point bending test, cantilever beam test, or tensile test). From the dynamic stress–strain responses at various temperatures, the dynamic modulus-temperature relationship of the polymer can be obtained. The dynamic moduli (storage modulus E' and loss modulus E'') of a thermoset resin change significantly around T_g. Two typical analysis methods for determining the T_g value in a DMA experiment are based on: (i) the temperature corresponding to the maximum of tan δ (i.e., E''/E') and (ii) the temperature corresponding to the storage modulus (E') onset. As the glass transition of a thermoset resin typically covers a wide temperature range, these two methods usually produce slightly different T_g values. Besides DMA, Differential Scanning Calorimetry (DSC) and Thermomechanical Analysis (TMA) are also commonly used to measure the critical temperatures (e.g., T_g or T_m) of a polymer by detecting the abrupt changes in the internal energy and the coefficient of thermal expansion (CTE), respectively.

Carbon fibre (CF) is a major reinforcement material for PMCs due to its high strength-to-weight ratio and modulus-to-weight ratio. In addition, CF and its composites could also be used as strain gages since the electrical resistance of CF increases when it is elongated. Wang and Chung [3] reported that the electrical resistance change of a CF before and after resin curing can be correlated to the residual stress of the CF inside a cured polymer coupon at room temperature. They measured the electrical resistance of the CF, which had its two ends protruded from the small polymer coupon, before and after resin curing. By putting the coupon under a tensile test, they were able to restore the electrical resistance of the CF by applying the tensile strain that corresponded to the theoretically estimated residual stress on the CF. Park et al. [4] performed a micromechanical study on single CF polymer coupons, with the same configuration as in [3], and reported that the difference in electrical resistivity of the CF before and after resin curing (with the same initial temperature and final temperature) is affected by the additives in the resin and can be related to the final fibre/resin interfacial performance. In [5], the cure-induced equivalent residual stress of the same type of single CF polymer coupon was simulated by the finite element method (FEM) and compared with the measured difference in electrical resistivity of the CF before and after resin curing. It was reported that the FEM simulated residual stress and the difference in electrical resistivity of the CF before and after resin curing both increase with a higher curing

---

* Tel.: +1 251 4607889.
E-mail address: kthsiao@SouthAlabama.edu

1359-835X/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.compositesa.2012.11.007

temperature. The interfacial shear strength (IFSS) experimental results further showed that the IFSS tends to increase when a higher curing temperature is used. Similar single CF polymer coupon configuration has also been modified by replacing the CF with a shape memory alloy (SMA) fibre in [6]. It was found that the cure induced residual stress causes the incomplete recovery of electrical resistivity of the SMA fibre before and after resin curing. In addition, the fibre–matrix interfacial sliding during a cyclic tensile test can be detected by the super-elastic behavior of the SMA fibre. In summary (as reported in [3–6]), this kind of single fibre polymer coupon method provides a sensory tool for studying the micromechanical interaction between the conductive fibre (such as the CF and the SMA fibre) and the polymer matrix. However, no approach has been reported to use such a sensor for quantifying thermomechanical properties (such as T_g or T_m) of a polymer.

This paper presents a new use of an embedded single CF to sense the thermomechanical behavior (particularly the T_g) from the surrounding polymer during the cure cycle. The electrical resistance history of the embedded CF is measured and analyzed along with the temperature history. The temperature effect is removed from the overall electrical resistance to approximate the strain-related electrical resistance. The experiments show interesting and repeatable relationship between the temperature and the strain-related electrical resistance. The significance of the results will be discussed and compared with reported DMA and TMA data of the same resin system.

2. Experiments and materials

A cyclic thermal test is performed to obtain the relationship between the electrical resistance (R) and temperature (T) of a stress-free CF. The two ends of the CF (T-300 by Toray) are bonded by conductive paint (CW2200MTP by Chemtronics Corp) to two copper wires that are taped to a rigid paper sheet hanging inside the oven. The distance between the two ends of the CF is adjusted so that the CF is not tensioned (i.e., stress free). The CF loosely hanged inside an oven is slowly heated and then cooled in a cyclic manor. The electrical resistance of the CF is measured by a multimeter (Agilent 34405 5-1/2 digit multimeter). The temperature is measured by a thermocouple, which is attached to the rigid paper sheet and placed near the CF, and a thermocouple-to-analog converter (Omega KK-K-24 and TAC-386-KC, ±2 °C accuracy).

To detect the thermomechanical behavior of a resin during the cure cycle, a CF (~45 mm long) is loosely held inside an open stainless steel mold (cavity dimensions: 305 mm (length) × 57 mm (width) × 25 mm (depth)) and a thermocouple is placed near the CF (see Fig. 1). A piece of Kapton® tape (by Dupont) is adhered to the mold under the CF to avoid short circuit. The entire mold assembly is placed inside an oven. The epoxy, which consists of EPON862 resin and EPIKURE Curing Agent W (purchased from Miller-Stephenson Chemical Co.) mixed at the weight ratio of 100:26 at 70 °C, is poured into the mold. The oven temperature is set to 120 °C for 4 h and then 177 °C (±5 °C) for another 4 h. Finally, the oven is turned off for cooling. The electrical resistance of the CF and the temperature measured by the thermocouple are recorded through the cure process. Two cure experiments are conducted separately to assess the repeatability.

3. Results and discussion

Fig. 2 shows the relation between the electrical resistance (R) and the temperature (T) of a CF during the cyclic thermal test. Note that the dimensionless resistance ($R^* = R(T)/R(T_0)$) of the CF under this cyclic thermal test is indeed the stress-free dimensionless resistance (R^*_{th}) of the CF. As plotted in Fig. 2a, the R^*_{th} overlaps with T when a negative scaling factor is used, which indicates a linear R^*_{th}—T relationship. Fig. 2b further shows that R^*_{th} linearly decreases as T increases. The R^*_{th}—T relation can be linearly modeled as $R^*_{th} - 1 = \beta(T - T_0)$; where the reference temperature T_0, the reference resistance $R(T_0)$, and the thermal coefficient β are 127 °C, 12140 Ω, and −0.00036906/°C, respectively.

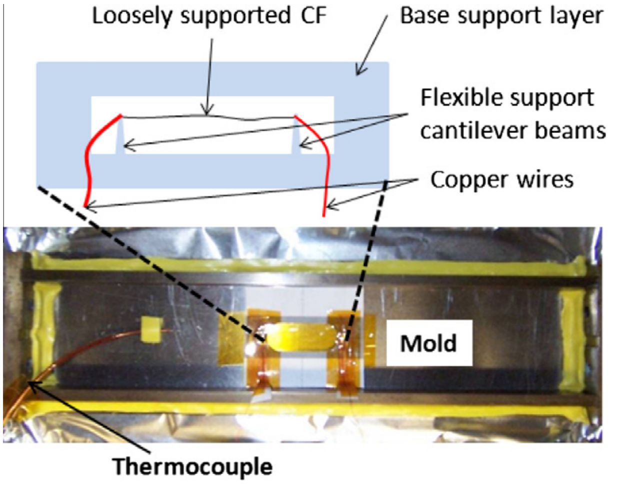

Fig. 1. The mold assembly with a single CF and a thermocouple for the cure experiment. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The histories of R^* and T of the two independently conducted cure experiments are plotted in Fig. 3a. A disturbance period is found in the beginning as the CF is disturbed by the non-isothermal resin flow that gradually settles in the open mold cavity. Fig. 3b further shows the histories of the stress-loaded dimensionless resistance (R^*_s, which is defined as: $R^*_s = (R^* - R^*_{th}) - R^*_{offset}$) and the temperature during the two cure experiments. Note that the offset dimensionless resistance (R^*_{offset}) is selected to make $R^*_s = 0$ in the beginning of the cooling stage. In Fig. 3b, all the experimental results show an interesting and repeatable correlation starting from around 23,000 s (which is within the 177 °C curing stage) until the completion of the cooling stage. Before 23,000 s, the resin is not fully solidified and its crosslinking (which causes the resin shrinkage) and relaxation both have notable influence on the CF (reflected on the change of R^*_s) during the 177 °C curing stage. Before solidification, the results of the two cure experiments are similar in trend but with slightly different details, which could be due to the interaction between the loosely supported CF and the viscoelastic resin as well as the inevitable small deviation between separate experiments. Nevertheless, Fig. 3b clearly indicates that R^*_s responds to the thermomechanical behavior of the cured epoxy, especially during the cooling stage. The R^*_s—T correlation during the cooling stage may depict the combined thermomechanical effect from the resin modulus change, resin thermal contraction, CF expansion, and the interaction between the CF and the resin.

To further investigate the thermomechanical interaction between the resin and the CF during the cooling stage, the close-up R^*_s—T curves for the two cure experiments are analyzed. Fig. 4a shows the overlapped results of the two experiments and validates the repeatability. Fig. 4b and Fig. 4c individually plot the R^*_s—T curves of the two experiments. Both experiments follow the same pattern with two significant knee-like points, i.e., T_{s1} and T_{s3}, along with an intermediate slope changing point T_{s2} in between. This R^*_s—T curve pattern is caused by (i) the resin contraction and (ii) the stress applied on the CF through the resin-CF interface. According to the TMA data reported on the EPON862/EPIKURE Agent W epoxy system [7], the CTE in the rubbery state is