Interfacial Properties of Electrodeposited Carbon Fibers Reinforced Epoxy Composites Using Fragmentation Technique and Acoustic Emission

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Abstract

Carbon fiber/epoxy composites using electrodeposited monomeric and polymeric coupling agents were compared with the dipping and the untreated cases. Treating conditions such as time, concentration and temperature were optimized. Four-fibers embedded micro-composites were prepared for fragmentation test. Interfacial properties of four-fiber composites with different surface treatments were investigated with simultaneous acoustic emission (AE) monitoring. The microfailure mechanisms occurring from fiber break, matrix and interlayer crackings were examined by AE parameters and an optical microscope. It was found that interfacial shear strength (IFSS) of electrodeposited carbon fibers was much higher than the other cases under dry and wet conditions. Well separated and different-shaped AE groups occurs for the untreated and ED treated case, respectively.

Keywords: Electrodeposition (ED), Interfacial shear Strength (IFSS), Four-fiber composites, Acoustic emission, Microfailure.

1. INTRODUCTION

IFSS can be improved by an introduction of chemical functional groups via the oxidation of fiber surface, plasma or commercial coupling agent treatments. Electrodeposition (ED) is a process that a polymeric film is deposited on a conductive fiber surface from a dispersion of colloidal ion in water with a charge opposite to that of the carbon fiber surface. By optimizing the treating process, a polymeric coating can be deposited with desired composition and thickness homogeneously to improve interfacial properties. The frequently-used micromechanical techniques to measure IFSS include the single fiber pull-out test, the fragmentation test (or called as a single-fiber-composites (SFC) test) etc [1-3].

The untreated, the dipping, and the ED carbon fibers/epoxy composites were evaluated using fragmentation technique with an aid of AE method to obtain the effect of the surface treatment on the interfacial adhesion. Microfailure modes and the inter-fiber distance effect were characterized while straining using the identical specimens under same load conditions.

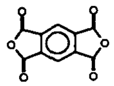
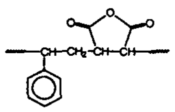
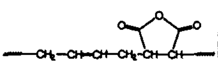
2. EXPERIMENTAL

2.1 Materials. Carbon fiber was supplied from Tae Kwang Co. (TZ-307). The carbon fiber has a density of 1.8 g/cm³ and average diameter of 7.9 μm. One monomeric pyromellitic dianhydrice (PMDA) and two polymeric coupling agents polystyrene-maleic anhydride (PSMA) and polybutadiene-maleic anhydride (PBMA) were used (Table 1). Epoxy (YD-128) resin was purchased from Kukdo Chemical Ind. Co Jeffamine D400 and D2000 were used as curing agents to control flexibility.

2.2 Methods

Fiber Surface Treatment by Dipping and ED. Fifty untreated carbon fibers with 7.9 μm diameter were fixed with regular distance apart in rectangular acrylic electrolytic frame. The frame acted as an anode in itself and the cathode was made of an aluminum plate bar with 2 cm width and 8 cm length as shown in figure 1. PBMA was diluted to the various concentrations with deionized distilled water. After anode frame and cathode bar were immersed into a certain electrolyte solution, voltage of 1.1 V was supplied to both electrodes by power source [4]. Typical locating time and applied voltage were 10 minutes and 1.1 V, respectively. After ED treated, carbon fibers were dried at room temperature without further thermal treatment, and some fibers were dried in the oven for comparison.

Table 1 Used one monomeric and two polymeric coupling agents.

	Chemical Name	Chemical Structure	Etc.
Monomeric	Pyromellitic dianhydride (PMDA) [1]		Type A
Polymeric	Polystyrene-maleic anhydride (PSMA) [2]		Type B
	Polybutadiene-maleic anhydride (PBMA) [2]		Type C

1) Aldrich, Ch.
2) Polysciences, Inc.

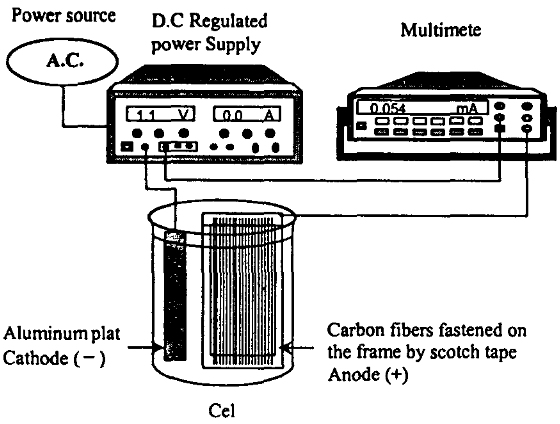


Fig. 1 Schematic plot of electrodeposition system.

Multi-Fiber Composites (MFC) Preparation. MFC specimens were made four-fibers embedded in epoxy matrix in silicon mould as shown in figure 2.

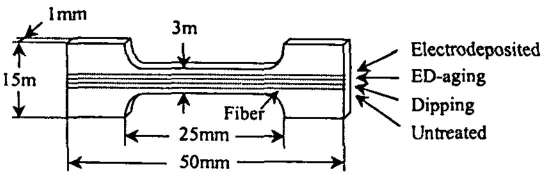


Fig. 2 Dimension of dogbone-shaped four fiber composites.

Single Fiber Strength and IFSS Measurement. The fragmentation test using MFC specimen was carried out to obtain IFSS using a specially-designed strain fixture. Ultimate fragment lengths within the matrix were measured, and subsequent failure process was observed via a polarized-light microscopy. The relationship among fiber tensile strength σ_f, aspect ratio, L_c/d and IFSS, τ was given by basically Drzal eq. and Weibull weakest link rule:

$$\tau = \frac{\sigma_f}{2\alpha}\Gamma\left[1 - \frac{1}{\beta}\right] , \quad \frac{\sigma_f}{\sigma_0} = \left(\frac{L}{L_0}\right)^{-\frac{1}{\rho}} \qquad (1)$$

where α and β are the scale and the shape parameters of the aspect ratio, respectively, and Γ is the Gamma function. σ_0 is the fiber strength at gauge length L_0, and ρ is the shape parameter of the Weibull distribution for fiber strength [5, 6]. Wet and aging tests were performed for the comparison.

Measurement of AE. Testing specimen was placed on the specially-designed desk-top tensile machine for applying unidirectional tensile force. AE sensor was attached to the center of the testing specimen using vacuum grease couplant.

After AE test, the number of fragmentation of fiber and the microfailure in the testing specimen were observed via the polarize microscopy. AE signals were detected by a wideband type sensor (model WD by PAC) with maximum sensitivity of −60 dB (ref. 1V/ubar) at 550 kHz. The sensor output was amplified by 60 dB at preamplifier and passed through a band-pass filter with a range of 100 kHz to 1200 kHz. Then the signal was fed into an AE signal processing unit, MISTRAS 2001 system. The threshold level was set to 30 dB. The output from preamplifier was also connected to the digital oscilloscope (LeCroy 9354A) [6, 7].

3. RESULTS AND DISCUSSION

3.1 Statistical Analysis of Fiber Strength Distribution. Using Weibull distribution, tensile strength of two treated fibers were compared with the untreated case. ED fiber showed rather lower values than dipping cases due to surface damage during ED procedure.

3.2 Effect of Treating Conditions on Adsorption Amount.

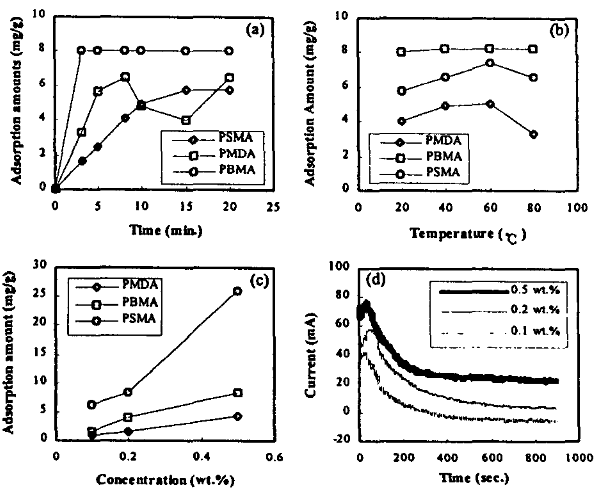


Fig. 3 The adsorption amount of coupling agents on carbon fiber bundle *versus* (a) treating time; (b) temp; (c) conc; (d) current change during ED for PMDA on conc.

In figure 3, currents increased initially, then decreased steadily. The higher solution concentration, the higher current. It is because adsorbed coupling agent depends upon the solution concentrations. Initial increase in current is due to the contribution of ionized coupling agents, whereas the following decrease.

3.3 Comparison of IFSS using Three Coupling Agents under Dry and Wet Conditions.

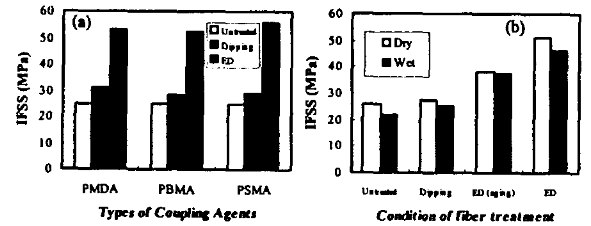


Fig. 4 IFSS depending on (a) different coupling agents; (b) fiber treatments and comparison of wet IFSS.

Figure 4 (a) shows the IFSS of ED, dipping, and the untreated case using three coupling agents. ED case shows significantly higher IFSS values than other two cases, especially dipping cases. It may be due to homogeneous coating on carbon fiber surfaces based on ED procedure. After dipping 80 ℃ for 30 minutes in figure (b), the ED treated IFSS shows the retention of adhesive bonding. Aged ED result appears rather lower IFSS value compared to ED case.

3.4 AE Analysis with Microfailure Mechanisms.

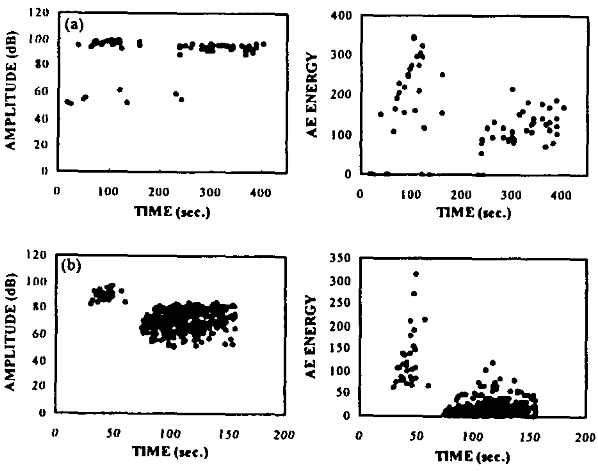


Fig. 5 AE amplitude and AE energy for (a) untreated; (b) ED composites as a function of measuring time.

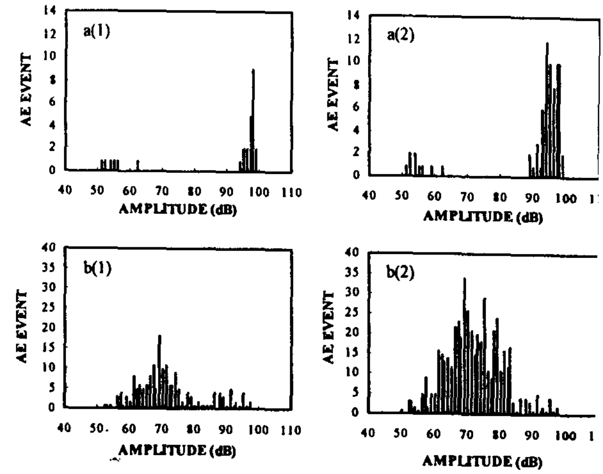


Fig. 6 Amplitude vs AE event depending on testing time: the untreated -- a(1) 0 to 120; a(2) 0 to 403 sec: ED -- b(1) 0 to 100; b(2) 0 to 153 sec.

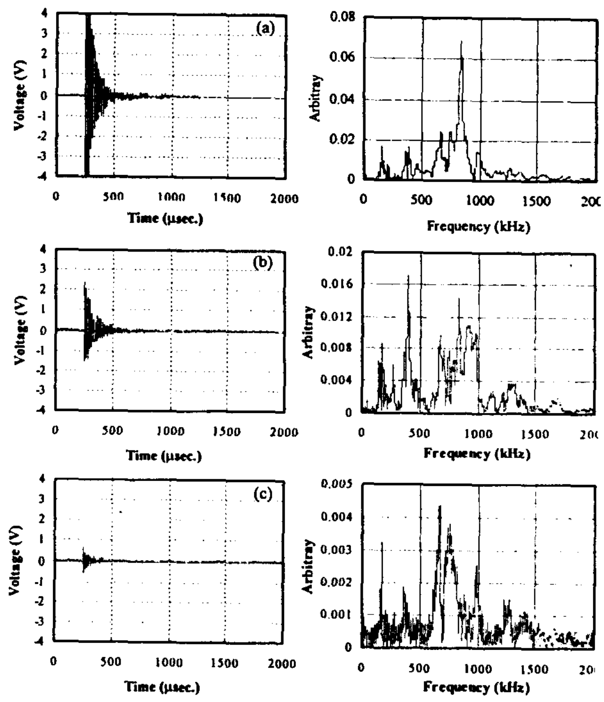


Fig. 7 AE waveforms from ED treated carbon fiber/epoxy composite while straining and fast *fourier* transform (FFT) results: (a) fiber breakage; (b) interlayer microfailure; (c) matrix cracking.

Figure 5 show AE amplitude and AE energy as a function of testing time. There are distinguishable two groups for both the untreated and ED cases. Compared to the untreated case, however, significant number AE events in the latter stage were observed, which may coming from interlayer microfailure and their matrix cracking. Figure 6 shows AE amplitude distributions of the (1) initial and (2) latter stages for (a) the untreated and (b) ED cases. After the latter stage, there are significant AE event occurring in the middle range. Figure 7 shows three different AE signals coming from the fiber breakage, the interlayer cracking, and matrix microfailure. Their relative three intensities were compared with each other. In addition, their characteristic frequencies by FFT analysis were observed, respectively.

4. CONCLUSIONS

Using monomeric and polymeric coupling agents carbon fiber/epoxy composites were compared in terms of treating time, concentration, and IFSS with an aid of AE. IFSS and their microfailure modes were correlated to each other under dry and wet conditions. Optimized treating conditions were obtained and the adsorption mechanism was investigated. DE treated case shows significant high IFSS compared to the untreated case, and especially the dipping case under both dry and wet conditions. AE results contributed to analyze IFSS significantly for the various treated carbon fibers.

ACKNOWLEDGMENT. This study was supported financially by KOSEF through Regional Research Center for Aircraft Parts Technology (ReCAPT), Gyeongsang National University, Korea.

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