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Cure monitoring and residual stress sensing of single-carbon fiber reinforced epoxy composites using electrical resistivity measurement

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Abstract

Temperature sensing and cure monitoring of the single-carbon fiber/epoxy composites were studied by the measurement of electrical resistivity as a new approach. IFSS and the difference in electrical resistivity (ΔR) between before and after curing were highest for the smallest gauge length of the specimen. As curing temperature increased, logarithmic electrical resistivity of steel fiber increased due to the increased the mean path of the free electron. On the other hand, those of semi-conductive carbon and SiC fibers decreased due to the intrinsic electrical properties based on the band theory. Residual stress built in the fiber was highest at the fiber axis direction, whereas residual stress of built in the matrix was relatively higher for the fiber circumference and radius direction. Residual stresses by the calculating method were consistent well with those from the finite element analysis (FEA). The behavior of electrical resistivity was responded well quantitatively with the change of curing temperature and epoxy matrix modulus. Electrical resistance measurement of conductive fiber composites can be applicable for the useful technique to evaluate cure monitoring and residual stress sensing.

Keywords: B. Interfacial strength; C. Residual stress; D. Acoustic emission; Electrical resistivity; Cure monitoring

1. Introduction

Recently a new evaluation technique of interfacial properties as well as curing characteristics and the residual stress were investigated by the measurement of electrical resistivity using various conductive fiber reinforced composites. The conductive fiber reinforced composites were studied as new self-strain and/or self-damage health monitoring sensors under temperature change and under applied load [1–3].

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 [4,5]. 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 [4]. For carbon fiber/polymer composites, the endothermic peak at the annealing temperature and frictional change in electrical resistance were studied by differential scanning calorimetry (DSC) and DC electrical resistance measurement. During cyclic heating/cooling

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processes, thermal interlaminar damages were also evaluated by the measurement of contact resistivity of carbon fiber laminates [5].

Cure monitoring and their characterization including residual stress were studied by means of the common methods such as thermal and dielectric analysis. To minimize residual stress in the thermosetting epoxy, the optimum cure cycle was evaluated using dilatometer and DSC. The volume change depending on the elapsed time and temperature was measured during various curing cycles. In DSC the differential heat flow between the sample and the reference was measured with linearly programmed temperature rise [6].

Dielectric sensor was used to measure the cure degree of carbon fiber/epoxy composite. Dielectric analysis or dielectrometry can be used to investigate the processing characteristics and chemical structure of polymers and other organic materials by measuring their dielectric properties. Under electrical field, the change of electrical dipole alignment and the charged ion mobility can be used to infer the information concerning on the bulk material properties, such as its viscosity, rigidity, reaction rate, and cure state, etc. During cure monitoring, dipoles and charged ions in dielectric materials could be rearranged under AC electrical filed. The alignment of dipoles and ions might be allowed during curing. However, the alignment could be difficult as the curing progressed [7–9].

Residual stress in the fiber-reinforced composite occurred usually due to thermal contraction of the matrix or the difference of thermal expansion coefficient (TEC) between fiber and matrix. This stress may affect on the interface between fiber and matrix, and may have a great influence on the mechanical performance of microcomposites. Residual stress can affect actually on the fiber stress or the interfacial shear strength (IFSS) [10,11].

The measurement of residual stress/strain on the fiber can be measured by Raman scattering method [12,13] and other optical technique [14]. These techniques are difficult to measure directly residual stress because the fiber strain is anisotropy and the fiber is embedded in the matrix. Genidy et al. [11,15] and Madhukar et al. [16–18] attempted to measure directly residual compressive stress to obtain optimized curing cycle with free stress. Inhomogeneous structure of polymeric composites causes to develop internal stresses due to matrix volume change during curing. Volume change occurs during curing and cooling down processes. Most of the previous studies [11–18] on residual stress were concentrated on the stress development during cooling down. A new testing method in this work was used to monitor the fiber stress that developed around single fiber composites (SFC) during heating or cooling. A relationship between cure cycle and residual stress was studied and it was shown that cure-induced stress and residual stress vary with different resin types.

The residual stress in SFC was experimentally evaluated by the technique based on the continuous monitoring during fragmentation testing [19]. The difference between the strain at the break of a single fiber in air and the strain embedded in a polymer matrix could be measured as a function of temperature. By considering the compressive fiber modulus, the strain difference might be converted into fiber compressive stress related to the matrix thermal shrinkage after curing the specimen [19]. Residual stress in bismaleimide/carbon fiber composite model depending on the curing cycle was studied using finite element analysis (FEA) [20]. The comparison of theoretical prediction, numerical simulation, and the experimental result shows a generally good agreement.

In this work, during curing process, the electrical resistance of carbon fiber composite was measured to evaluate curing characteristics and residual stress change depending on the gauge length, curing temperature and matrix modulus with differing curing agent ratios. Residual stress was measured by the von Mises criterion and then the equivalent stress was evaluated by FEA. The relationship between the electrical resistivity, residual stress and IFSS was investigated for single carbon fiber/epoxy composites. Ultimately the temperature sensing and cure monitoring were correlated by the electrical resistance measurement.

2. Experimental

2.1. Materials

Carbon fiber of 8 μm (Taekwang Industrial Co., Korea) and of 18 μm (Mitsubishi Chemical Co., Japan) in average diameter was used as conductive reinforcing materials. SiC fiber (Textron Co.) of 138 μm and steel fiber (No. 1 guitar string of Segovia Instruments Co., Korea) of 280 μm were used for comparison. 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 mixing ratio of D-400 versus D-2000.

2.2. Methodologies

2.2.1. Preparation of testing specimens

Two type specimens were prepared for electro-micromechanical test and curing monitoring. Fig. 1(a) exhibits a dogbone-shaped specimen to measure electrical resistivity change during curing, and then to evaluate IFSS