



Using silane-functionalized graphene oxides for enhancing the interfacial bonding strength of carbon/epoxy composites



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ABSTRACT

Silane-functionalized graphene oxides (sGOs) were fabricated with four different self-assembled monolayers (SAMs) to reinforce an epoxy adhesive, with the aim of improving the bonding strength of carbon/epoxy composites. The oxygen-containing groups on the surface of graphene oxide (GO) were converted by the SAMs to amine, epoxy, or alkyl groups. The successful reaction between the silane molecules of the SAMs and functional groups of GO was evidenced by the results of different characterization methods such as Fourier transform infrared spectroscopy. It was found that the average thickness of the sGO flakes was higher than that of GO flakes. The bonding strength of a carbon fiber/epoxy composite, tested with a single lap joint bonded with an epoxy adhesive, was increased by 53% after the addition of a sGO that contained amine groups. These results show that sGOs, especially those containing amine functional groups, can strengthen the interfacial bonding between the carbon fibers and epoxy adhesive.

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1. Introduction

Ultralight, carbon-based, composite structural materials have been used extensively in recent years in the aerospace and automobile industries because of their excellent mechanical properties such as high specific strength, modulus, and damping capacity [1]. These carbon-based composites have been used to enhance the mechanical performance of conventional structures through the application of various techniques such as adhesive bonding [2–4]. Graphene (G) and graphene oxide (GO) have attracted a great deal of scientific interest because of their large surface area ($2630 \text{ m}^2 \text{ g}^{-1}$), high intrinsic mobility ($200,000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$), high Young's modulus ($\sim 1.0 \text{ TPa}$), high thermal conductivity ($\sim 5000 \text{ W m}^{-1} \text{ K}^{-1}$), and high optical transmittance ($\sim 97.7\%$). However, the properties of G- and GO-polymer composites are still lower than the theoretical values because of π - π stacking that induces molecular aggregation, as well as the weak interactions between G/GO and the polymer matrix [5–8]. Therefore, understanding the nature of the G/GO composite interface is crucial for

a wide range of applications using composites, with a comprehensive understanding of the effect of bonding constituents on the bonding performance being key to this understanding [9]. Among the different applications to mechanical structures, the bonding between composites and other materials, or between composite layers, has become an important issue because in many cases near-net-shape molding is not applicable [9]. Composite parts can be joined by two methods: mechanical fastening and bonding. For mechanical fastening, a number of holes are machined in the composite parts, which may cause some problems such as stress concentration and increasing the weight of the structure. The bonded joint is more efficient because the load distributed between the composite parts can be transferred more uniformly over a wide area and no holes are needed to join the parts. However, the critical drawback of this method is that the bonded composite parts are hard to repair [10,11]. Bonding can be classified as co-cure bonding or adhesive bonding depending on the bonding method used. According to previous studies [12–17], the bonding strength of composites and adhesives can be affected by several conditions, including the temperature, moisture, and pressure of the environment. Therefore, investigating bonded joints and the optimization of bonding methods is necessary to create composites with improved mechanical strength. Many technical methods for improving bonding characteristics of composites

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materials have been introduced and the representative methods for surface treatments are plasma surface treatments [18–20], heat treatment [21], acid treatment [22], excimer laser treatment [23], and mechanical abrasion (sandpaper, sandblast) [20]. Those conventional methods modify the bonding surfaces by controlling processing parameters such as plasma power, type of acid and sand particle size to investigate the effects of the surface modification on the bonding strength of carbon/epoxy composites. And those conventional methods were proved to be effective for enhancing static and fatigue bonding strengths.

GO contains many functional groups, including hydroxyl, carboxyl, carbonyl, and epoxide groups, which provide the reaction sites for covalent functionalization. In addition, the strong affinity of these functional groups toward polar aprotic solvents allows GO to be easily dispersed. Therefore, GO could be used in composites for bonded joints. Furthermore, the potential covalent functionalization of the GO surface could be used to improve the interfacial interactions between the GO and polymer matrix. It is thought that the physical absorption and chemical linking of functional molecules on the GO surface could enhance the compatibility between the GO and polymer matrix, which would improve the mechanical properties of composite materials.

In this study, coupling agents composed of GOs functionalized with self-assembled monolayers (SAMs) were used to bond joints in a composite system. The functionalization of GO was achieved by using four different SAMs: (3-aminopropyl)triethoxysilane (APTES), (3-aminopropyl)trimethoxysilane (APTMS), (3-glycidyloxypropyl)trimethoxysilane (GPTMS), and triethoxymethylsilane (MTES). The oxygen-containing groups on the surface of the GO were converted to other functional groups (amine, epoxy, or alkyl groups) depending on the SAM used. Adhesive-bonded single lap joint specimens made of carbon/epoxy composites and an epoxy adhesive were prepared to estimate the effects of GO and the silane-functionalized GOs (sGOs) on the bonding characteristics with tensile tests. Based on the experimental results, the effects of surface functionalization on the properties of GO and the bonding performance of the sGOs are discussed.

2. Experimental procedures

2.1. Synthesis of GO

GO was prepared using a modified Hummers method [24]. Briefly, 2 g of graphite powder (Alfa Aesar graphite powder, universal grade, 200 mesh, 99.9995%) was stirred with 2 g of NaNO_3 in 100 mL of concentrated H_2SO_4 for 1 day in an ice water bath. After stirring, 12 g of KMnO_4 was gradually added. Once it was thoroughly mixed, the ice bath was removed and the solution was stirred at 35 °C until a highly viscous liquid was obtained. After adding 200 mL of pure water followed by 200 mL of H_2O_2 , the mixture was centrifuged at 8000 rpm and washed with HCl and water. The centrifuging and washing processes were repeated a few times. Finally, the GO was dried at 50 °C for 24 h in a vacuum oven.

2.2. Synthesis of sGOs

GO nanosheets with many oxygen-containing functional groups, i.e., hydroxyl, carboxyl, and carbonyl groups, on their basal planes and edges were reacted with SAMs. To silanize the GO, 20 mg of GO was added to a three-neck flask containing 40 mL of deionized (DI) water and dispersed with a sonic bath for 60 min. Then, 5 mL of a 0.2 M toluene solution containing the SAM (APTMS, APTES, MTES, or GPTMS) was added. The SAMs were purchased from Sigma–Aldrich. The brown solution of the

homogeneously dispersed GO was stirred and heated at 70 °C for 24 h. After being heated, the color of the mixture had changed to dark brown, indicating the formation of the sGO. It was then washed with ethanol and DI water and dried at 50 °C for 24 h in a vacuum oven. An ultrasonicator (Sonicator Microtip Probes, SONICS VCX-750, SONICS & MATERIALS Inc., USA) was used to exfoliate the GO and sGO powders. The GO or sGO powder (10 mg) was then mixed with ethylene glycol (10 mL) in a 20 mL flask. The solution was sonicated (300 W) for 4 h, which were the optimized conditions for this experiment.

2.3. Preparation of specimens for tensile testing

To estimate the effect of GO and the sGOs on the bonding characteristics, adhesive-bonded single lap joint specimens were prepared according to international standards [25,26]. A unidirectional carbon/epoxy prepreg (USN125, SK Chemical, Korea) was used. The composite specimens were fabricated by stacking 16 prepregs with a 0° stacking angle and using a bonding area of 25 × 20 mm. The fabrication process was as follows. The composites were fabricated with a vacuum bag de-gassing molding process in an autoclave using the recommended curing cycle. The bonding surface was treated with an abrading paper (#120) to expose the carbon fibers, which would create an effective GO or sGO coating on the surface. The required amount of GO or sGO solutions were prepared with ethylene glycol (1.0 mg mL⁻¹) using an ultrasonic treatment. To remove contaminants from the bonding surfaces, they were cleaned with acetone, isopropyl alcohol, and DI water for 10 min per solvent with an ultrasonic cleaner (WUC-A03H, DAIHAN Scientific Inc., Korea). UV/ozone treatment was performed with a UV/ozone cleaner (AH-1700, Ahtech LTS, Korea) to remove the remaining impurities and introduce various oxygen-containing functional groups to the hydrophilic surfaces. As a result, the wettability between the carbon fibers and GO or sGO was improved. The thoroughly dispersed GO or sGO solution was applied to the bonding surfaces and spread with a precision applicator bar (Elcometer 3520, Elcometer, UK) to form the coating. In order to remove any residual solution, the coated bonding surfaces were dried at 90 °C for 3 min. The surfaces coated with GO or sGO were then bonded at room temperature with an epoxy adhesive (DP-460, 3M, USA) to fabricate the adhesive-bonded single lap joint. By using a special jig with a 0.5-mm-thick gap, a bonding thickness of 0.5 mm was achieved. The tabs at both ends of the specimen were bonded with the same epoxy adhesive to minimize any bending moments generated by the eccentricity of a line load.

2.4. Characterization of materials

Field emission scanning electron microscopy (FE-SEM, JEOL, JSM-5410LV, Japan) was used to take images of GO and sGO flakes. The FE-SEM samples were prepared by the ultrasonic treatment of GO and sGO dispersions (1.0 mg mL⁻¹) in ethylene glycol. The dispersions were then spin-coated onto SiO_2/Si substrates. Synchrotron radiation photoemission spectroscopy (SRPES) was performed at the Pohang Acceleration Laboratory in an ultrahigh vacuum chamber (base pressure of $\sim 10^{-10}$ Torr) in a 4D beamline that was equipped with an electron analyzer and a heating element. The onset of photoemission, which corresponded to the vacuum level at the surface of GO and sGO, was measured using a 250 eV incident photon beam with a negative bias on the sample. The results were corrected by using Au 4f as an internal reference to avoid charging effects. For Fourier transform infrared (FT-IR) spectroscopy, the synthesized materials were pressed into pellets with potassium bromide and scanned with radiation ranging from 400 to 4000 cm⁻¹. Thermogravimetric analysis (TGA, TGA-2050, TA

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