Composites Science and Technology 69 (2009) 2008–2014

Contents lists available at ScienceDirect

Composites Science and Technology

journal homepage: www.elsevier.com/locate/compscitech

Impedance spectroscopy for progressive damage analysis in woven composites

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ARTICLE INFO

Article history:
Received 10 February 2009
Received in revised form 30 April 2009
Accepted 13 May 2009
Available online 20 May 2009

Keywords:
A. Flexible composites
A. Polymer matrix composites
B. Electrical properties
B. Fatigue
D. Impedance spectroscopy

ABSTRACT

Current applications require an understanding of the relationships that exist between the functional characteristics of composite material systems and the long-term behavior of those materials under mechanical loading. Using electrochemical impedance spectroscopy, a method was established and used to characterize damage initiation and progression in thin woven glass/epoxy composites. Monitoring the evolution in residual strength due to out of plane bending fatigue, a relationship was established between impedance at different frequencies of excitation and the development of damage in the material. When compared to common methods of measuring change in physical properties or visual damage, the metrics defined by impedance spectroscopy measurements were found to show larger, more consistent, and clearer distinctions as damage developed.

1. Introduction

Engineered material as thin layers or sheets are pervasive and have become an important class of materials for modern technology in general, and for flight related devices in particular. The fundamental nature of these materials is often defined by the device for which they are designed and engineered, and by the manufacturing process that must be designed to create them. Although much work has been done to characterize damage in composite materials, there is a significant void of understanding of damage accumulation mechanisms for highly heterogeneous materials which may fail by multiple modes. Advancing the understanding of such materials, especially in terms of their mechanical behavior, would further and extend their development and use.

For large strain values, and for long term service of such materials, analysis requires monitoring the mechanisms and extent of damage accumulation. For this purpose, cyclic out of plane bending was introduced to thin structural composites as a method of inducing non-uniform distributions of damage. Electrochemical impedance spectroscopy (EIS) was then employed as a means of characterizing the instantaneous material state. EIS is an electrochemical technique with common applications in corrosion, battery development, fuel cell development, pain characterization, sensor development, and physical electrochemistry [1]. In this technique the impedance is measured directly in the frequency domain by applying a single-frequency voltage across a material volume and measuring the phase shift and amplitude of the resulting current at that frequency. The frequency is then swept through a user defined range and the corresponding currents recorded. Using this method, any intrinsic property that influences the conductivity of a material system (i.e. changes in the material state) can be studied.

The history of response of a structural component to an applied condition (such as mechanical, chemical, hydrothermal or time-dependent load) depends on the underlying physics of evolution of material variables which control the ability of the material to react to that applied condition. The popular term "progressive damage" is often used to characterize such degradation behavior in general. There is an extensive literature on representations of damage and degradation in composite material systems based on change in physical properties (e.g., strength and modulus) or some measurable quantity (such as damage area). In this study, the ability of EIS to detect and represent progressive damage in composite materials is explored by comparing changes in the impedance spectra to the evolution of physical properties such as residual strength.

The introduction of electrical conduction paths into the material by micro-damage caused by mechanical loading was postulated as the mechanism that connects the material state to the EIS data. Since the material tested, a glass/epoxy composite, is essentially an insulator, which is incapable of providing a significant signal observable through the EIS method, the undamaged material provided a null result. By exposing the material to a certain level of moisture, hydration of the material enabled any incipient cracks to become utilized as conduction paths which enabled EIS to be employed. The nucleation and growth of those cracks was monitored and quantified using several forms of microscopy which present a strong correlation with EIS results.

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doi:10.1016/j.compscitech.2009.05.007

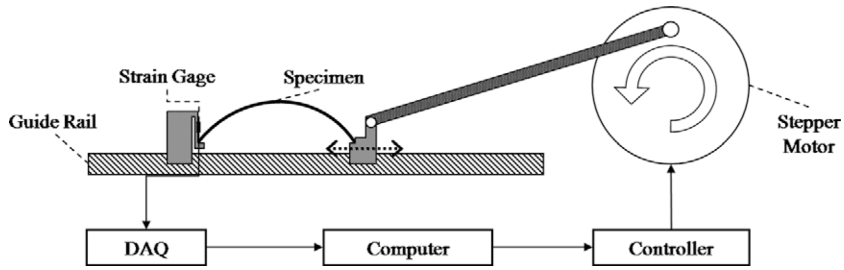

Fig. 1. End-loaded bending rig schematic for cyclic loading of composite specimens.

2. Experimentation

2.1. Material

Norplex-Micarta, a manufacturer of high performance thermoset composite laminates, provided the thin laminated composites used for fatigue testing. The composite material chosen (NP130) is made with a plain weave, E-glass fabric combined with a halogenated epoxy resin matrix. The manufacturers estimate 55% volume fraction of fiber. The samples were composed of five plies, where each ply consisted of a single layer of fabric with two principle fiber directions of 0° and 90°, with the warp direction defined as the 0° orientation. The total thickness of the samples was 1 mm. Samples were cut at various orientations (0°, 45°, 90°) relative to the principal axis thus allowing examination of material state changes as a function of loading angle.

2.2. End-loaded bending

To induce damage and mechanical degradation into specimens, cyclic out of plane bending by end-loading was implemented. The out of plane behavior was selected because of its relevance to thin-section materials, its ability to concentrate degradation, and its relationship to nonlinear shear strain effects which are often the basis for progressive damage development. Fig. 1 shows an illustration of the cyclic bending experiment.

Using the theory of the elastica, and based on the previous work of Mahieux et al. [2], a relationship between the end-to-end distance of a bent specimen and the strain at the center of the specimen (point of maximum strain) was derived. Details of the analysis can be found in Refs. [2,3]. Through this setup the loading histories of fatiguing samples can be monitored and later related to its corresponding material state.

2.2.1. Preliminary testing

To provide a baseline to select which range of strains a specimen will experience while in cyclic bending, quasi-static tests were performed wherein specimens of each orientation were bent until fracture. The end-to-end distance at failure was then recorded and the corresponding maximum strain calculated. Identical specimens were also pulled in a simple tension test for comparison. Table 1 summarizes the results of this comparison of strain to break.

Comparing the strain to break in bending with that in tension, it has been observed that there is a significant difference for off-axis specimens. Based upon these preliminary test observations it has been suggested that the failure of on-axis specimens experiencing end-loaded bending is dominated by tension, while the off-axis specimens experience other failure modes.

By adjusting the bending rig and the specimen length, the maximum strain of a specimen in bending during a fatigue cycle was controlled. For the 0° and 90° orientations the specimens were cut to dimensions of 152.4 mm × 17.8 mm, and 101.6 mm × 17.8 mm for the 45° orientation. The location of the fixed end support on the bending rig was adjusted thus allowing three levels of maximum applied strain, Fa (∼50%, ∼60%, ∼70% of the strain to break in bending), to be experienced during cyclic testing.[1] For each orientation a series of ten identical specimens was fatigued at a rate of 0.5 Hz until failure. Table 2 summarizes the cycles to failure for all orientations and Fa levels. The accuracy and repeatability of all tests was verified using a Weibull analysis and all results were found to be within the common confidence range.

2.3. Electrochemical impedance spectroscopy

In order to try to make direct associations between the loading histories and data, and the local material state changes associated with the damage that accumulated during the cyclic loading, EIS has been adapted to monitor the local details of damage development and accumulation. Details of the experimental methods developed for this purpose and a proof of concept can be found in Ref. [3].

EIS tests were conducted using a Gamry Reference 600 Potentiostat along with Gamry's EIS300 software. In this application, a sequence of impedance measurements were carried out starting at an initial frequency of 1 MHz and stopping at a final frequency of 100 Hz. A schematic of the test setup used for EIS testing is presented in Fig. 2.

In this setup, rectangular composite specimens being tested were placed between two 17.78 mm × 50.8 mm × 6.35 mm copper plates aligned in parallel with one another. The contact plates and specimen were compressed via a toggle clamp, which allows for a consistent clamping force, and the leads of the potentiostat were then electrically connected to the copper. The two-terminal test connection used was chosen based on the fact that the impedance of the sample is so much greater than the impedance of the test cables, so that any errors introduced by the cables (that could otherwise be minimized through the use of a four point connection) are unlikely to significantly affect the sample measurement results [1]. It should be noted that preliminary testing investigated the size and effect of the contact electrodes. Results suggest that the output signal is indeed damage area dependent. For the scope of this paper, the setup described above was solely used leaving further investigation in this area for future work.

2.3.1. Basic theory of EIS

Electrochemical impedance spectroscopy is a frequency response technique where a sinusoidal electric potential is applied to a system, and the responding sinusoidal current signal is measured. Typically, a spectrum is generated by sweeping a range of frequencies and measuring the impedance at each point. Impedance is a totally complex resistance encountered when a current flows through a system which is most often interpreted as a circuit made of resistors, capacitors, or inductors, or any combination of

[1] The complex behavior of the specimen limited the various levels of maximum strain allowed to just one level of 70% for the 45° orientation.