Nondestructive Damage Sensing and Cure Monitoring of Carbon Fiber/Epoxyacrylate Composite with UV and Thermal Curing using Electro-Micromechanical Technique

Jin-Woo Kong*, Dae-Sik Kim*, Joung-Man Park*,†, Jae-Rock Lee**

Electro-Micromechanical 시험법을 이용한 탄소섬유 강화 Epoxyacrylate 복합재료의 UV 및 열경화에 따른 비파괴적 손상 감지능 및 경화 Monitoring

공진우* · 김대식* · 박종만*,† · 이재락**

Key Words: ultraviolet (UV) curing, electrical resistance (ER), electro-micromechanical technique, apparent modulus, interfacial shear strength (IFSS), residual stress, TEC

ABSTRACT

Interfacial evaluation, damage sensing and cure monitoring of single carbon fiber/thermosetting composite with different curing processes was investigated using electro-micromechanical test. After curing, residual stress was monitored by measurement of electrical resistance (ER) and then it was compared to correlate with various curing processes. In thermal curing, curing shrinkage appeared significantly by matrix shrinkage and residual stress due to the difference in thermal expansion coefficient (TEC). The change in electrical resistance (ΔR) on thermal curing was higher than that on ultraviolet (UV) curing. For thermal curing, apparent modulus was the highest and reaching time until same strain was faster. So far thermal curing shows strong durability on the IFSS after boiling test.

Nomenclature

τ	: Interfacial shear strength (IFSS)
σ_R	: Residual stress
ΔR	: Change of electrical resistance
$\Delta\rho$	: Change of electrical resistivity
L_{ec}	: Electrical Contact length
TEC	: Thermal expansion coefficient

1. INTRODUCTION

Thermosetting resins such as epoxy, unsaturated polyester (UP) and vinyl ester can be cured by thermal process involved room temperature, ultraviolet (UV) radiation and combination of mentioned two methods. UV curing process has a very attractive advantages compared to thermal curing. Their major advantages are a high-speed process, low energy consumption due to operating in room temperature, and environmentally friendly because of avoiding solvent [1,2]. The electro-micromechanical technique had been studied as an economical and new nondestructive evaluation (NDE) method for curing monitoring, stress or strain sensing, characterization of interfacial properties, and nondestructive behavior because conductive fiber can act as a sensor in itself as well as a reinforcing fiber [3,4]. Residual stress in the fiber-reinforced composite occurs during curing process due to the thermal contraction of the matrix and/or the difference in thermal expansion coefficient (TEC) between fiber and matrix. The effect on residual stress is reflected in the mechanical performance of cured composites [5]. In this work, interfacial properties, damage sensing and cure monitoring of carbon fiber reinforced epoxyacrylate composites were investigated using micromechanical test and electrical resistance (ER) measurement. The change in electrical resistance (ΔR) during curing process, under

---

*Department of Polymer Science and Engineering,
Engineering Research Institute,
Gyeongsang National University
**Advanced Material Division,
Korea Research Institute of Chemical Technology
†To whom correspondence should be addressed.

cyclic changeable stress/strain and durability test were correlated with mechanical properties of matrix with curing conditions.

2. EXPERIMENTAL

2.1. Materials

Two kinds of carbon fibers were used and their average diameters were about 18 μm (Mitsubishi, Chemical Co., Japan) and 8 μm (Taekwang Co., TZ-307, Korea), respectively. E-glass (Dow Corning Co.) and C-glass fibers (Gangnam Co., Korea) were used and their average diameters were about 30 μm and 10 μm. Epoxyacrylate resin (Construction Chemical Co., Korea) was used as a matrix and 1-hydroxy cyclohexyl phenyl ketone (IR-184C, Ciba Co., Switzerland), was used as a photoinitiator.

2.2. Methodologies

2.2.1. Specimen Preparation and IFSS Measurement: The carbon fiber with 18 μm diameter was fixed with regularly separated distance in a steel frame. Microdroplets of epoxyacrylate matrix were formed on each carbon fiber of 18 μm using carbon fiber of 8 μm in diameter as a tip pin. Microdroplet specimens were cured with UV and thermal conditions. A microdroplet specimen was fixed by the microvice using a specially designed micrometer. The IFSS, τ was calculated from the measured pullout force, F using the following equation,

$$\tau = \frac{F}{\pi D_f L} \tag{1}$$

where D_f and L are fiber diameter and fiber embedded length in the matrix, respectively.

2.2.2. Electrical Resistance (ER) Measurements: Figure 1 shows the scheme of (a) UV curing process and electrical resistance measurement (b) during curing and (c) under cyclic loading. During curing process and under cyclic loading, the electrical resistance was measured using a HP34401A digital multimeter. For durability test the specimen was soaked in 98°C boiling water for 3 hours and absorbed moisture was evaporated during measuring the electrical resistance. For the electrical resistance measurements under cyclic load, strain-stress curves were measured by mini-UTM (Hounsfield Test Equipment Ltd., U.K.). Testing speed and load cell were 0.5 mm/minute and 100 N, respectively. The calculation method of the electrical resistivity, ρ is as follows:

$$\rho = \left(\frac{A}{L_{ec}}\right) \times R \tag{2}$$

where R is the electrical resistance, A is the cross-section area of conductive fiber, and L_{ec} is the electrical contact length between voltage contacts.

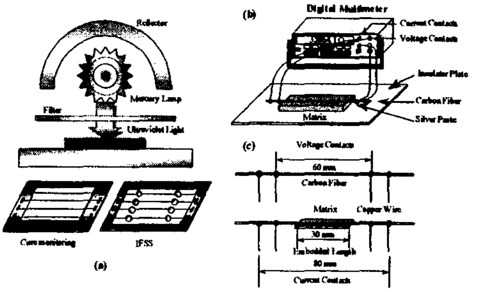

Fig. 1 Scheme of (a) UV curing process and electrical resistance measurements (b) during curing and (c) under cyclic loading.

3. RESULTS AND DISCUSSION

3.1 Cure Shrinkage: Figure 2 shows phenomenon of cure shrinkage for various fibers/epoxyacrylate composites in (a) thermal and (b) UV curing. Cure shrinkage may depend on matrix shrinkage and thermal shrinkage. For thermal curing, fiber waviness was observed for all fibers due to high cure degree and thermal shrinkages, and good interfacial adhesion. In case of kevlar and zylon fibers, kink band was observed instead of fiber waviness. This kink band was induced by compressive stress. For UV curing, any fiber waviness was not observed for all fibers. It might be because of the fast curing time and low interfacial adhesion.

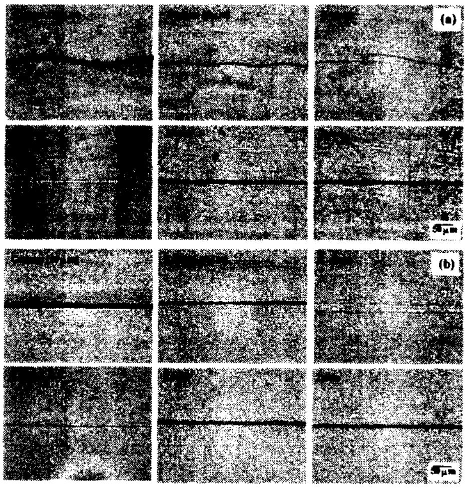

Fig. 2 Cure shrinkage of various fibers/epoxyacrylate composite: (a) thermal; and (b) UV curing.

3.2 Cure Monitoring: Figure 3 shows the temperature change of (a) air state and (b) epoxyacrylate matrix during curing. For air state without matrix, temperature increased smoothly and then decreased during cooling process. In case of epoxyacrylate matrix, temperature increased suddenly due to the heat of curing. Theses results indicate that epoxyacrylate is cured well by UV.

Figure 4 shows the change of ER for (a) thermal curing and (b) UV curing. In case of thermal curing case, ER decreased with increasing temperature and then they increased during cooling process. Change of temperature was corresponded reversibly with ER. Final ER increased more compared to the initial state. The changes in ER (ΔR) during curing may be affected by matrix thermal shrinkage or residual stress by matrix mechanical properties. For UV curing, ER decreased suddenly by heat of matrix curing and then increased gradually during cooling process. ΔR in UV curing case was lower than that of thermal curing case. The result indicates that residual stress in thermal curing is higher than that of UV curing case in Figure 2(a).

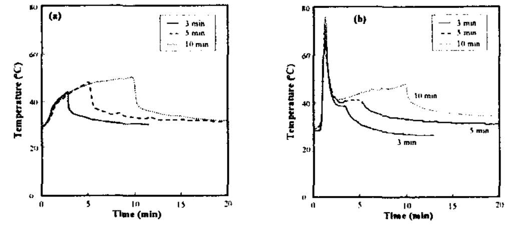

Fig. 3 Temperature change (ΔT) of (a) without matrix; and (b) epoxyacrylate matrix during UV curing.

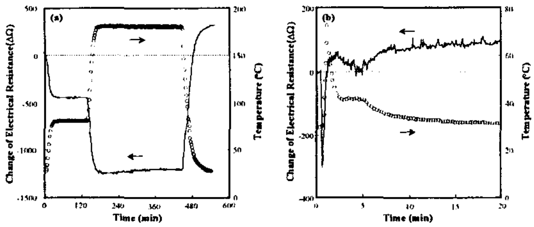

Fig. 4 Cure monitoring for carbon fiber/epoxyacrylate: (a) thermal; and (b) UV curing.

3.3 IFSS and Microfailure Mode: Interfacial properties of carbon fiber reinforced epoxyacrylate composite were compared with differing curing conditions. Figure 5(a) shows the force-extension curves with thermal and UV curing. Applied force was transferred from matrix to fiber until the maximum force reached and then pulling-out occurred. After pulling-out, the frictional force appeared differently with fiber surface roughness and interfacial adhesion, etc. Maximum pullout and frictional forces of thermal curing case were higher than those of UV curing case. It might be due to high cross-linking density and interfacial adhesion. Figure 5(b) shows the IFSS of carbon fiber/epoxyacrylate with thermal curing and UV curing time. IFSS of carbon fiber/epoxyacrylate composite cured by thermal process was higher than UV curing cases. In UV curing case IFSS was improved with increasing UV curing time until 5 minutes and then saturated. It may be due to the difference of degree of curing and cross-linking density.

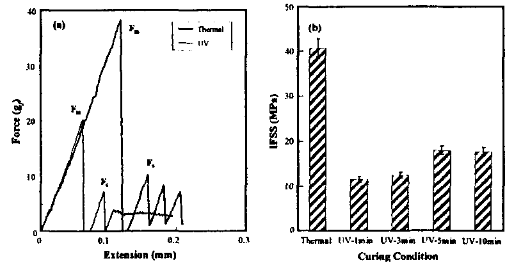

Fig. 5 (a) Force-extension curves and (b) IFSS of carbon fiber/epoxyacrylate composite with thermal and UV curing.

Figure 6 shows typical microfailure modes for the carbon fiber reinforced epoxyacrylate composite with (a) thermal and (b) UV curing. For thermal curing, epoxyacrylate microdroplet exhibited more likely plastic deformation and ductile microfailure mode, whereas in case of UV curing case microdroplet appeared brittle microfailure mode. Meniscus angle between fiber and matrix in thermal curing was lower than that of UV curing case. In case of thermal curing, viscosity of epoxyacrylate was low compared to UV curing case due to the cure at high temperature. Meniscus angle indicates surface wetting and interfacial adhesion.

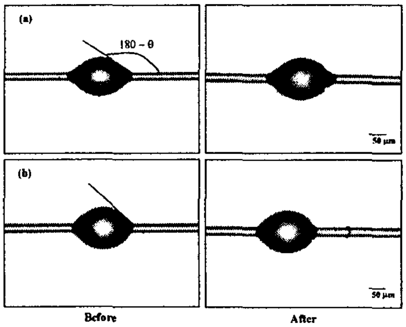

Fig. 6 Typical microfailure modes of carbon fiber/epoxyacrylate: (a) thermal; and (b) UV curing.

3.4 Damage Sensing and Durability: Under the same strain, the stress of carbon fiber/epoxyacrylate composite for measuring ER appeared differently with their mechanical properties of matrix. Figure 7 shows the changes of stress for (a) thermal and (b) UV curing under cyclic strain. The stress of thermal curing case was higher than that of UV curing case. Figure 8 shows the changes of strain and electrical resistivity for (a) thermal curing and (b) UV curing under changeable stress. The strain and ER of thermal curing case were higher than those of UV curing case, and the reaching time until the same stress was faster in thermal curing case. This tendency could be related to their apparent modulus, which provides the interfacial information: i.e., apparent modulus means the fiber modulus embedded in the matrix in stress-strain curve comparing to the carbon bare fiber modulus in itself [6]. Figure 9(a) shows strain-stress curves with curing conditions. The slope in curve was apparent modulus that increased with improving matrix modulus. Apparent modulus was consistent well with ΔR shown in Figure 9(b).

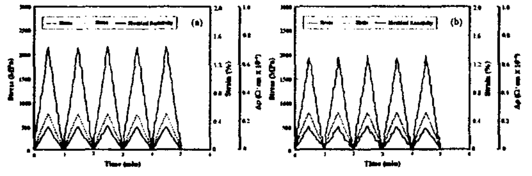

Fig. 7 Changes of stress and electrical resistivity under cyclic strain.

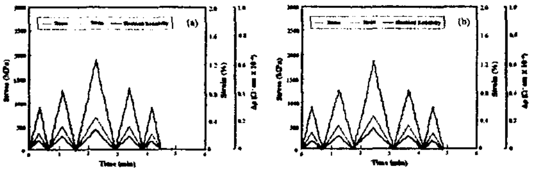

Fig. 8 Changes of strain and electrical resistivity under changeable stress.

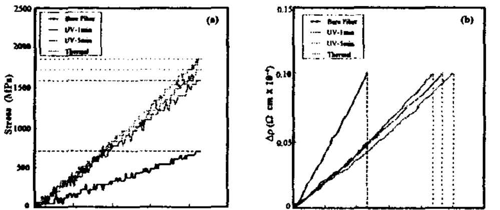

Fig. 9 (a) Strain-stress curves and (b) Δρ for apparent modulus.

Figure 10 shows the changes of electrical resistivity for carbon fiber/epoxyacrylate composite under durability test. Electrical resistivity increased gradually. It could be because evaporating moisture from matrix resulted in the recovering matrix modulus. The change of electrical resistivity was the lowest in thermal curing case due to their high cross-linking density and low moisture absorption.

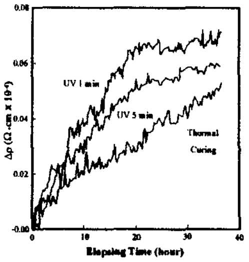

Fig. 10 Δρ for carbon fiber/epoxyacrylate composite under durability test.

4. CONCLUSIONS

Interfacial evaluation, nondestructive damage sensing and cure monitoring of carbon fiber/epoxyacrylate composites were performed using microdroplet test and electrical resistance measurement. In thermal curing, curing shrinkage appeared significantly by matrix shrinkage and residual stress due to the difference in thermal expansion coefficient (TEC). IFSS of carbon fiber/epoxyacrylate composite cured by thermal process was higher than UV curing cases. In UV curing case IFSS was improved with increasing UV curing time until 5 minutes and then saturated. It may be due to the difference of curing degree. The change in ER on thermal curing was higher than that on UV curing, which indicates higher residual stress and IFSS. For thermal curing, stress up to same strain was the highest and reaching time until same stress was faster. These tendencies could be related to their apparent modulus, which can provide the interfacial information. The highest apparent modulus for thermal curing case was correlated consistently with IFSS and matrix modulus involving cross-linking density.

ACKNOWLEDGMENT: This work was financially supported from CAMP of the 21th Frontier R&D Program funded by the Ministry of Science and Technology, Korea though Engineering Research Institute (ERI), Gyeongsang National University.

REFERENCES

(1) J. Y. Zhang, G. Windall and I. W. Boyd, *Appl. Surf. Sci.*, 7383(2001), pp. 1-5.
(2) E. Bemporad, F. Carassiti, A. Tara, G. Gallinaro and M. Paris, *Rad. Phy. Chem.*, 57(2000), pp. 393-397.
(3) S. Wang, S. I. Lee, D. D. L. Chung and J. M. Park, *Compos. Interf.*, 8(6) (2001), pp. 435-441.
(4) J. M. Park, S. I. Lee, K. W. Kim and D. J. Yoon, *J. Colloid Interf. Sci.*, 237(2001), pp. 80-90.
(5) S. Lee and G. S. Springer, *J. Compos. Mater.* 22, 1998, p. 15.
(6) S. Wang, S. I. Lee, D. D. L. Chung and J. M. Park, *Compos. Interface*, 8(6), 2001, p. 453.