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Rapid and nondestructive analysis of quality of prepreg cloth by near-infrared spectroscopy

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Abstract

A near-infrared spectroscopy analysis technique has been developed to measure the resin content, the volatile content and the pre-curing degree of resin during the manufacture of prepreg cloth by solution impregnation process. A principal components regression (PCR) algorithm was used to model the relation between the components content and the spectra. The calibration results had correlation coefficient of 0.9440 for the resin content (root mean square error of calibration, RMSEC = 0.490), of 0.9417 for the volatile content (RMSEC = 0.249) and of 0.9401 for the pre-curing degree (RMSEC = 0.186), respectively. The method was applied in the product line to analyze the quality of prepreg cloth directly, and the measurement process was accomplished in less than 2 min without sample destruction. The results showed that the maximal error were −0.70%, −0.546% and 0.378% for the resin content, the volatile content and the pre-curing degree, respectively. The study indicates that NIR spectroscopic method is a good alternative for the quality analysis of prepreg cloth.

Keywords: D. Nondestructive testing; Near-infrared spectroscopy; A. Phenolic resin; Prepreg

1. Introduction

Prepreg is the important intermediate of polymeric composites, and its quality will greatly influence the properties of final composite products. The resin content, the volatile content and the pre-curing degree, i.e., three factors, are important to ensure the quality of the prepreg. Traditional analysis methods such as solvent extraction, burn-off and acid digestion are often used to measure the three factors. However, these methods suffer some disadvantages such as the sample destruction, the high cost of disposal of chemical wastes and the excessive time to perform these tests.

Recently, a series of investigations on the analysis of the quality of the prepreg have been carried out in succession, in which the methods utilized include the γ-ray reflectance [1], ultrasonics [2,3], photometry [4,5], β-ray transmission [6,7] and infrared spectroscopy (IR) [8] techniques. Although these methods overcome most shortcomings accompanied with the traditional analysis methods, they cannot monitor the three factors at the same time. Apparently, it was desirable to devise a method which would be quick and nondestructive, moreover, which can measure the three factors of the prepreg.

Near-infrared (NIR) spectroscopy is an excellent alternative analytical method. Although NIR spectra are difficult to interpret visually, since they lack the sharp absorbance peaks of traditional IR spectra, sophisticated calibration algorithms can be used to determine which wavelengths or combination of wavelengths correlate to changes in sample properties. One

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important feature of NIR is that it can be performed on the raw product without destroying the sample. Secondly, the sample time can be reduced to only a few minutes per sample. The wide applicability and potential of NIR spectroscopy for polymer analyses have been recognized [9–15]. NIR spectra contain information related to polymer properties [16] such as composition, conformation and crystallinity, therefore NIR can be widely applied for various polymer analyses in conjunction with chemometric calibration methods.

The aim of this work was to develop a NIR technique to analyze the quality of the prepreg cloth. The calibration models about the resin content, the volatile content and the pre-curing degree were established respectively by using principal components regression (PCR), and the measurement accuracy of the NIR method was ascertained directly from analysis of the production line of the prepreg cloth.

2. Experimental

2.1. Materials

Phenolic resin (Barium hydroxide as the accelerating agent) was provided by Beijing Research Institute of Material and Technology, China. Resurrection glass cloth (BWT-260) was obtained from Shanxi Huate glass fibre Ltd., China. Ethanol was purchased from Shenyang Chemical Reagent Factory, China.

2.2. Samples and NIR spectra collection

The solution impregnation method was used to manufacture the prepreg cloth. The impregnation procedure is shown in Fig. 1. The prepreg cloth was produced at the speed of 90 m/h, and the width of the cloth was 80 cm. A FT-NIR Systems model MATRIX-E spectrometer (Bruker Co., Germany) was assembled between the take up mechanism and the dry tower as shown in Fig. 1. The spectrometer can perform noncontact analysis of the product. In the test, the light from the sources was focused on to the moving prepreg cloth, and then the diffuse reflectance spectra from the prepreg cloth were recorded by the spectrometer. A gilded metal plate was placed under the prepreg cloth in order to enhance the diffuse reflectance effect. When the spectrometer completed a collection of spectrum, the corresponding piece was cut from the prepreg cloth exactly and was regarded as one sample. The size of each sample was 16×8 cm. NIR spectra were collected over the 12,000–4000 cm^{-1} with a resolution of 16 cm^{-1}, each sample spectrum was obtained by averaging 4 scans. All the 93 samples and their NIR spectra were collected in two months.

2.3. Chemical analyses

The resin content, the volatile content and the pre-curing degree of the prepreg cloth samples were measured according to China Standard GB7192, GB6056 and GB2576. The test specimen was divided into two equal parts A and B, and weighed separately to the nearest 0.0001 g to obtain the initial weight G_A and G_B. Part A was placed in the oven at 160 °C for 10 min, cooled in a desiccator, and immediately weighed to obtain the weight G_{A1}. Part B was dissolved in acetone for 10 min, placed in the oven at 160 °C for 10 min and weighed to obtain the weight G_{B1}, then was placed in muffle furnace at 600 °C for 10 min and weighed to obtain the weight G_{B2}. The volatile content ($V\%$), the resin content ($R\%$) and the pre-curing degree ($C\%$) were calculated as follow:

$$V\% = (G_A - G_{A1})/G_A \times 100, \tag{1}$$

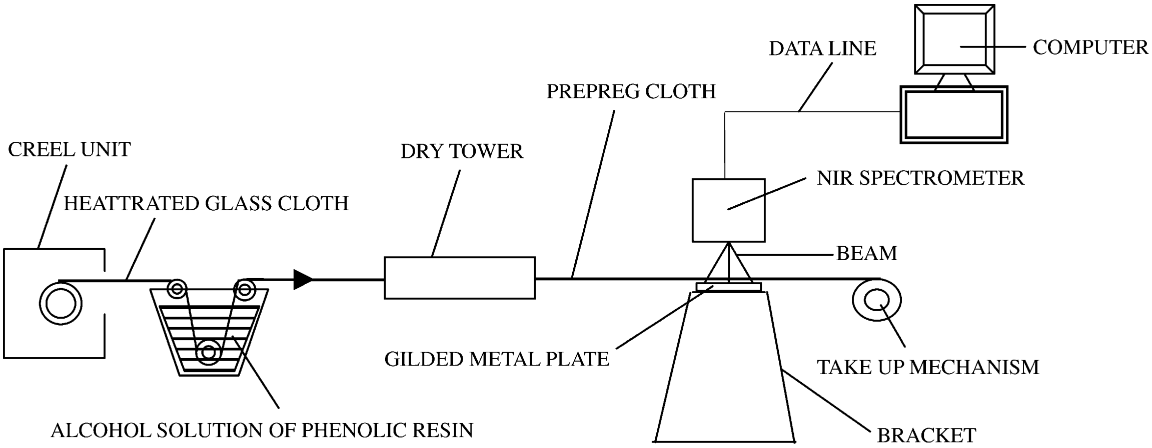

Fig. 1. Schematic of manufacture process of prepreg cloth.