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# Fatigue life evaluation and crack detection of the adhesive joint with carbon nanotubes



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#### ABSTRACT

For a composite material having a high specific strength and specific stiffness with excellent damping and good impact properties, joint design is a very important consideration because an improper design may lead to overweight or defective structures. Adhesive bonding does not require holes and distributes the load over a larger area than mechanical joints. As the use of adhesively bonded joints subjected to cyclic loading has increased in recent years, it is important to measure and improve the fatigue cracking and the lifetime of these adhesive joints.

In this paper, the static and dynamic strengths of adhesive joints incorporating carbon nanotubes were compared to those of adhesive joints without carbon nanotubes. Composite to aluminum single-lap joints were fabricated and their strengths were evaluated.

From the tests, fatigue strengths of the adhesive joints increased when the adhesive of the adhesive joint had carbon nanotubes although their static strengths decreased. Also, crack initiation and propagation can be effectively detected by measuring the variation of equivalent resistance when carbon nanotubes are dispersed into the adhesive in the adhesive joint.

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#### 1. Introduction

Joint design in composite structures is a very important consideration because improper design may lead to overweight or defective structures. The joining of composite materials has traditionally been achieved by mechanical fastening or adhesive bonding [1–3]. Adhesive joints can distribute the load over a larger area than mechanical joints and they have excellent insulating properties, superior damping and noise reduction capability. Of the commonly used structural adhesives, the epoxy-based adhesives are widely employed for joining various components largely because of their relatively high modulus and strength. However, since adhesive bonding joints are very sensitive to surface treatment, service temperature and other environmental conditions, they are not preferred for use in joining primary structures. To improve the mechanical properties of an adhesive and its joint, the metallic or non-metallic powders have been widely used as filler material [4].

The discovery of carbon nanotubes (CNTs) with their exceptional mechanical properties has lead to novel approaches, including using them as reinforcing nanofillers in composite materials [5]. Davey [6] has demonstrated that carbon nanotubes (CNTs) provide the potential for improving resin-dominated

properties, such as interlaminar strength, toughness, and thermal and environmental durability. Srivastava [7] has studied the effects of the addition of inorganic nano-particles and demonstrated that MWCNT filled epoxy resin adhesive gives higher bonding strength for C/C and C/C-SiC substrates than the unfilled epoxy resin bonded substrates. Yu et al. [8] has proved that multi-walled carbon nanotubes (MWCNT)/epoxy resin composite gives excellent fracture toughness and fatigue strength. The addition of MWCNT in epoxy can significantly improve the fatigue life of epoxy. Shokrieh and Rafiee [9] has studied the tensile behavior of an embedded carbon nanotube in polymer matrix with non-bonded interphase region. Rahman et al. [10] has investigated the effect of interaction of MWCNTs with epoxide groups and fiber/matrix bonding using scanning electron microscope (SEM). All results were compared with the control (reference) epoxy composites results containing no MWCNTs.

Vega et al. [11] has used single-walled carbon nanotubes (SWCNTs) as a sensor and proved that SWCNTs could be used to monitor internal stresses developing during the curing process of thermoset materials. Also, method of on-line health monitoring of adhesive joints using a fiber-optics or piezoelectric sensor were proposed by several researchers [12–14].

In this paper, the static and dynamic strengths of adhesive joints with carbon nanotubes were compared to those of adhesive joints without carbon nanotubes. Composite to aluminum single-lap joints were fabricated and their strengths were





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evaluated. Also, the equivalent resistance and capacitance of the adhesive layer were measured by means of a fabricated electric circuit and the crack initiation and propagation of the adhesive joints were evaluated.

#### 2. Manufacture of the adhesive joint

The single lap joints were manufactured by ASTM D1002, D5868 standard and their schematic diagrams are shown in Fig. 1. The adhesive length and thickness were 40 mm and 2 mm, respectively. 6061-T6 aluminum and USN 125 carbon epoxy prepreg were used for the composite to aluminum adhesive joints. The stack sequence of the composite specimen was [0/45/0/-45]2s and its mechanical properties are summarized in Table 1. The USN 125 carbon epoxy prepreg were cured in an autoclave at 120 °C for 120 min.

Epoxy adhesives (KSR 177) and hardener (G 640) from KUKDO Chemical Co. were used for the adhesive joint. Table 2 shows the mixing ratio and strength of adhesives. Hanwha Nanotech Co. CM 95 carbon nanotubes were used and their diameter and length were 10-15 nm and  $10-20 \mu \text{m}$ , respectively. The 80E three-roll mill of EXAKT Co. were used and 2 wt% carbon nanotubes were dispersed into the adhesive. The mixing and dispersion of the nanotubes were conducted by the three-roll mill and the roll gaps were adjusted. The three-roll mill was used one time at the 20  $\mu$ m gap, one time at the 15  $\mu$ m gap and five times at the 10  $\mu$ m gap. The adhesive with dispersed carbon nanotubes was applied to the aluminum composite specimen and a fixture was used for controlling the adhesive thickness. Fig. 2 shows the schematic diagram of the adhesive joint fixture. The surface treatment of the adherend greatly affects the strength and failure mode of the adhesive joint. The surfaces of the aluminum and composites were polished with 120 mesh sandpaper and were corroded by 10% nitric acid and 90% ethanol for five minutes. After the corrosion step, the surfaces were cleaned and dried using acetone. An adhesive thickness of 2.0 mm was controlled by the adhesion fixture, as shown in Fig. 2. The assembled adhesive joints were cured in an oven at 80 °C for 120 min. The cured adhesive joints were cut by diamond wheel cutter and the residuary fillets of the adhesive joints were removed using a razor.

#### 3. Tensile test of the adhesive joint

Composite to aluminum adhesive joints were manufactured and their strengths were evaluated. An Instron Co. 5582 universal testing machine was used for the tensile test of the adhesive joint and its crosshead speed was 1.27 mm/min.

Fig. 3 shows the force-displacement curves of the adhesive joints. As shown in Fig. 3, the strengths of adhesive joints without carbon nanotubes were 4.86 MPa, and the strengths of adhesive

#### Table 1

Material properties of carbon/epoxy composite material of SK co.

Property	Symbol	Value
Elastic modulus in fiber-direction Elastic modulus in transverse directions Shear modulus in 1–2 and 1–3 planes Shear modulus in 2–3 plane	E1 E2 G12, G13 G23	131.0 GPa 8.20 GPa 4.50 GPa 3.50 GPa
Poisson's ratios Tensile strength in fiber-direction Compressive strength in fiber-direction Tensile strength in transverse direction Compressive strength in transverse direction Shear strength in 1–2 and 1–3 planes Shear strength in 2–3 planes	V12, V12 V23 XT XC YT YC S12, S13 S23	0.281 0.470 2000 MPa 1400 MPa 61 MPa 130 MPa 70 MPa 40 MPa

### Table 2

Material properties of the epoxy adhesive.

Item	KSR-177	
Lap shear strength	80.8	
Substrate condition: No sanding treatment on Aluminum		
Mixing ratio: KSR-177/G-640 = 100/53		
Curing condition: 80 °C for 2 h		



Fig. 2. Schematic diagram of the adhesion fixture.

joints with 2 wt% carbon nanotubes were 3.08 MPa. Therefore, the strengths of adhesive joints with 2 wt% carbon nanotubes were about 36.62% lower than those of adhesive joints without the carbon nanotubes.

Fig. 4 shows the fractured surfaces of the adhesive joints after the tensile test. As shown in Fig. 4(a), inter-laminar failure of composite adherend was observed in the adhesive joint without carbon nanotubes. However, in Fig. 4(b), it is not possible to distinguish



Fig. 1. Schematic diagram of the adhesive single lap joint.

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