



# Development of a carbonization-in-nitrogen method for measuring the fiber content of carbon fiber reinforced thermoset composites



Qiushi Wang<sup>a</sup>, Haibin Ning<sup>a,\*</sup>, Uday Vaidya<sup>a</sup>, Selvum Pillay<sup>a</sup>, Leigh-Ann Nolen<sup>b</sup>

<sup>a</sup>Department of Materials Science and Engineering, Materials Processing and Applications Development (MPAD) Center, University of Alabama at Birmingham, Birmingham, AL 35294, United States

<sup>b</sup>Toray Carbon Fibers America, Decatur, AL 35601, United States

## ARTICLE INFO

### Article history:

Received 1 October 2014

Received in revised form 11 February 2015

Accepted 28 February 2015

Available online 7 March 2015

### Keywords:

A. Carbon fiber  
E. Thermosetting resin  
D. Thermal Analysis  
E. Lay up

## ABSTRACT

Carbon fiber reinforced thermoset composites such as carbon fiber epoxy composites are widely used in aircraft and aerospace, and are being increasingly used in automotive applications because of their light-weight characteristics, high specific strength, and stiffness. The carbon fiber content in the composite plays a critical role in enhancing structural performance. The carbon fibers contribute to the strength and stiffness; therefore, the mechanical properties of the composite are greatly influenced by the carbon fiber content. Measurement of carbon fiber content is essential for product quality control and process optimization. In this work, a novel carbonization-in-nitrogen (CIN) method is developed to characterize the fiber content in carbon fiber thermoset composites. A carbon fiber composite sample is carbonized in a nitrogen environment at elevated temperatures, alongside a neat resin sample. The carbon fibers are protected from oxidation while the resin (the neat resin and the resin matrix in the composite sample) is carbonized under nitrogen environment. The neat resin sample is used to calibrate the resin carbonization rate and calculate the amount of the resin matrix in the composite sample. The new method has been validated on several thermoset resin systems, and found to yield accurate estimation of fiber content in carbon fiber thermoset composites.

Published by Elsevier Ltd.

## 1. Introduction

Carbon fiber reinforced thermoset composites have been widely used in many high-performance applications because of their widely described advantages, especially high specific strength, stiffness and stiffness-to-weight ratios, and many other advantages [1–4]. It is known that the carbon fiber content, as one of the most important parameters, determines the mechanical and other properties of carbon fiber composites. Smith et al., Chen et al. and Simon et al. have studied the effect of fiber content on resulting properties of composite materials [5–7]. Carbon fibers act as the primary reinforcement to provide strength and stiffness for fiber reinforced composites.

Traditionally, standard methods established by ASTM D3171-Standard Test Methods for Constituent Content of Composite Materials are applied for determining fiber content in carbon fiber composites [8]. These methods require that the resin matrix be completely removed from a carbon fiber composite either by acid digestion or burn-off in air and only the carbon fibers remain in

the residue. Despite widespread use, the acid digestion and burn-off-in-air methods are not without limitations. The acid digestion method has been restricted to certain resin systems, such as polypropylene and epoxy, which could be dissolved completely in acids. Highly hazardous acids such as hydrochloric and sulfuric acids used in the acid digestion method could pose potential hazards to the operator, as well as the environment. In addition to the excessive time required for acid digestion, there are several semi-crystalline resins (e.g. polyimide) that cannot be dissolved in solvents (e.g. hydrochloric acids) [9]. The burn-off-in-air method is only applicable for resins that are easy to degrade at elevated temperatures. High temperature resins, such as epoxy (decomposition temperature >400 °C) typically used in aircraft and automobile applications, will only totally degrade in air at temperatures that will also cause the carbon fibers to oxidize [10]. In addition, airborne carbon particles from matrix burn-off in air pose potential hazards to the operator and the environment.

Another method used for measuring carbon fiber content is Thermogravimetric Analysis (TGA), as reported by Polis [11]. In this method, a sample weighing less than 0.1 g is used. This small sample size is not sufficiently representative of carbon fiber composite structures. The TGA method has not been widely adopted because

\* Corresponding author. Tel.: +1 205 996 7390; fax: +1 205 934 8485.

E-mail address: [ning@uab.edu](mailto:ning@uab.edu) (H. Ning).

of low confidence level which is derived from using small sample size (0.1 g) to represent large composite structures.

Optical microscopy is another method which has been reported by Purslow et al. and Cohen et al. [12,13]. The fiber content is measured through optical image analysis, with the image obtained from a polished composite sample. The accuracy of this method is highly dependent on sample preparation, number of cross-sections being examined, and image filtering. In addition, a sufficient number of cross-sectional surfaces should be examined in order to obtain a fiber content representative of the whole sample. This may also lead to misrepresentation of studying three-dimensional features (fiber volume/weight content) using two-dimensional characterization method.

In this paper, a generalized method is developed to measure the carbon fiber content in carbon fiber reinforced thermoset composites.

**2. Experiment**

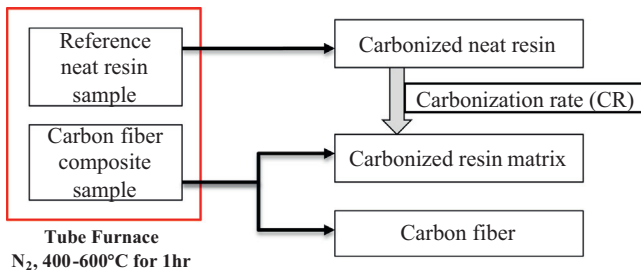
*2.1. Procedure description*

A carbonization-in-nitrogen (CIN) method is developed to characterize carbon fiber reinforced thermoset composites. A carbon fiber reinforced thermoset composite is carbonized in a nitrogen-purging tube furnace along with a *reference neat* (unreinforced) resin. The *reference neat resin* is the same as that used in the carbon fiber reinforced composite. However, it may be difficult to procure the neat resin sample in some cases, which could be the drawback of this method. During carbonization in nitrogen, the resin matrix in the carbon fiber composite and the *reference neat resin* are carbonized under high temperature (400–600 °C) and inert atmosphere. The inert atmosphere is required to avoid oxidation of the carbon fibers. The products of the carbonization of the neat resin and the resin from the composite include both gaseous fumes that are vented and carbonized residue that remains in the furnace. The carbonized residue from the *reference neat resin* provides the percentage of carbonization of the resin. This residue information can be used to calculate the amount of resin matrix in the composite. Fig. 1 illustrates the procedure. Carbonization rate (CR) of the neat resin can be estimated from the neat resin sample (*m*) and its residue ( $m_{cr} - m_c$ ) in accordance with Eq. (1):

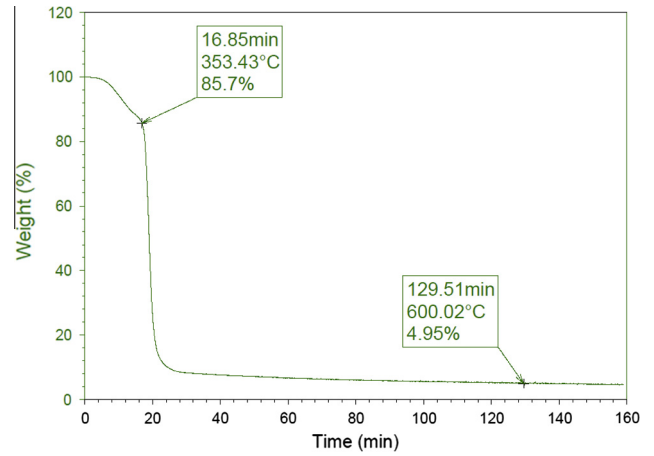
$$CR = (m_{cr} - m_c) / m \tag{1}$$

where CR – carbonization rate (a value between 0 and 1) of the reference neat resin,  $m_{cr}$  – residue mass from the reference neat resin sample in its crucible,  $m_c$  – the crucible mass and *m* – reference neat resin sample mass.

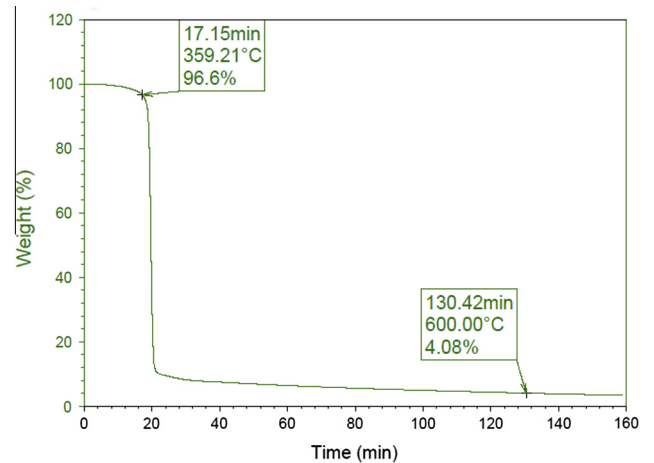
The carbon fiber content (weight fraction),  $W_f$ , in the composite sample could be calculated in accordance with Eq. (2) that is developed for the first time:



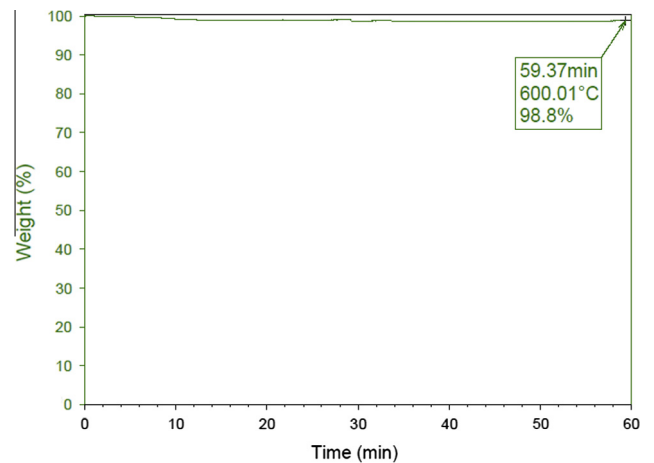
**Fig. 1.** The carbonization-in-nitrogen flow chart. Note that the carbonization rate is used for calculating the amount of resin matrix in the composite sample. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 2.** TGA plot of the mass fraction loss of epoxy resin at 600 °C. Note that the resin still has residue after 60 min in nitrogen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 3.** TGA plot of the mass fraction loss of vinyl ester resin at 600 °C. Note that the resin still has residue after 60 min in nitrogen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 4.** TGA plot of the mass fraction loss of carbon fiber at 600 °C in nitrogen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Download English Version:

<https://daneshyari.com/en/article/7891797>

Download Persian Version:

<https://daneshyari.com/article/7891797>

[Daneshyari.com](https://daneshyari.com)