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Material properties

Interfacial and wetting properties of carbon fiber reinforced epoxy composites with different hardeners by electrical resistance measurement

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ABSTRACT

In this research, interfacial and wetting properties of N,N,N,N-tetraglycidyl-4,4-diaminodiphenylmethane (TGDDM) epoxy resin with two hardeners with different chemical structure were evaluated by electrical resistance (ER) measurement. The heat of reaction of TGDDM epoxy with the two different hardeners, 33 and 44 di-amino di-phenyl sulphone (DDS), was analyzed by differential scanning calorimetry (DSC). The TGDDM epoxy exhibited different mechanical properties with the two different DDS hardeners. Combined ER, wetting measurements and the microdroplet test were used for evaluating the spreading effect and interfacial shear strength (IFSS) of carbon fiber (CF) reinforced TGDDM epoxy composites with these different hardeners. The heat of reaction and mechanical properties of TGDDM/DDS were influenced by the chemical structure and different free volumes of the epoxy resins. The relationships between the ER-wetting results and the IFSS were internally consistent. Ultimately it was demonstrated that ER measurements makes it possible to estimate the interfacial and wetting properties of CF reinforced epoxy composites.

1. Introduction

It is possible to have some control over the structure of epoxy composite matrices to optimize mechanical and thermal properties for different applications [1,2]. The different functional groups of epoxy resin are important to improve the properties of composites matrices [3,4]. Bisphenol-A type epoxy normally has two kinds of functional groups. Its exact structure is mainly controlled by different hardening conditions resulting in two (or perhaps three) kinds of functional groups in the epoxy resin during polymerization [5–7]. High polymerization is accomplished by increasing the functional groups of the epoxy for rigid composites. Improvement in matrix properties has been accomplished by controlling the functional groups thereby improving the total performance of the epoxy composites [8–10].

Generally, highly polymerized polymers exhibit a high T_g, and with four kinds of functional groups of TGDDM epoxy resin it is possible to produce composites for high reasonable high temperature applications [11,12]. High temperature composites have a variety of potential applications in heavy industry, aerospace, other transportations vehicles etc. The thermal properties of polymers may be enhanced by the arrangement of the polymer chains, by inserting reinforcing particles in the polymer matrix, and polymer packing effects have been shown to be important in controlling thermal properties in epoxies [13,14]. High packing effects of epoxy resins results in higher polymerization and more rigid epoxy formulations. Optimizing the formulation between epoxy and hardener can improve the thermal and mechanical properties of composites [15].

In this study, micromechanical testing combined with ER-wetting methods were used to evaluate interfacial and wetting properties of CF/TGDDM/DDS composites in which different hardeners were used to obtain different chemical structures. DSC was

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performed to compare the heat of reaction of TGDDM/DDS with the different DDS hardeners. ER wetting measurements were used to evaluate spreading effects of epoxy resin on CF tows during curing in the autoclave. ER change ratio of the CF tows was altered by different epoxy wetting and curing conditions with the different hardeners. In addition, the IFSS between matrix and fiber for the different DDS type hardeners was evaluated using the microdroplet test. Next results of mechanical tensile tests, IFSS measurements, wetting, etc. were compared with the ER testing to establish correlations between them and look for causes and effects for the observed behavior.

2. Experimental

2.1. Materials

TGDDM (N,N,N,N-tetraglycidyl-4,4-diamino diphenylmethane, Huntsman Co. Ltd., U.S.A.) with four kinds of functional groups was used as composite matrices. 33 DDS (33 diamino diphenyl sulphone, Huntsman Co. Ltd., U.S.A.) and 44 DDS (44 diamino diphenyl sulphone, Hunstman Co. Ltd., U.S.A.) were used as hardeners. Carbon fiber (T700SC, Toray Co. Ltd., Japan) was used as reinforcement and to evaluate interfacial and wetting properties to the composite resins. Fig. 1 shows the chemical structures of the epoxy resin and these two hardeners.

2.2. Methodologies

2.2.1. Heat of reaction of TGDDM with different hardeners and curing cycle temperature

The curing kinetics of epoxy resin with different hardeners and curing cycle temperatures was investigated via DSC (Q1000, TA Instruments) experiments [16]. The heats of reaction of 5 (±1) mg samples of TGDDM/DDS, with the two different hardeners, were analyzed by DSC at 100 °C/minute. The optimum curing cycles for with two different chemical structures was observed for as: (1) 120 °C/1 h → 180 °C/1 h, (2) 140 °C/1 h → 180 °C/1 h, (3) 160 °C/1 h → 180 °C/1 h, (4) 180 °C/1 h → 200 °C/1 h, and (5) 200 °C/1 h → 220 °C/1 h.

2.2.2. Wetting evaluation of epoxy resin in CF tow using electrical resistance method during cure cycling in autoclave

The CF tows were wet by TGDDM epoxy resins with the two different hardeners. An amount of 0.5 g of CNT epoxy resin was dropped onto the tow for each specimen, as shown in Fig. 2 [17].

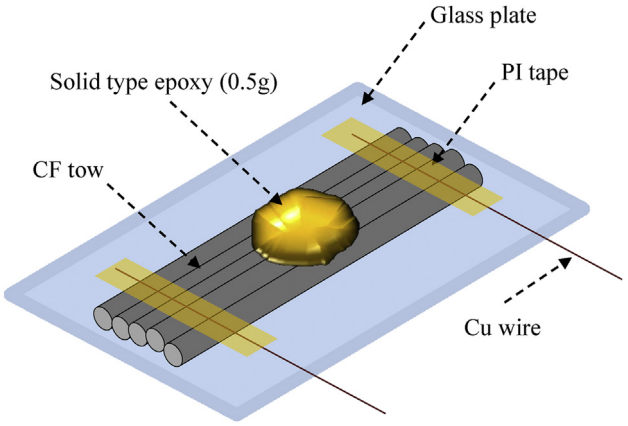

Fig. 2. Schematic sketch of ER wetting test specimens.

The resin-spreading process was observed, using ER the method, during the curing process in an autoclave. The cured epoxy resin in the CF tow was also compared for the two different hardeners under a transmission microscope. The length of the CF tows was 4 cm and a gauge length of 2 cm was used for measuring ER. The ER of each specimen was analyzed using a multi-meter (34401A, Agilent Tech., U.S.A.) with 4-probe method. ER-wetting measurement was tested in an autoclave during curing process. The first step was to get a base ER change ratio of bare CF tow specimens in Fig. 2. The second step was to test ER change ratio of TGDDM/DDS on CF tow with spreading during curing process using ER-wetting method.

2.2.3. Microdroplet test of CF/TGDDM with different hardeners

The IFSS between carbon fiber and epoxy resin was also measured using a microdroplet pull-out test. Carbon fibers were fixed in a steel frame at regular separated distances. Microdroplets of TGDDM with the different hardeners were formed on each fiber using a tip-pin. Microdroplets of DDS/TGDDM were put into an autoclave at 200 °C for 1 h for pre-curing and then 220 °C for 2 h for post curing.

One of the major advantages of microdroplet technique is that the value of forces at the moment of debonding can be measured. The microdroplet specimen was fixed by a microvice using a specially-designed micrometer. The IFSS was calculated from the measured pull-out force, *F*, using the following equation:

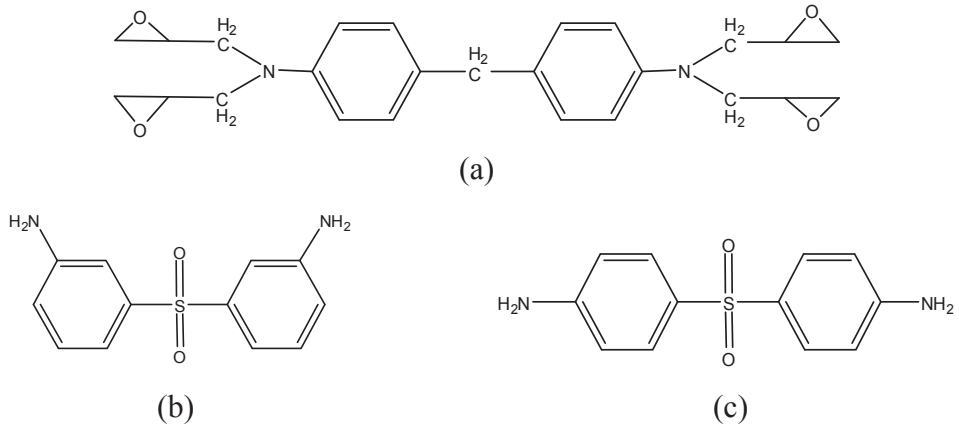

Fig. 1. Epoxy and hardener materials condition: (a) TGDDM: N,N,N,N-tetraglycidyl-4,4-diaminodiphenylmethane; (b) 33 DDS: 33 di-amino di-phenyl sulphone; and (c) 44 DDS: 44 di-amino di-phenyl sulphone.