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Test Method

In-process acquisition of cure-dependent viscoelastic properties of carbon fiber reinforced composites using micromechanics-based guided wave analysis

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ABSTRACT

An ultrasonic guided wave-based method for cure monitoring and estimation of anisotropic viscoelastic properties of carbon fiber reinforced plastics is developed. A guided wave propagating in the transverse direction of a unidirectional carbon fiber reinforced plastic in vacuum bag molding is measured, and its energy velocity and attenuation are obtained after gelation. The transfer matrix method and micromechanical modeling are used to calculate the energy velocity and attenuation in a stratified plate as a function of the complex modulus of the resin. The development of the complex modulus during cure is estimated by comparison of the experimental data with the numerical analysis, and the storage modulus and loss modulus are expressed as functions of the degree of cure using differential scanning calorimetry. The method developed in this study enables non-invasive in-process measurements of the cure state and viscoelastic properties of the carbon fiber reinforced plastic during the cure cycle.

1. Introduction

The development of residual stresses is a significant problem in the manufacture of thermoset carbon fiber reinforced plastics (CFRPs). Distortion of the shape of CFRP components results when the residual stresses are released after demolding [1–4]. The assembly of parts with a dimensional misalignment may cause additional stresses, and can lead to premature failure of the components. Moreover, a process-induced residual stress can be sufficiently high to cause matrix cracking even before external loading [1,2].

It has been reported that judicious selection of process parameters greatly contributes to the reduction of residual stresses and the resulting shape distortion [2,5]. Optimum design of temperature distribution based on multiple heaters has also been studied to minimize warpage of CFRP [6]. To select the optimum process parameters to minimize the effects of residual stresses, a process simulation to predict residual stresses are required. Process simulation of thermoset CFRP requires the elastic modulus, cure shrinkage strain and coefficient of thermal expansion as input material properties for the calculation [1–3,7,8]. Since the development of a residual stress is dominant after gelation, the measurements of these properties after the gel point are essential [9,10].

The cure shrinkage strain and coefficient of thermal expansion can be measured using an embedded optical fiber sensor during the cure cycle. It has been reported that a fiber Bragg grating (FBG) sensor embedded in a CFRP can measure the cure shrinkage strain during an isothermal cure process, and the coefficient of thermal expansion can be obtained from the strain change during the cooling stage [11,12]. The elastic modulus during cure is generally measured by a dynamic mechanical analyzer (DMA) or rheometer [5,13–15]. For a cure simulation, the elastic modulus needs to be expressed as a function of the degree of cure (DOC). Testing with a DMA or rheometer is performed for partially cured samples with different DOC values or during curing in the equipment. The development of the DOC is obtained using differential scanning calorimetry (DSC) adopting the same temperature profile as the curing process. However, the development of the elastic modulus measured by a DMA or rheometer may differ from that in the actual curing process because these measurement methods are performed in conditions that do not match the real processes of CFRP. Hence, a method that can measure the development of the elastic modulus during an actual cure cycle is required.

Ultrasonic measurements have been studied for monitoring cure reaction of thermoset resins and for measurement of the evolution of their elastic modulus. Maffezzoli et al. [16], Rath et al. [17], Lionetto et al. [18] and Ghodhbani et al. [19] performed measurements of the ultrasonic sound velocity and attenuation using a pulse-echo or

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through-transmission mode setup during an isothermal process. In these studies, the measured parameters were the sound velocity and attenuation of the longitudinal wave propagating in the through-thickness direction of the tested plate. This setup offered analytical solutions for one-dimensional wave equation, and the relationship between these waveform parameters and the dynamic modulus of the resin could be determined. An ultrasonic guided wave has also been used for cure monitoring. Guided wave is a wave that propagates along the waveguide interacting with boundaries. Vogt et al. [20] used an ultrasonic guided wave propagating in a wire inserted in a curing resin. Although the evolution of cure could be qualitatively measured using this method, vacuum bagging or closure of the mold in composite production causes detrimental effects on the results of this measurement. Pavlopoulou et al. [21] performed measurements on an ultrasonic guided wave propagating in a CFRP at different cure levels. A pitch-catch mode with two piezoelectric transducers attached to the sample surface was adopted for their experiments. They studied feature extraction from the response signal to capture different cure levels. Hudson et al. [22] developed an in-process cure monitoring system based on a guided wave measurement for cure monitoring of a CFRP laminate. They used piezoelectric transducers mounted on the CFRP laminate in a vacuum bag to measure the time of arrival (TOA) and amplitude of the guided wave propagating in the transverse direction of the CFRP. They showed that sharp changes in the TOA and amplitude could be obtained after gelation. Although viscoelastic properties of resins can be easily obtained by the measurement of a one-dimensional longitudinal wave, the guided wave-based method to estimate the change in viscoelastic properties during cure has not been reported to the best of the authors' knowledge. Ultrasonic methods can facilitate a non-invasive measurement of the cure process of composite materials. Since the guided wave technique measures a wave propagating between a transmitter and receiver mounted on the single side, it has the potential to measure the cure state at a location which is not accessible for a point-by-point through-thickness measurement method like the through-transmission technique. Moreover, this approach can enable the monitoring of the cure state distribution with a smaller number of piezoelectric sensors.

To achieve more efficient cure monitoring and viscoelastic modeling for the process simulation, the present study develops an ultrasonic guided wave-based method for cure state monitoring and the estimation of the development of viscoelastic properties. In this work, an ultrasonic guided wave propagating in a CFRP was measured during an isothermal cure process using two piezoelectric transducers mounted on the CFRP laminate as the transmitter and receiver. Semi-analytical modeling to predict the energy velocity and attenuation of the guided wave from the complex modulus of the matrix resin was developed using a micromechanics model for the composite. In this calculation, the waveguide was assumed to be a stratified plate composed of CFRP, mold plate, release film and vacuum bag. The transfer matrix method was used to calculate the dispersion characteristics of the energy velocity and attenuation of the wave in the stratified media. The development of the complex modulus of the matrix resin was estimated from the comparison of the experimental data with the calculation.

2. Experimental

2.1. Material and method

A guided wave propagating in a unidirectional CFRP was measured to obtain the changes in the energy velocity and attenuation during cure. The tested material was an uncured 12-layer unidirectional carbon fiber/epoxy laminate (T700SC/2592 prepreg, Toray Industries, Inc.). Fig. 1 shows the experimental setup used for the ultrasonic guided wave measurements. The size of the CFRP prepreg was 200 mm × 200 mm. 50 μm-thick PTFE release films were mounted on the top and bottom surfaces of the CFRP. The CFRP sandwiched by the

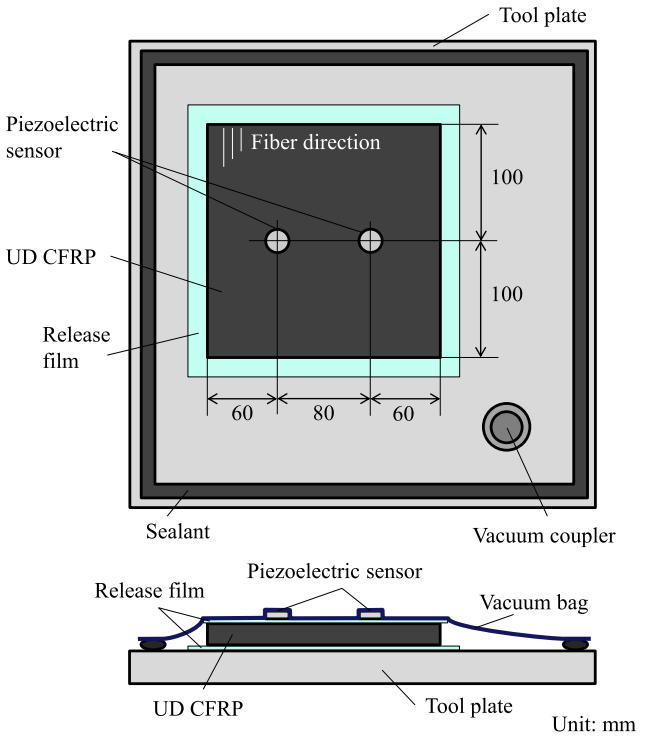

Fig. 1. Experimental setup for ultrasonic guided wave measurement during a cure cycle.

release films was then placed on a 1.93 mm-thick stainless plate. Two circular piezoelectric sensors (C-64, Fuji Ceramics Corporation) were placed on the release film on the top surface of the CFRP. The piezoelectric sensors were heat-resistant sensors with a Curie temperature of 345 °C. The diameter and thickness of the sensor were 20 mm and 0.4 mm, respectively. These two sensors were used as a transmitter and receiver, and were placed such that the guided wave propagating in the transverse direction of the unidirectional CFRP was measured. The distance between the center of the two sensors was 80 mm. The CFRP and sensors were covered by a 50 μm-thick vacuum bag. The CFRP was cured under the vacuum pressure. This pressure allowed the sensors to achieve intimate contact with the tested material.

The transmitter was excited by a five-cycle Hanning windowed 120 kHz sinusoidal waveform with a peak-to-peak voltage of approximately 10 V supplied by a function generator (AFG 3021C, Tektronix, Inc.). Measurement of the voltage of the receiver was triggered by the excitation signal. The receiver voltage was filtered and amplified by a weak signal preamplifier (5307, NF Corporation), and measured by an oscilloscope (Picoscope 5442B, Pico Technology). The preamplifier amplified the signal by 50 times. Acquisition of waveforms was performed at 20 s intervals. The CFRP was cured in an oven following the temperature profile shown in Fig. 2. The temperature ramp was 2 °C/min in the beginning of the cure cycle, then the temperature was held at 90 °C for 5 h. After the completion of the isothermal process, the tested material was cooled down from 90 °C to room temperature. Evolution of the DOC of the prepreg produced by this temperature profile was measured by DSC (DSC-60 Plus, Shimazu Corporation). The DOC at an oven time t was calculated from the ratio of the heat of reaction for partial cure until time t to that for full cure. The heat of reaction for partial cure was obtained by measuring the residual heat of reaction of the partially cured sample. Measurement of the residual heat of reaction was performed for samples with different cure levels to obtain the DOC over the cure cycle. The evolution of the DOC during the cure cycle is plotted in Fig. 2, and a sigmoidal curve is fitted to the data. The DOC increases with time and approaches a value of approximately 0.9. The evolution of DOC is almost completed at an oven time of approximately