

In-situ toughened CFRP composites by shear-calender orientation and fiber-bundle filtration of PA microparticles at prepreg interlayer



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ABSTRACT

Epoxy matrix toughened by polyethersulfone (PES) and polyamide (PA) microparticles was designed and the in-situ interlaminar toughened carbon fiber/epoxy composites were fabricated. Synergistic toughening effect of PES and PA on epoxy matrix was achieved due to semi-IPN structure of PES toughened matrix and uniform dispersion of PA microparticles. Shear-calender orientation of PA microparticles was found during prepreg processing and the microparticles remained on the surface of prepreg due to fiber-bundle filtration. The in-situ formed toughening interlayer of PA microparticles and interfacial bonding between PA and epoxy matrix were detected, which resulted in enhanced fracture toughness, CAI, and transverse flexural strength of the composite based on the PES/PA synergistically toughened matrix. SEM images of fracture morphology of the composite showed evidence of enhanced plastic deformation created by PES and PA, and crack deflection and bridging by PA microparticles.

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1. Introduction

Carbon fiber reinforced preregs, as the important intermediate materials of CFRP composites, have been extensively used in aerospace industry and particularly in manufacturing load-bearing structural parts of aircraft [1,2]. The hot-melt prepreg was usually fabricated by applying a uniform resin film to a release paper and further impregnating the fiber bed with upper and lower resin films via hot pressing, in which the relative amount of fibers and resin in a composite part and the distribution of resin within the fiber network could be accurately controlled [3–8]. However, the mechanical performance of prepreg-based composite laminates in the through-thickness or Z-axis direction was normally much lower than that in the in-plane direction, which could result in interlaminar fracture or delamination in composite laminates [9].

Various methods have been attempted to improve delamination resistance of prepreg-based composites by either directly toughening matrix or interleaving composites with a discrete interlayer [10]. Directly toughening the matrix resin usually could not be translated to an improvement in interlaminar toughness of composite due to the constraining action from the neighboring plies [11]. Interleaving, a more effective method, has been employed as crack arrester to interrupt crack propagation in fiber reinforced

composites [12–16]. Recently, interleaves made from electrospun thermoplastic nanofibers and toughened epoxy films, such as PSF nanofibers, MWNTs-EP/PSF hybrid nanofibers, epoxy/PA films, and CNF-epoxy bucky papers have been extensively studied [17–20].

It has also been reported that the Nylon-12 particles sifted evenly onto each ply of carbon fiber fabric greatly increased the fracture toughness of CFRP composites prepared using RFM process [21]. Although these interleaving methods were effective on interlaminar toughening of CFRP composites, considerable addition to complexity of manufacturing process, such as complicated interlayer preparation and interleaving process before laminate fabrication, was a potential issue in the above techniques and limited their industry scale production. Such an issue could be eliminated by incorporating in-situ formed interlayer in the CFRP composites during prepreg processing. Therefore, it was of great practical importance to disperse toughening particles into resin matrix and then control their distribution during prepreg processing to achieve in-situ interlaminar toughening of CFRP composites.

In this contribution, PES and PA microparticles synergistically toughened epoxy matrix was prepared to fabricate unidirectional carbon fiber reinforced preregs using a conventional hot-melt method. The distribution state of PA microparticles during prepreg processing was investigated. Special focus was placed to specifically study the mechanical properties and interlaminar morphologies of the composites affected by PES and PA microparticles. The

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chemical interactions between PA and epoxy matrix, and the fracture morphology of the composites were also studied to elucidate the in-situ interlaminar toughening mechanisms. Finally a schematic model of the interlaminar toughening mechanism was proposed.

2. Experimental

2.1. Materials

Diglycidyl ether of bisphenol-F (DGEBF, EPON 862) and triglycidyl-p-aminophenol (TGAP, MY0600) were produced by Shell Chemical and Huntsman, respectively. Polyethersulfone (PES-OH, Mn = 36,000) was supplied by Jilin University, China. The PA microparticles (PA6, Orgasol 1002D NATI, Melting Temperature = 217 °C) with an average particle size of 20 μm were purchased from Arkema, France. Typical scanning electron microscope (SEM) images of the PA microparticles are presented in Fig. 1. The hardener 3, 3'-diaminodiphenyl sulphone (3, 3'-DDS) was produced by Suzhou Yinsheng Chemical Co., Ltd, China. The carbon fibers (T800HB-12K-50B, density: 1.8 g/cm³) were purchased from Toray Industries, Japan. Polyethylene glycol (PEG600) was purchased from Beijing Chemical Agent Co.

2.2. Preparation of epoxy matrix

Four kinds of epoxy matrix were prepared consisting of EP, EP/PES, EP/PA, and EP/PES/PA. DGEBF and TGAP (weight ratio of 1:1) were mixed to form the basic epoxy formulation (EP) at 120 °C. To fabricate EP/PES blend, PES (20 wt% of EP) was added to EP and stirred (at 150 °C) until PES was dissolved. PA microparticles (15 wt% of EP) were added to the EP and EP/PES blend by mechanical stirring (at 120 °C) to form EP/PA and EP/PES/PA blends, respectively. Hardener 3, 3'-DDS (30 wt% of EP) was then added to the EP, EP/PES blend, EP/PA blend, and EP/PES/PA blend to prepare EP matrix, EP/PES matrix, EP/PA matrix, and EP/PES/PA matrix, respectively. The average density of the resin matrix was about 1.4 g/cm³.

2.3. Preparation of CFRP prepreg

CFRP prepregs based on EP, EP/PES, EP/PA and EP/PES/PA matrix were defined as FE, FEP, FEPA and FEPP prepregs, respectively. First, the epoxy resin was applied to a release paper by shear rollers at 95 °C to prepare a uniform resin film, and the amount of the resin per unit area was controlled at (42.5 ± 2) g/m². Then, the resin

films were superimposed on each side of carbon fiber bundles, which were aligned in one direction in sheet form and heated up to 120 °C and pressurized so that the carbon fibers were impregnated with resin matrix to form a unidirectional prepreg. The amount of the carbon fiber per unit area was (165 ± 2) g/m².

2.4. Sample preparation

The single-edge-notch bending (SENB) specimens of various epoxy matrix were cured at a schedule (130 °C for 2 h, 180 °C for 2 h and 200 °C for 1 h) for fracture toughness test. To test CAI (compression after impact) property, 32 plies of 350 mm × 350 mm prepregs of [45/0/−45/90]_{4S} stacking sequence were stacked (according to GB/T 21239-2007) on the laboratory-made mold, and cured with flat-plate vulcanizer at three stages: 130 °C for 2 h, 180 °C for 2 h and 200 °C for 1 h. Following the same curing stages, 24 plies of 180 mm × 150 mm prepregs and 16 plies of 180 mm × 80 mm prepregs were laminated for G_{IC} (mode-I interlaminar fracture toughness) and transverse flexural tests, respectively. Teflon film with 12 μm thickness was placed at the mid-plane of laminate to provide an initial edge crack length for G_{IC} test.

To prepare a specimen for phase contrast microscopy (PM), EP/PES/PA matrix was applied to a glass slide and cured following the same schedule as aforementioned. For polarizing microscopy observation, EP/PA blend without hardener and the PEG/PA blend (fixed PA content: 10 wt%) was applied to a glass slide and heated up to 220 °C (above the Melting Temperature of PA microparticles) and then cooled down at 5 °C/min to room temperature in a vacuum oven.

2.5. Characterizations

2.5.1. Morphology observations and thermal analysis

Phase contrast microscope (PM, Leica DM2700P, Germany) and transmission optical microscope (TOM, Olympus BX51TRF, USA) were used for characterizing the dispersion state of PA microparticles in epoxy matrix and resin film, respectively. Stereo microscope (SM, Leica M165 FC, Germany) was used to examine the distribution of PA during prepreg processing. Surface morphology of prepregs and fracture surfaces of the composites were observed by FE-SEM-Supra55 (Zeiss, Germany). Polarizing microscopy (POM, Leica DM2700P, Germany) was used to study the recrystallization of EP/PA and PEG/PA blends. DSC (Q200, TA Instruments, USA) was used to study the sequential heating and cooling scans (5 °C/min) of the epoxy/PA blend.

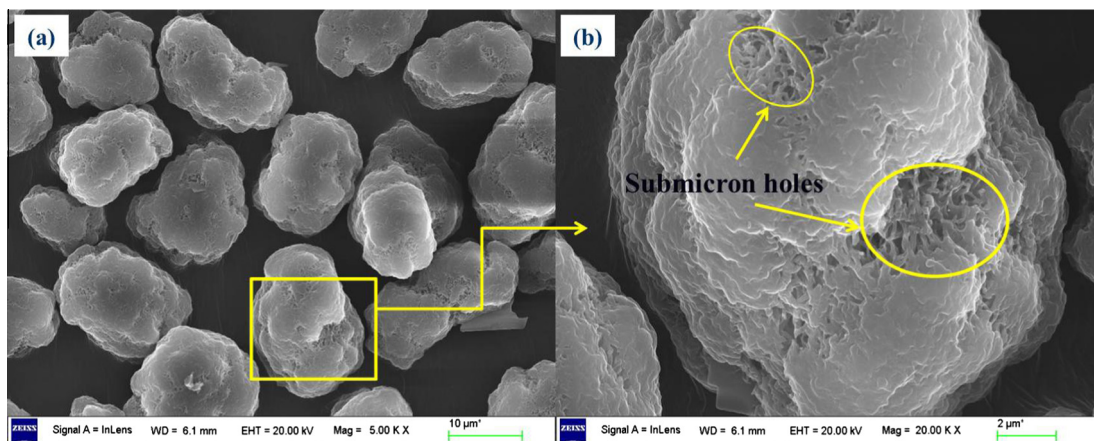


Fig. 1. SEM images of PA microparticles: (a) Low magnification image; (b) High magnification image showing a rough surface covered with submicron holes. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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