Composites: Part A 52 (2013) 31–37

Contents lists available at SciVerse ScienceDirect

Composites: Part A

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

# Low viscosity cyanate ester resin for the injection repair of hole-edge delaminations in bismaleimide/carbon fiber composites

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ARTICLE INFO

*Article history:*
Received 23 July 2012
Received in revised form 9 April 2013
Accepted 2 May 2013
Available online 15 May 2013

*Keywords:*
A. Polymer–matrix composites
A. Thermoset resin
D. Mechanical properties
B. Delamination
Composite repair

ABSTRACT

The repair efficiency of bisphenol E cyanate ester (BECy) resin was investigated for the injection repair of high temperature polymer–matrix composites by ultrasonic C-scan mapping, fluorescent dye penetration, optical microscopy, hole plate shear (HPS), and post delamination compression tests. Bismaleimide/carbon fiber (BMI–cf) composites were chosen as a model substrate. A vacuum-based resin injection repair method was used for repairing the pre-damaged composite specimens. The effect of surface wettability on the repair efficiency of BECy on BMI–cf composite substrate was studied by temperature dependent contact angle measurements. C-scan, fluorescent dye penetration, and optical microscopy images of pristine, delaminated, and repaired specimens reveal efficient infiltration of resin in specimens repaired at elevated temperatures. The repair efficiency calculated from HPS and post delamination compression tests was observed to be 155% and 100%, respectively, illustrating the capability of BECy for repairing high temperature structural composites.



## 1. Introduction

Polymer matrix composites are gradually replacing the metal alloys in advanced structural applications owing to their high specific stiffness and strength. Polymer matrix composites are typically multi-layer materials consisting of continuous fiber reinforcements embedded in a rigid polymer matrix, resulting in excellent in-plane properties. A major disadvantage of this laminate structure is its high susceptibility to defects and damage in the form of interlaminar fracture, or delamination. This damage can greatly compromise the structural integrity of the material [1–5]. Delamination damage is frequently encountered in composites as a result of low energy impact and cyclic thermo-mechanical loading. Patch and scarf repairs are common practices for repairing damaged parts. Although these repair techniques avoid dismantling the damaged structure by applying a reinforcing patch, either bonded or bolted to the composite structure, their applicability is hindered due to limited access to the damage area and the potential for additional damage accrued during the process of removal of the original material. With the growing demand for polymer matrix composites, new repair methods must be developed to overcome the limitations in conventional repair methods. Alternatively, the delamination may be repaired by injecting resin via an access hole into the failed area. This eliminates the need to remove the outer undamaged plies and may result in higher repair strength, provided the adhesive strength of the injected resin is adequate.

In the aerospace industry, high temperature composites are increasingly used for engine cowlings, thrust reversers, and for structural skins subjected to supersonic flows [6]. Next generation airframes, however, are incorporating an increasing amount of bismaelimide based composite structure due to its enhanced, high service temperature capability and increased strength. Determining suitable repair resins for these composite material systems is an active area of development. In order to repair such advanced composites by an injection repair method, the injecting resin must meet the thermo-mechanical properties of the composite. Russell and Bowers [7] identified the following requirements for successful repair: (1) the repair resin should have a cure temperature comparable to the service temperature of the composite material, (2) the resin should cure without releasing volatiles, and (3) the fracture toughness and crack growth resistance within the re-bonded interfaces should be similar to that of the pristine material. The glass transition temperature ($T_g$) of the cured resin used to repair high temperature panels is critical for a successful repair. The mechanical strength of the repaired structure will decrease significantly when the service temperature of the composite exceeds the $T_g$ of the adhesive. Therefore, the $T_g$ of the cured resin should be significantly above the operational temperature to avoid potential degradation in strength near the service temperature. The flow capability, which depends on the viscosity and surface energy of the resin and substrate, is also a major factor that determines the extent of resin infiltration into the microcracks caused by delamination.

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1359-835X/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.compositesa.2013.05.001

Recently, we have reported that bisphenol E cyanate ester (BECy) is suitable for injection repair of delaminated bismaleimide–carbon fiber (BMI–cf) composites owing to its intrinsic properties, such as low viscosity at room temperature, high thermal stability after cure, high delamination strength, and high fracture toughness on BMI–cf substrates at temperatures as high as 200 °C [8]. The present work focuses on the resin infiltration into composite delaminations and on the effectiveness in restoring the strength of damaged BMI–cf composite parts. The delamination damage was created by a hole plate shear (HPS) technique where a static out-of-plane compressive load is applied to a central hole drilled in the center of the composite panels. Specifically, the success of the resin infiltration was investigated comprehensively by observing in-plane and cross-section views of repaired composite specimens using C-scan imaging, optical microscopy, and fluorescent dye penetration techniques. The mechanical efficiency of the repaired composite specimens was evaluated by studying the in-plane compressive strength and interlaminar shear strength using HPS and post delamination compression tests respectively.

## 2. Experimental

### 2.1. Materials

The adhesive resin, bisphenol E cyanate ester (BECy) monomer, is a commercially available resin from Bryte Technologies (Morgan Hill, CA) with product number EX-1510, and was used as received without further purification. The liquid phase organometallic-based polymerization catalyst (EX-1510-B, Bryte Technologies) was supplied with the resin and was used at the manufacturer's suggested loading of three parts per hundred parts resin (phr).

Bismaleimide–carbon fiber (BMI–cf) prepreg was supplied as HTM 512-2 prepreg by Advanced Composites Group, Inc. (Tulsa, OK). The version of the HTM 512-2 prepreg constitutes of balanced $2 \times 2$ twill weave with 12 K high strength carbon of 660 g/m$^2$.

### 2.2. Composite panel manufacturing

The composite panels were manufactured according to specific test requirements. Nine layers of the prepreg were hand-laid at ±45° orientation to achieve a thickness of 0.25 in in the final composite plate. The prepreg plies were cured in an auto-series hot press from Carver. Inc. IA, USA. The panels were processed at 190 °C under 0.6 MPa (90 psi) pressure for 6 h, followed by a free-standing post-cure for 8 h at 240 °C based on a schedule suggested by the supplier.

Square plates $10.16 \times 10.16$ cm ($4 \times 4$ in.) were machined from the $25.4 \times 25.4$ cm ($10 \times 10$ in.) plate and a 0.63 cm (0.25 in.) diameter hole with a 82° countersink about one third of the way through was drilled at the center of each plate to facilitate damage initiation and resin injection repair steps. This configuration was chosen based on an early work by Russell and Bowers to represent a typical aerospace fastener hole [7].

### 2.3. Contact angle measurements

The contact angle measurements were carried out with a ramé-hart 100-00 115 NRL contact angle goniometer, ramé-hart instrument co., NJ. It is equipped with a camera and a video monitor. In order to investigate the wettability of BECy on a BMI–cf composite damaged surface, a $1.27 \times 1.27$ cm ($0.5 \times 0.5$ in.) delaminated ply taken from a damaged BMI–cf composite panel was used as substrate. The prepared substrate is expected to simulate the delaminated fracture surfaces. The substrate was dried at 150 °C in a convection oven. A temperature controlled sample chamber was used for measuring the temperature dependent contact angle. The temperature dependent contact angle of the BECy resin on five different damaged BMI–cf composite substrate was measured between 30 and 150 °C with an increment of 10 °C step. Due to huge scattering between the contact angles curves measured on different surfaces, only two representative curves were considered for discussion. The contact angles from both left and right sides of the resin drop were measured and averaged. The drop was allowed to equilibrate for 10 min before each measurement.

### 2.4. Ultrasonic scanning (C-scan)

An air-coupled, through-transmission ultrasonic system with 120 kHz focused probes was used to create C-scans. Prior to scanning, the hole in each plate was filled with putty and the plates were surrounded by a foam frame. Both these techniques were used to minimize the noise around the hole and edges by preventing excess sound leaks. The scans were obtained for the pristine, delaminated, and repaired samples to study the infiltration efficiency of the resin.

### 2.5. Imaging and quantifying the damage

Pristine, delaminated, and repaired HPS specimens were sectioned diagonally through the center of the hole to image the cross-section by optical microscopy and fluorescent dye penetration measurements, as shown in Fig. 1. The cross section surface was finely polished before microscopic images were collected in reflection mode using Olympus BX51. The fluorescent dye penetration tests were performed by using Zyglo ZL-56 fluorescence dye penetrant [9]. In both optical microscopy and fluorescent dye penetration measurements, the surface images were merged to produce the microscopic image of a 2.56 cm (1-in.) cross-section from the damaged zone. The optical microscopic images were further used to quantify the crack filling capability of BECy resins. The delaminations, microcracks, and air voids in the samples were

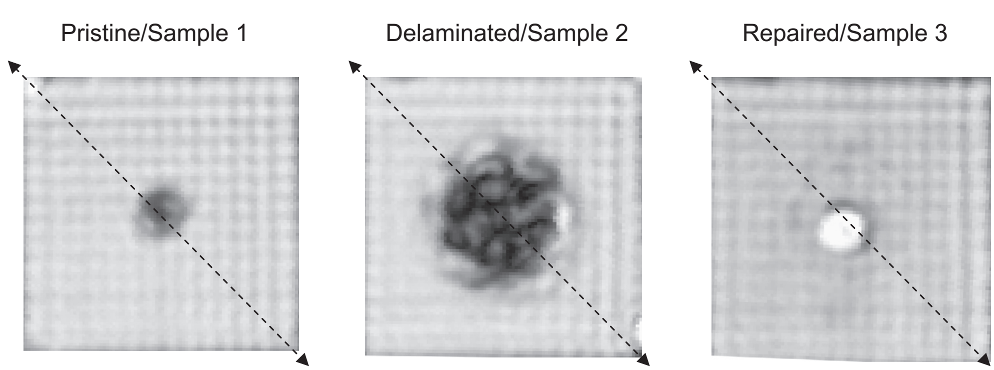


**Fig. 1.** C-scan images of pristine, delaminated, and repaired specimens used for fluorescent dye penetration and optical microscopy investigations.