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Thermo-gravimetric analysis method to determine the fiber volume fraction for PAN-based CFRP considering oxidation of carbon fiber and matrix

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ABSTRACT

The properties of fiber reinforced composites are mainly determined by the fraction of reinforcement and matrix. Thus, to design a system based on composite materials, it is vital to carefully measure the volume fractions of the composites with a proper method. Digestion by strong acid or ignition at high temperature in an oxidizing environment are conventional for measuring content fractions. In essence, these methods assume that the reinforcement does not lose weight by digestion or ignition. However, by neglecting reported vulnerable oxidization characteristics of carbon fiber, these conventional methods result in inaccurate fiber volume fractions of carbon fiber/epoxy composites. In this study, an effective and accurate method, having only 2 steps in measuring process and 1.5%p maximum error, for determining the fiber volume fractions of two different PAN (Polyacylonitrile)-based carbon fiber reinforced composites via thermo-gravimetric analysis was developed and subsequently verified using the results from 80 microscopic images.

1. Introduction

The fiber volume fraction is fundamental and important for designing composites. Despite the superior performance and expanding applications of composite materials [1–3], it is almost impossible to design composites without the basic information of the fiber volume fraction. The fiber volume fraction can affect most of the mechanical and thermal properties of composites. Currently, there are three conventional methods for measuring the carbon fiber volume fraction, that is, an image processing method using microscopic cross-section images, a digestion method with strong acid, and an ignition method at high temperatures. The image processing method is only accurate for the cross-section of unidirectional carbon fiber composites. Due to the non-uniform brightness of the cross-section of angled carbon fibers, the accuracy is reduced for woven and multi-directional carbon fiber composites. Digestion and ignition methods are good options for determining the contents of composites based on ceramic fibers, which are not reactive with oxygen, such as glass fiber/epoxy composites. However, these two oxidizing methods are not appropriate for determining the content of carbon fiber/epoxy composites. This is because many researchers have reported that general carbon fibers become oxidized, thus causing a weight loss that cannot be ignored. It has been reported that various types of carbon fibers, such as the T300 carbon fiber, exhibited weight losses of approximately 40% after half an hour at 600 °C [4]. Although the rate of burning of carbon fibers in composites is very continuous and stable and is also lower than that of epoxy resin, the amount of burning mass of carbon fiber during measurement cannot be neglected [4–9]. Therefore, the accuracies of most of the determined fiber volume fractions of carbon fiber/epoxy composites should be taken with caution. To increase the accuracy by considering the weight loss of carbon fibers, some methods using thermo-gravimetric analysis (TGA) have been proposed. Cho-Rok Moon et al. first conducted an experiment with TGA at high temperatures to measure the volume fraction of composites reinforced by carbon fiber [10]. Then, they conducted pyrolysis by TGA using a certain weight of carbon fiber without the resin matrix and compensated for the additional mass loss of carbon fiber. Yee and Stephens minimized the additional mass loss of carbon fiber in a

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nitrogen gas atmosphere at high temperatures and deducted the mass of the non-oxidized resin matrix by conducting TGA with glass fiber composites containing the same matrix resin [11]. Wang also conducted pyrolysis in a nitrogen environment along with a reference resin at the same time to compensate for the rate of carbonization of epoxy to determine the volume fraction of carbon fiber [12]. These methods are effective and also accurate for measuring the fiber volume fraction in carbon fiber/epoxy composites. However, these methods are disadvantageous because additional independent experiments using carbon fiber or matrix resin are required to compensate for the excessive or remaining weight of the matrix. In the case of commercial prepregs, the combination of the resin ingredient and treatment of carbon fiber are unique and classified because they are proprietary. Therefore, it is impossible to obtain information on the exact epoxy system or treated carbon fiber of commercial composites. Information on the fiber volume fraction is too important and fundamental to be ignored for designing, verifying, and even reverse engineering composite materials.

In this research, a new efficient and accurate TGA method for experimentally determining the carbon fiber volume fraction by considering the carbon fiber oxidation characteristics was developed without conducting any separate experiments using carbon fiber or epoxy. The method included the oxidation characteristics of composites and carbon fibers. The proposed method was verified using two different composites reinforced by PAN-based carbon fibers, TR50s and M55J, by both fiber counting and image processing methods of 80 cross-section microscopic images of uni-directional composite specimens.

2. Experimental

2.1. Sample preparation

The two different types of composite laminates were manufactured in an autoclave. One of the prepregs utilized was USN 125 B (SK chemical). The reinforcement was TR50S pyrofil, which has a middle range stiffness of 250 GPa and a high strength of 4 GPa. The other prepreg utilized was M55J/M18 (Hexcel). The M55J fiber has a very high stiffness of approximately 550 GPa, but a low strength of 2 GPa. Both fibers in the two types of composite laminates were part of a series of PAN-based carbon fibers. For the curing process, 16 layers of prepregs were stacked at the same zero degree for microscopic image processing. For applying the method to quasi-isotropic specimen, conventional 16 layers stacking sequence, $[0/\pm45/90]_{2s}$ was adopted. The curing process was conducted by an autoclave at Korea Advanced Institute of Science and Technology. The curing conditions were 180 °C and 120 °C at 4 bar for the M55J/M18 and USN125B prepregs, respectively.

Fig. 1 shows the procedure of the sample preparation. After curing, the plate was cut into a square with a side length of 20–25 mm. The densities of the specimens were measured following ASTM D792, and the average values from five different measurements were determined [13]. The measured specimen was cut into small square shapes, that is, 1 mm by 1 mm and 2 mm by 5 mm, for TGA analysis and microscopic image processing. The small specimens for image processing were mounted by epoxy, and the surfaces were treated with a polishing machine, MetPrep3/pH3.

2.2. Image processing and fiber counting methods

The cross-section of the prepared specimens was imaged by an AxioImager A2m™ Upright Microscope. The magnification was 200 times, and the raw image contained 2048 pixels in the row and column lines. Five sets of images were obtained in the thickness direction, and the four images were obtained from top to bottom. To count and select only the pixels standing for fibers or epoxy, free software, Image J, was used. In the Image J software, the areas of reinforcement and matrix were distinguished by an automated processing method. In the conventional area method, the appropriate threshold level should be specified; otherwise,

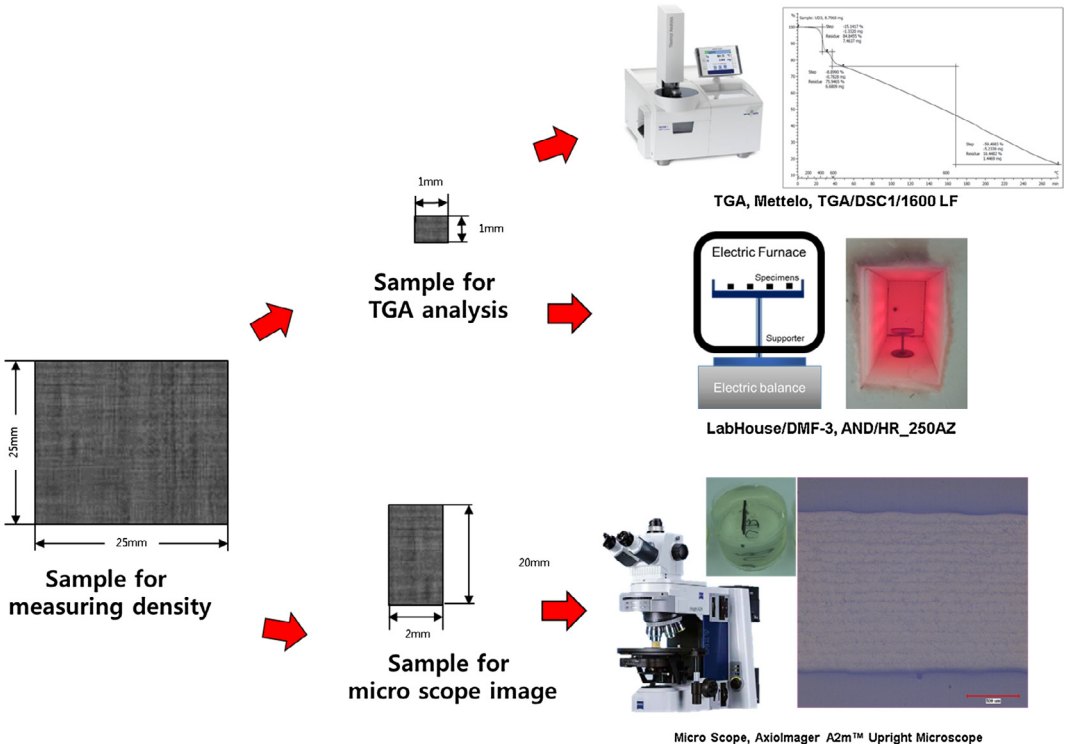

Fig. 1. Schematic of the sample preparation for the measurement method to determine the fiber volume fraction. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)