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Effect of microscale oil penetration on mechanical and chemical properties of carbon fiber-reinforced epoxy composites

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The effects of microscale oil penetration on mechanical and chemical properties of carbon fiber-reinforced polymer (CFRP) composites were investigated to address the adverse effects of lubricating oil on lightweight composites and improve the oil-proof performance of composites. The CFRP composites were confirmed to have lower mechanical properties after impregnation with oil at elevated temperature due to microscale oil penetration in the vicinity of carbon fibers on the surface of epoxy composite, and a high resin content and high heat curing conversion of epoxy polymer could reduce the oil absorption and tensile strength reduction of CFRP composite.

Introduction

Carbon fibers have been used in a wide range of applications due to their high strength, light weight, and low thermal expansion [1–3]. They are usually used in a polymer composite with thermosetting resin for mechanical reinforcement. The resulting carbon fiber-reinforced polymer (CFRP), consisting of carbon fibers embedded in a thermosetting matrix, has excellent mechanical properties and chemical stability, and therefore it can replace metals that suffer from corrosion or larger thermal expansion [4–6]. Additionally, because of the comparatively lower price than other fibers such as glass or polymer fibers, CFRP composite has attracted much attention in automobile, aerospace, civil engineering, and sport applications.

Rigid thermosetting resins are often used to provide the high mechanical strength and Young's modulus for reinforced plastic materials. Among them, bisphenol A type epoxy resin, which forms crosslinked network through chemical reaction of the epoxy rings, has excellent mechanical properties and adhesion [7–14]. Therefore, it is often used in CFRP composite to create a networked matrix structure to reduce the weight, meanwhile providing mechanical strength and hardness similar to or better than those of typical metals. However, CFRP composites containing epoxy resins also tend to be brittle. Hence, much effort has been devoted to solving this problem, in order to reduce the impact of external force or environmental factors on the mechanical properties and durability of the composite.

Recently, CFRP composites with superior properties have been studied for use as functional plastic materials in wide application areas to replace conventional metal materials [7–14]. For this purpose, it is important to study the adhesion between the carbon fibers and epoxy matrix resin, which strongly affects the mechanical properties of the composite [15,16]. Some approaches, such as using an internal filler or surface modification of the material, have been found to improve the adhesive interaction between carbon fibers and polymer matrix. However, in industries such as the automobiles or aerospace, there has been insufficient analysis on the effects of environmental factors in these composites. For example, CFRP composites can replace metals to reduce the weight and thermal expansion in the automotive industry [17–22]. However, unlike metals, CFRP composite can be affected by its contact with automotive oil, which is mostly organic compounds. Limited studies have suggested that oil can reduce the surfacial adhesion of epoxy resin with metal or carbon fiber [23,24], potentially reducing the mechanical properties and durability of the composite. Especially, study on the relationship between thermal curing behavior of epoxy resin and oil absorption of CFRP composites, as well as the associated changes in their

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mechanical and chemical properties has not been reported in the literature.

In the present study, we investigated the effects of microscale oil penetration on mechanical and chemical properties of carbon fiber-reinforced epoxy composites. CFRP composites prepared with different thermosetting conditions and resin contents were compared, and their thermal curing conversions were examined by Fourier-transform infrared spectroscopy (FT-IR). Considering the accelerated condition in automotive applications, the heat-cured CFRP composites were impregnated in high-temperature lubricating oil for 24 h, and weighed to measure the oil absorption. The microscale oil penetration can reduce the tensile strength of CFRP composites by weakening the adhesion between the carbon fiber and the epoxy resin, as confirmed in the scanning electron microscopy (SEM). The degree of oil absorption and tensile strength reduction of CFRP composite were investigated according to the epoxy resin content and heat curing conversion to improve the oil-proof performance of CFRP composites. The relationship between oil absorption and the resulting changes in the CFRP composite will provide important information for applying these materials to automotive and aerospace industries.

Experimental

Materials

Two CFRP prepregs (USN 200E and USN 200B) were obtained from SK Chemicals (Seongnam, Korea). The epoxy resin in them is K51, which contains the diglycidyl ether of bisphenol A and formulated hardener, was manufactured by the same company. The epoxy resin content of USN 200E and 200B was 25 wt.% and 33 wt.%, respectively, and characteristics of the used CFRP prepregs were summarized in Table 1. As the two prepregs have the same absolute amount of carbon fibers and different relative resin contents, USN 200E with lower epoxy resin content is thinner and lower in mass. The lubricant (SK ATF-SP3) used for oil impregnation test were purchased from SK lubricants (Seoul, Korea). The acetone used to dry the oil was purchased from Samchun Pure Chemical Co., Ltd. (Pyeongtaek, Korea).

Preparation of CFRP specimens

The tensile strengths of CFRP composites were tested based on the ASTM D3039 standard testing method for polymer matrix composites. The test specimen had overall length 250 mm, width 5 mm, and thickness 0.169 or 0.199 mm. The prepared specimens were thermally cured according to soft curing conditions in a convection oven (Jeiotech, OV-11E) at 125 °C for 90 min. To obtain high exothermic reaction conversion of the epoxy resin, specimens were also cured at the same temperature for a longer period (120 min), which will be called the hard curing condition.

Oil absorption test of CFRP composite

The thermally cured CFRP specimens were immersed in a steel pot containing 1000 ml of lubricating oil. To simulate the high-temperature environment in automotive applications, the steel pot was heated in a convection oven at 150 °C for up to 24 h. Afterwards, the oil on the outside of the specimen was removed by a dry method. The oil-impregnated specimen was placed between tissue papers wetted with acetone, and pressed under a book weighing 1 kg for 3 h. This step was repeated twice.

Test of CFRP contamination in oil

Bulk-sized CFRP specimens were also placed in a small amount of oil to investigate possible cross-contamination of the lubricating oil by the CFRP composite. A test specimen (30 mm overall length, 20 mm width, and 0.3 mm thickness) was prepared under the hard curing condition at 125 °C for 120 min, and then immersed in a 50-ml beaker with 5 ml of oil. The beaker was heated in a convection oven at 150 °C for 24 h.

Characterization

The amount of oil absorbed by the CFRP composites was measured by the mass difference of the thermally cured specimen before and after oil impregnation using an electronic scale (Mettler Toledo, ME204). The FT-IR spectra (Jasco FT/IR-406 plus) were used to analyze the reaction conversion of epoxy resin and contamination in the lubricating oil. Tensile strength of the CFRP composites was measured by a universal testing machine (Shimadzu, AGS-X), with the specimens evenly clamped using hydraulic type jigs. A field emission SEM (FE-SEM, SU-70, Hitachi) instrument was used to examine the surface and cross section of the CFRP specimens prepared under different thermal curing conditions. The thermal expansion coefficients of the CFRP materials were measured by a thermomechanical analysis (TMA, TA instrument Q400).

Results and discussion

Heat curing conversion of epoxy resin in CFRP composites

The FT-IR spectra for the CFRP composites under different heat curing conditions are shown in Fig. 1(a) and (b) for USN 200E and 200B (25 wt.% and 33 wt.% epoxy resin content), respectively. For the unreacted carbon fibers, the spectra were normalized to the C–H stretching vibration at 1235 cm^{-1} [25,26]. In general, epoxy resin in the composite undergoes ring-opening reaction of epoxide at high temperatures. This reaction cross-links the polymer molecules with each other to enhance the mechanical properties of the CFRP composite. This polymer network may also form a uniform surface of CFRP covered with epoxy resin, reducing the oil absorption tendency of carbon fibers due to the hydrophobic nature of carbon fiber. Similar to the literature values, pristine CFRP composites showed an epoxide peak at 910 cm^{-1} [27,28]. The area under this peak was used to calculate the heat curing conversion using the following equation:

$$\textit{Heat curing conversion}\ (\%) = \frac{A_{uncured} - A_{cured}}{A_{uncured}} \times 100 \quad (1)$$

where $A_{uncured}$ and A_{cured} are the peak areas of uncured and cured CFRPs, respectively. Under the soft curing condition at 125 °C for 90 min, the USN 200E and USN 200B composites showed similar heat curing conversions (76.6% and 75.3%, respectively), as shown in Fig. 1(c). These low values indicate that both the uncured epoxy

Table 1
Characteristics of the CFRP prepregs.

Product	Resin	Carbon fiber content (wt.%)	Epoxy resin content (wt.%)	Thickness (mm)	Total areal density (g/m^2)
USN 200E	K51	75	25	0.169	267
USN 200B	K51	67	33	0.199	299