Cure Monitoring and Nondestructive Evaluation of Carbon Fiber/Epoxy Composites by the Measurements of Electrical Resistance and AE

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Abstract

Cure monitoring and nondestructive characteristics of carbon fiber/epoxy composites were evaluated by the measurements of electrical resistance and acoustic emission (AE). Logarithmic electrical resistivity of the untreated single-carbon fiber composite increased suddenly to infinity when the fiber fracture occurred, whereas that of the electrodeposited composite increased relatively broadly up to infinity. As curing temperature increased, logarithmic electrical resistivity of steel fiber increased. On the other hand, electrical resistance of carbon fiber decreased due to the intrinsic electrical properties based on the band theory. The apparent modulus of the electrodeposited composite was higher than that of the untreated composite due to the improved interfacial shear strength (IFSS).

Key Words: *cure monitoring, electrical resistance, electrodeposition (ED), acoustic emission(AE), apparent modulus*

Designation

R: Electrical resistance, ρ: Electrical resistivity

ΔR: The difference of electrical resistance

1. Introduction

Many techniques were applied to investigate interfacial adhesion in composite materials. The most common micromechanical techniques to evaluate IFSS include the single-fiber pullout test and the fragmentation test (or called as single-fiber composite (SFC) test) *etc.* Recently, several researchers have evaluated composite characteristics by the measurement of electrical and micromechanical properties [1,2].

Temperature sensing and cure monitoring have been studied as an economical new evaluation for the monitoring of curing characteristics, interfacial properties and nondestructive behavior because conductive fiber can act as a sensor in itself as well as a reinforcing fiber. The electrical resistance difference and residual stress were investigated for single carbon fiber composite, and residual stress affected on the interfacial adhesion between fiber and matrix in composite materials. Electrical resistivity with microfailure mechanisms and nondestructive characteristics was recently studied in conductive fiber composites [3,4].

AE is well known as one of the important NDT methods. The AE can monitor the fracture behavior of composite materials, and can characterize many AE parameters to understand the type of microfailure sources during the fracture progressing. When tensile loading is applied to a composite, AE signal may occur from fiber fracture, matrix cracking, and debonding at the fiber-matrix interface. AE energy released by the fiber fracture could be greater than that associated by debonding or matrix cracking [5]. In this work, electrical resistance measurement and AE were used to evaluate cure monitoring and nondestructive characteristics in conductive carbon fiber composites.

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2. Experimental

2.1 Materials

Carbon fiber of 8 μm (Taekwang Industrial Co., Korea) in average diameter was used as conductive reinforcing materials. Testing specimens were prepared with epoxy resin (YD-128, Kukdo Chemical Co., Korea). Epoxy resin is based on diglycidyl ether of bisphenol-A (DGEBA). Polyoxypropylene diamines (Jeffamine D-400 and D-2000, Huntzman Petrochemical Co.) were used as curing agents. The flexibility of specimens was controlled by the mixing ratio of D-400 *versus* D-2000. Polybutadienemaleic anhydride (PBMA, Polyscience Inc.) was used as a polymeric coupling agent to improve IFSS by electrodeposition (ED).

2.2 Methods

2.2.1 Preparation of testing specimens

Two type specimens were used for cure monitoring and nondestructive evaluation. Fig. 1(a) exhibits a dogbone-shaped specimen to measure electrical resistivity change during fragmentation test and AE. Fig. 1 (b) shows the specimen for cure monitoring and stress-strain sensing.

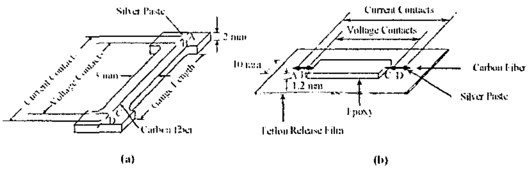

Fig. 1 Testing specimens

2.2.2 Measurement of electrical resistance

Electrical resistivity was obtained from the measured electrical resistance, cross-sectional area of the conductive fiber (A), and electrical contact length (L_{ec}) of the testing fiber connecting to copper wire. The relationship between electrical resistivity (ρ) and resistance (R) is as follows:

$$\rho = \left(\frac{A}{L_{ec}}\right) \times R \tag{1}$$

Electrical resistance was measured by four-point probe method as shown in Fig. 1. Silver paste was used as electrically connecting glue at junctions A, B, C and D. The voltage was measured between junctions B and C, and the current was supplied between junctions A and D. Total electrical resistance (R_{Tot}) between B and C may include R_c based on the contact resistance by silver paste beside R_f due to the electrical resistance by the fiber as follows:

$$R_{Tot} = R_c + R_f \tag{2}$$

Since the value of R_c is negligibly small due to very high conductivity of silver paste comparing to R_f, it can be considered that the voltage developed between junctions B and C becomes nearly fiber resistance,

$$R_{Tot} \cong R_f \tag{3}$$

2.2.3 AE measurement

AE sensor was attached in the center of the testing composite using vacuum grease couplant. AE signals were detected by a miniature sensor (Resonance Type, Model PICO, Physical Acoustics Corporation) with peak sensitivity of 54 Ref. V/(m/s) [68 Ref. V/mbar] and resonant frequency at 550 kHz. The sensor output was amplified by 40 dB at preamplifier and passed through a band-pass filter with a range of 200 kHz to 750 kHz. The threshold level was set as 40 dB. The signal was fed into AE processing unit (MISTRAS 2001 System).

3. Results and Discussion

3.1 Measurements of electrical resistance and AE

Fig. 2 shows the comparison of logarithmic electrical resistivity depending on the ED treatment in both single 8 μm and 18 μm carbon fiber composites. Logarithmic electrical resistivity of the untreated carbon fiber composites increased comparatively suddenly comparing to the ED carbon fiber composites. It can be because of the retarded fracture time due to the improved interfacial adhesion.

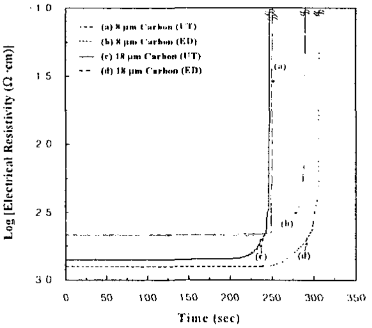

Fig. 2 Logarithmic electrical resistivity of various carbon fiber composites

When tensile stress was transferred from matrix to fiber by the external deformation, a reinforcing fiber could be endured well against the applied tensile stress and could not be broken easily. The precise observation of the shape of the electrical resistivity depending on fiber diameter exhibited the different trends. When a fiber was broken for the first time, the logarithmic electrical resistivity increased abruptly to infinity in the case of thin 8 μm fiber composite. On the other hand, the electrical resistivity exhibited smooth increment in the thicker 18 μm carbon fiber composite, and finally the electrical resistivity reached to infinity. It can be due to the fiber diameter effect by a very abrupt change of the electrical resistivity for thinner 8 μm carbon fiber composite than the thicker 18 μm case.

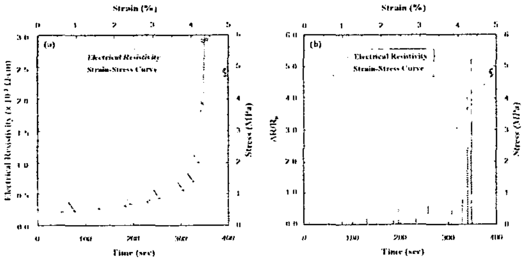

Fig. 3 Electrical resistivity for fiber fractures in ten-carbon fiber composite

Fig. 3 (a) shows the electrical resistivity and stress as a function of the elapsed time and the applied strain in 8 μm ten-carbon fiber/epoxy composites. When the first fiber fracture occurred in the individual carbon fiber, electrical resistivity increased stepwise in ten-carbon fiber composites. Arrow marks indicate the first fracture of each individual fiber. The electrical resistivity plot exhibited the exact amount of the change by the subsequent fiber fractures. Fig. 3(b) shows the electrical resistance difference ($\Delta R/R_0$) depending on the elapsing testing time and describes the relative changing ratio of the electrical resistance. Electrical resistance difference increased exponentially depending on the increase of the number of the first fracture of each carbon fiber. It can be because the electrical resistance of remaining fibers provide the dominant effect on the total electrical resistance.

Fig. 4 shows the electrical resistance and AE signals depending on the ED treatment in ten-carbon fiber composite. Electrical resistance change was in accordance with detected AE signals of the first fracture at the individual carbon fiber in both the untreated and ED treated composites. The AE event number of high AE amplitude group in Fig. 4(b) was larger than the number in Fig. 4(a). Especially, AE event number of the interlayer failure in the ED fiber composites exhibited a well-separated group, whereas the untreated composite did not show such an interlayer group.

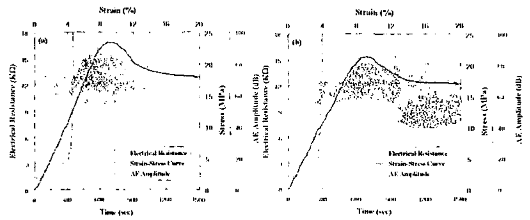

Fig. 4 Electrical resistance and AE depending on surface treatment

3.2 Temperature sensing and cure monitoring

Fig. 5 shows the comparison of electrical resistivity change in bare fiber without matrix and single-fiber embedded epoxy composite during curing process. As curing temperature increased, logarithmic electrical resistivity of steel fiber increased, whereas that of carbon fiber was trended to decrease. Electrical resistivity of SiC fiber exhibits nearly same behavior like carbon fiber. This can be due to the difference of inherent electrical properties between steel and carbon fibers based on the band theory. Logarithmic electrical resistivity of three bare fibers before curing is equal to the value after curing. On the other hands, electrical resistivity of their composites after curing was higher than that of the before curing. It could be explained that residual stress occurred during curing due to the different TEC between fiber and matrix.

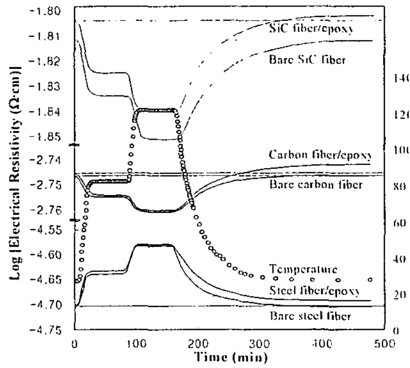

Fig. 5 Electrical resistivity of each fiber as function of curing temperature

Fig. 6(a) shows the behavior of the electrical resistivity for single carbon fiber/epoxy composites with curing temperature during curing process. As curing temperature increased, the difference in electrical resistivity before and after curing increased. This can be considered that relatively higher residual stress occurred for both the fiber and matrix at higher temperature. In Fig. 6(b), the difference in electrical resistivity before and after curing in the condition (3) with the optimum composition was largest under same curing temperature, whereas those of the condition (1) and (2) were smaller.

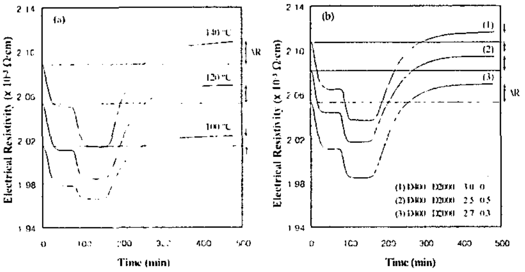

Fig. 6 The change of electrical resistivity during curing process

3.3 Stress-strain sensing

Fig. 7 shows the behaviors of stress-strain and electrical resistivity of the (a) untreated and (b) ED treated carbon fiber composites under 3 cyclic loadings. The elapsed measuring time of the ED treated composite is shorter than that of the untreated composite. This can be due to the enhanced interfacial properties.

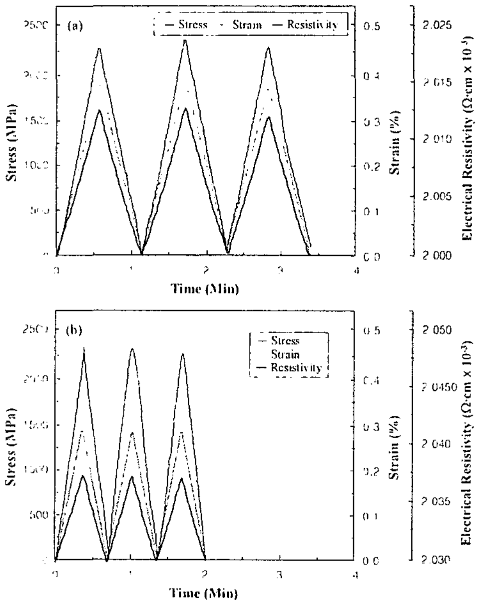

Fig.7 Electrical resistivity and stress-strain as function of the surface treatment

The strain of the ED treated carbon fiber case is smaller than that of the untreated carbon fiber composite until the maximum load. This can be due to higher apparent modulus of composite due to the improved IFSS. The behavior of electrical resistivity for the fiber surface modification was responded quantitatively well for the change of stress-strain.

4. Conclusions

Cure monitoring and nondestructive characteristics were evaluated by the measurement of electrical resistance and AE. Logarithmic electrical resistivity of the untreated case increased suddenly to infinity, whereas the ED case increased broadly to infinity. In single- and multi-carbon fiber composites, the number of AE signals of the ED treated cases is much more than that of the untreated cases. As curing temperature increased, the difference of electrical resistivity before and after curing increased in carbon fiber composites. This can be due to higher residual stress at higher curing temperature. The elapsed measuring time of the ED treated composite was shorter than that of the untreated composites. This can be due to the improved interfacial properties. The behavior of electrical resistivity was responded quantitatively with the change of stress-strain. Electrical resistance measurement and AE can be very useful tools to evaluate cure monitoring and nondestructive characteristics in conductive carbon fiber composites.

Acknowledgement

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