



# Effects of dispersion methods and surface treatment of carbon nanotubes on defect detectability and static strengths of adhesive joints



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## ABSTRACT

Adhesive bonding and bolting are two typical joining methods of composites. Adhesive bonding does not require holes, and the load is distributed over a larger area than that in mechanical joints. However, such bonding is highly sensitive to the surface treatment, service temperature, humidity, and other environmental conditions. In particular, the formation of kissing bonds (due to surface defects or contamination) reduces the bonding strength significantly. The electrical-impedance method using carbon nano-tubes (CNTs) is a highly promising technology that helps in detecting different types of bonding defects. In this study, aluminum-to-aluminum adhesive joints with 1 wt% CNTs were fabricated. The static strengths and defect detectabilities determined using the electrical-impedance method were then evaluated. To uniformly disperse the CNTs into the adhesive, a sonication process, a three-roll-mill process, and solvent were employed. The defect detectabilities and static strengths of the adhesive joints produced using five different types of dispersion methods were then evaluated. Moreover, the defect detectability and static strength of the adhesive joints with respect to the surface treatment of the CNTs were investigated.

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## 1. Introduction

Adhesive bonding and bolting are two typical methods of joining composites. Adhesive bonding does not require holes. Moreover, the loads are distributed over a larger area than that in mechanical joints [1]. However, adhesive-bonded joints are very sensitive to the surface treatment, service temperature, humidity, and other environmental conditions. In particular, the presence of kissing bonds (due to surface defects or contamination) leads to serious degradation of the bonding strength.

Carbon nano-tubes (CNTs), which have remarkable mechanical and electrical properties, have been investigated over the past two decades because of their many possible applications. Yu [2] proved that multi-walled carbon nanotubes (MWCNTs)/epoxy-resin composite have excellent fracture toughness and fatigue strength. Jojibabu et al. [3] studied the rheological properties, thermal stability, and lap shear strength of epoxy adhesive joints reinforced with different carbon nanofillers, such as MWCNTs, graphene nanoplatelets (GNP), and single-walled carbon nanohorns (CNH). Kim et al. [4] studied the rheological properties and mechanical properties of CNT/epoxy composites with respect to their surface modifica-

tion. Wernik and Meguid [5] experimentally proved that the improvement in the mechanical properties of CNT-reinforced epoxy adhesives was the most significant for CNTs in the range of 1.0–1.5 wt%. Zielecki et al. [6] demonstrated that the fatigue properties of peel-loaded adhesive joints were improved by dispersing 1 wt% MWCNTs in the epoxy-based adhesives.

Thostenson and Chou [7] studied an in situ-sensing method to detect localized damage of mechanically fastened joints using 0.5-wt% CNTs. Kang et al. [8] detected the initiation and propagation of cracks by measuring the variation in the equivalent resistance of the adhesive joints containing CNTs. Kim et al. [9] suggested an electrical-impedance method to detect the surface defect in an adhesive joint. They proved that the electrical-impedance method using CNTs is a very promising technology that helps detecting different types of bonding defects.

In this study, aluminum-to-aluminum adhesive joints with 1 wt% CNTs were fabricated. The static strengths and defect detectabilities were then evaluated using the electrical-impedance method. To uniformly disperse the CNTs into the adhesive, a sonication process, three-roll-mill process, and solvent were employed. The defect detectabilities and static strengths of the adhesive joints obtained using five different types of dispersion methods were evaluated. Additionally, the defect detectability and static strength of the adhesive joints with respect to surface treatment of CNTs were investigated.

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## 2. Manufacturing process

The aluminum-to-aluminum adhesive single lap joints with CNTs were fabricated. Aluminum alloy 5052, along with an epoxy adhesive (YD-128) and a hardener (G-640) obtained from KUKDO Chemical Co., was used as the material for the adhesive joint. The CNTs were NANOSOL-R obtained from CNT Solution Co. Table 1 lists the characteristics of the materials used in the adhesive joints.

Fig. 1 shows the schematic of a single lap adhesive joint. If the aluminum is surface treated, the strength and failure mode of the adhesive joint are significantly affected. In this study, the surface of the aluminum was abraded using a 70 mesh sandpaper. The abraded aluminum surface was cleaned, and subsequently, dried using acetone. The release film was attached onto one side of the adhesive area to produce an artificial defect, as shown in Fig. 1. The CNTs in the adhesive were dispersed using five different types of dispersion methods. The static strength and defect detectability of the adhesive joint containing the CNTs were then evaluated. Table 2 lists the five different types of dispersion methods. The VC505-75 sonicator obtained from SONICS & Materials Co. and

80E three-roll-mill machine obtained from EXAKT Co. were used to disperse the CNTs into the adhesive, as shown in Fig. 2. Gaps 1 and 2 obtained in the three-roll-mill process can be controlled separately; the minimum adjustable gap is 1  $\mu\text{m}$ . The 1% CNTs were subsequently inserted into a resin and a hardener using an electronic scale.

In Method 1, the resin (YD-128) was sonicated in a beaker. The sonicated resin was then mixed with the hardener by hand.

In Method 2, the same sonication process, as that in Method 1, was repeated for the resin. The sonicated resin was mixed with the hardener using the three-roll-mill machine. The three-roll-mill process was performed three times. Gaps 1 and 2 obtained in the first milling process were 45  $\mu\text{m}$  and 30  $\mu\text{m}$ , respectively; those obtained in the second milling process were 30  $\mu\text{m}$  and 15  $\mu\text{m}$ , respectively; and the ones obtained in the third milling process were 15  $\mu\text{m}$  and 10  $\mu\text{m}$ , respectively.

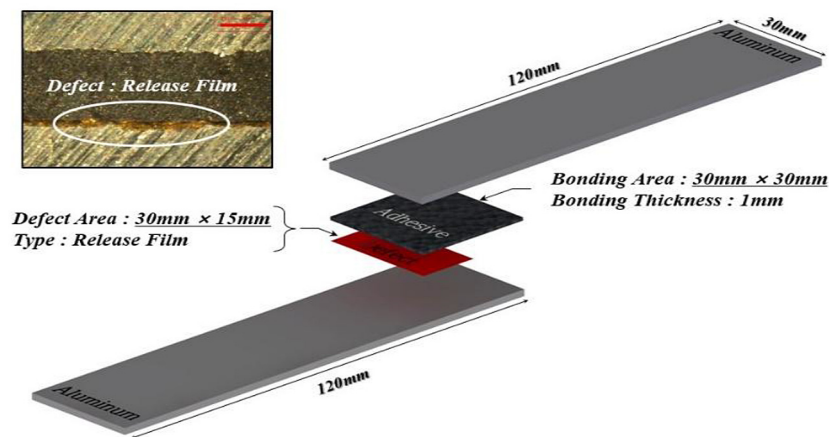
In Method 3, the sonication process used for the resin was the same as that used in Method 1. The sonicated resin was processed three times using the three-roll-mill machine, with Gaps 1 and 2 set the same as that in Method 2. The processed resin was mixed with the hardener four times using the three-roll-mill machine. Table 2 lists the gaps obtained using the three-roll-mill machine employed in Method 3.

In Method 4, the resin was not sonicated. The other processes were the same as those in Method 3.

In Method 5, the resin was diluted with dimethylformamide (DMF). The sonication process used for the resin is the same as that used in Method 1. The hardener was processed three times using the three-roll-mill machine. Gaps 1 and 2 were set the same as those in Method 2. The sonicated resin and milled hardener were mixed using the three-roll-mill machine, with Gaps 1 and 2 set the same as those in Method 2.

**Table 1**  
Materials of adhesive joint.

Item	Model & Manufacturer	Note
Aluminum	Al 5052	–
Adhesive	Resin: YD-128 Hardner: G-640 KUKDO CHEMICAL Co.	Mixing Ratio: YD-128/G-640 = 100/59 Curing condition: 80 °C for 2 h
CNT	NANOSOL-R CNTSOLUTION Co. Diameter: 10–15 nm	MWCNT Purity: 95% Length: 10–20 $\mu\text{m}$



**Fig. 1.** Schematic of adhesive single lap joint.

**Table 2**  
Dispersion methods.

No.	Sonication	Three-roll-mill [Gap 1, Gap 2], Unit: $\mu\text{m}$	Solvent
METHOD 1	Sonication (Resin + CNT)	–	–
METHOD 2	Sonication (Resin + CNT)	Step-1: Resin + Hardener [45,30 30,15 15,10]	–
METHOD 3	Sonication (Resin + CNT)	Step-1: Resin (Sonicated) [45,30 30,15 15,10] Step-2: Resin + Hardener [45,30 30,15 15,10 10,5]	–
METHOD 4	–	Step-1: Resin [45,30 30,15 15,10] Step-2: Resin + Hardener [45,30 30,15 15,10 10,5]	–
METHOD 5	Sonication (Resin + CNT)	Step-1: Hardener [45,30 30,15 15,10] Step-1: Resin (Sonicated) + Hardener [45,30 30,15 15,10]	DMF

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