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14. ABSTRACT Prognosis is the general pursuit of predicting future performance on the basis of current assessment and history of behavior. The focus of this research is very different from the traditional concept of prognosis. We are concerned with heterogeneous materials which have meso-phases that are very brittle, e.g., woven fiber reinforced polymer composites. Large (nonlinear) strains occur in such materials, and are responsible for important performance characteristics. Prognosis requires that we follow the mechanisms and extent of the extensive damage accumulation. We considered non-uniform distributions of damage caused by out-of-plane bending, and introduce a new method of following material state change, based on multi-physics formulations. Specifically, we use electrochemical impedance spectroscopy to follow and understand conductivity changes as a second material property that varies with changes of state. With this new methodology, we address the question of how to predict future performance based on understandings of material state changes induced by prior service					
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Final Technical Report

**PREDICTIVE METHODS FOR LARGE-SCALE PROGRESSIVE DAMAGE IN
STRUCTURAL COMPOSITES FOR AIRCRAFT APPLICATIONS**

FA9550-07-1-0460

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ABSTRACT

Prognosis is the general pursuit of predicting future performance on the basis of current assessment and history of behavior. The traditional concept of prognosis is most familiar in the context of medical evaluation of the condition and prospects of a patient. For structural mechanics, there is an array of contexts and interpretation. One such scenario is the prediction of the stability or likelihood of collapse from information about response to 'safe' loading conditions.[1,2] Another context has to do with the large context of health monitoring, in which structural characteristics such as stiffness,[3] or more localized information from either passive (e.g. acoustic emission) or active (e.g. surface wave excitation response)[4,5] assessment is used to evaluate 'degradation' as a method of estimating future performance. A rather robust literature presents a context of fracture mechanics, focused on localized deformation, crack initiation and propagation, in some cases including specific address of the effects of local constituent particle effects on that sequence on prognosis of performance and life.[6] And finally, there are contexts based on crystal plasticity in which, for example, microstructure characterizations are used anticipate the relationship between shear strain and damage nucleation on a local scale, e.g., at grain boundaries.[7]

The focus of this research is very different. We are concerned with heterogeneous materials which have meso-phases that are very brittle, e.g., woven fiber reinforced polymer composites. Large (nonlinear) strains occur in such materials, and are responsible for important performance characteristics.

We will approach these requirements by establishing constitutive relationships for highly nonlinear strains (up to 20 percent) in woven-fiber reinforced polymer composites as a foundation of understanding stiffness and strength changes in anisotropic materials as state changes associated with distributed micro-cracking in uniform stress states.

For such materials, and for large strain and long term conditions, prognosis requires that we follow the mechanisms and extent of the extensive damage accumulation. For this purpose, we will introduce out-of-plane bending to thin structural composites as a method of introducing non-uniform distributions of damage, and introduce a new method of following material state change, based on multi-physics formulations. Specifically, we

use electrochemical impedance spectroscopy to follow and understand conductivity changes as a second material property that varies with changes of state.

With this new methodology in hand, we address the question of how to predict future performance based on understandings of material state changes induced by prior service.

We will offer suggested directions and observations in this report, but the later part of the effort is work in progress.

OBJECTIVES OF THE PROGRAM:

1. To establish relationships between functional properties and characteristics of fiber-reinforced composite materials (e.g. conductivity and impedance) and long-term mechanical behavior under repeated loading.
2. To establish relationships between material state in composite materials (and state changes during long-term mechanical behavior) to functional properties and characteristics.
3. Use the foundation of understanding of the relationship of material state changes to functional property changes to construct a multiphysics prognosis analysis that correctly relates the material state and functional behavior (as measured by impedance spectroscopy) to long-term mechanical behavior.
4. Construct a simulation methodology for prognostics in fiber reinforced composite materials for which the materials are the sensors; the material state changes are the theory; and multiphysics is the method.

STATUS OF THE EFFORT:

The present program began in the second half of 2007. In the last annual report (2007-2008), we introduced a novel idea of measuring material state changes during cyclic loading, following functional property changes during that loading using electrochemical impedance spectroscopy (EIS), and relating the two data sets with multiphysics analysis. A cyclic loading rig for end-loaded compression (producing out of plane bending) was designed and built. Woven glass-epoxy composites (of several types) were cycled to various stages of 'damage' as indicated by changes in mechanical properties and visible degradation. Data were obtained relating EIS measurements at different frequencies and in different hydrations to damage development for several materials and mechanical loadings. These data, which we believe to be the first of this kind, firmly establish the feasibility of the concept and method [8].

Continuing with that work using EIS, we now have further understanding of the damage initiation and development caused by cyclic loading. A systematic approach in which the evolution in residual strength due to fatigue in bending was monitored, and a relationship

between electrochemical impedance at different frequencies of excitation and the development of damage in that material was established. Metrics defined by impedance spectroscopy measurements were found to show large, consistent, and clear distinctions as damage developed with remarkable sensitivity which may not be otherwise possible through measuring change in physical properties or visual damage [9].

Another important aspect of the work is formulation constitutive relationships for large-deformation nonlinear strains. Significant progress has been made in this area, specifically for in-plane loading, as reported earlier [10] and current focus is on such formulation for out of plane deformation (end-loaded bending) over the elastic-plastic range [11].

Then a multiphysics model of the impedance spectroscopy method will be constructed and those material state changes will be used in an attempt to predict the observed EIS changes. That multiphysics will form the basis for an engineering durability model based on electrochemical impedance spectroscopy interpretations that associate the micro-details of how a composite material is made and the history of degradation (associated with individual use) with specific prognosis for long term performance, with attendant reductions in design, manufacturing, and maintenance costs and increases in reliability and durability.

GLIMPSE OF ACCOMPLISHMENTS AND NEW FINDINGS:

EIS Measurement:

- Built an cyclic end-loaded bending test rig for fatigue of composite samples [8]
 - Rig can simultaneously fatigue four identical samples
 - User defined cycle rate
 - User defined maximum cycle strain
 - In-situ monitoring of end force
 - In-situ monitoring of strain throughout sample via relation of end-to-end distance (theory of elastica)

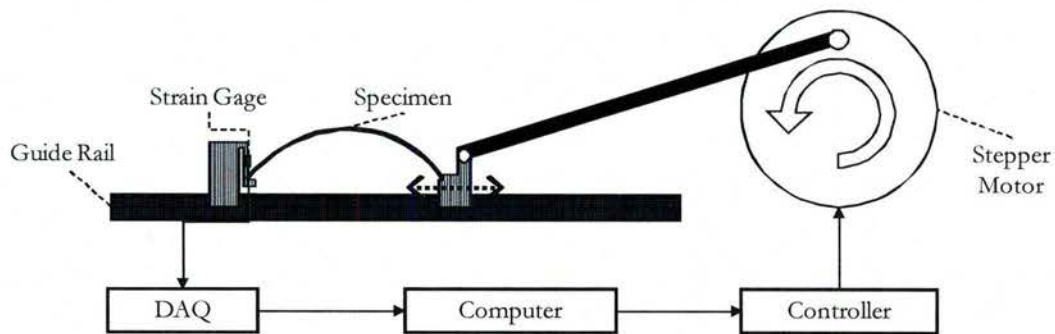


Figure 1: End-loaded bending rig schematic for cyclic loading of composite specimens

- Developed a method using Electrochemical Impedance Spectroscopy to repeatedly measure the impedance of thin woven glass/epoxy composites over a defined range of frequencies [8]
 - Built controlled test setup with humidity chamber, small 2" x 0.7" electrodes for localized testing, and toggle clamp for constant pressure during tests

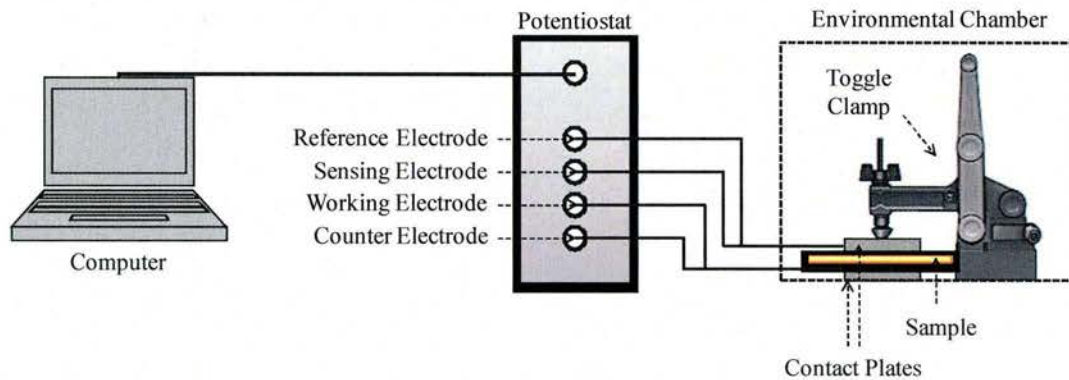


Figure 2: Schematic of EIS experimental setup for examining damage in composite samples

- Characterized the chosen glass fiber/epoxy composites (Norplex Micarta NP130) through tension testing at various orientation in order to obtain a nonlinear and rate dependent constitutive model for progressive failure modeling and analysis [10]
 - Six orientations
 - Full Weibull analysis (statistical confidence)

- Master curve obtained
- Monitored the evolution in residual strength and breaking strain of composite specimens due to out of plane bending fatigue [9]
 - Residual strength and strain to break were determined for three levels of fatigue life (25%, 50%, 75%)
 - A residual stress and strain analysis was conducted for a single F_a level of 0.5 for 0° and 90° orientation, and 0.7 for the 45° orientation
- Monitored the evolution of the area fraction of visual damage of composite specimens due to out of plane bending fatigue
 - To quantify the area of possible conduction paths in a damaged cycle, a visual analysis was conducted on specimens that had been fatigued out of plane for various levels of fatigue life.
 - To visually observe the damage incurred during bending fatigue the best method found involved the use of a dye penetrant
 - This method was particularly attractive because the cracks which the dye penetrant highlights are the same conduction paths that are observable through EIS
- Established a relationship between impedance at different frequencies and the changes in the resistance and capacitance during specific ranges of frequency. The following two plots used for the EIS analysis are the Nyquist plots of the imaginary versus the real part of the impedance, and the Bode plot of impedance magnitude versus frequency both for the 90° orientation.

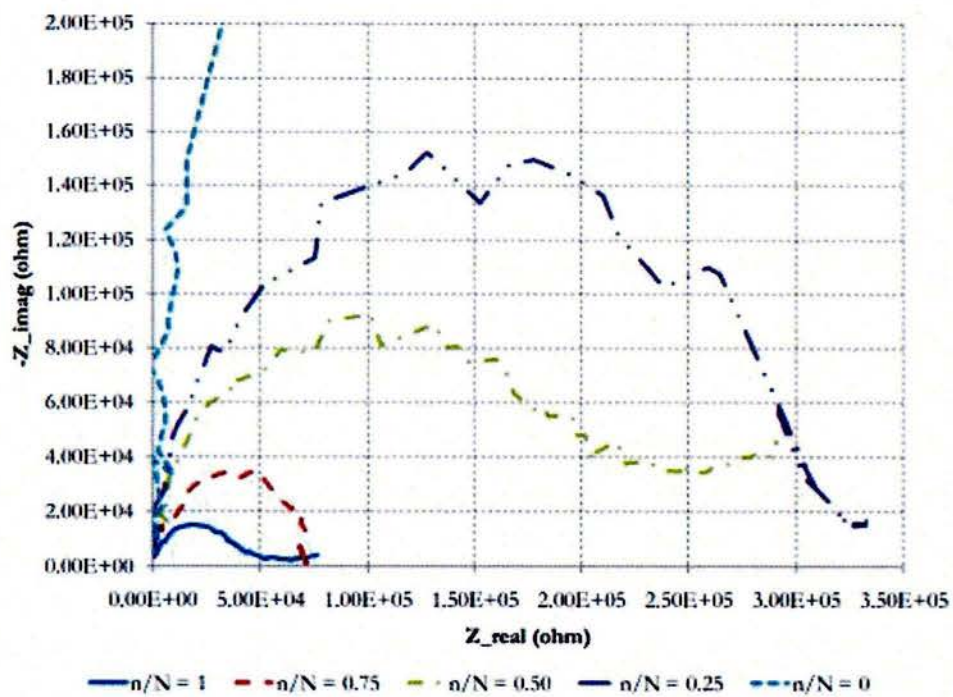


Figure 3: Nyquist plot – 90° specimen

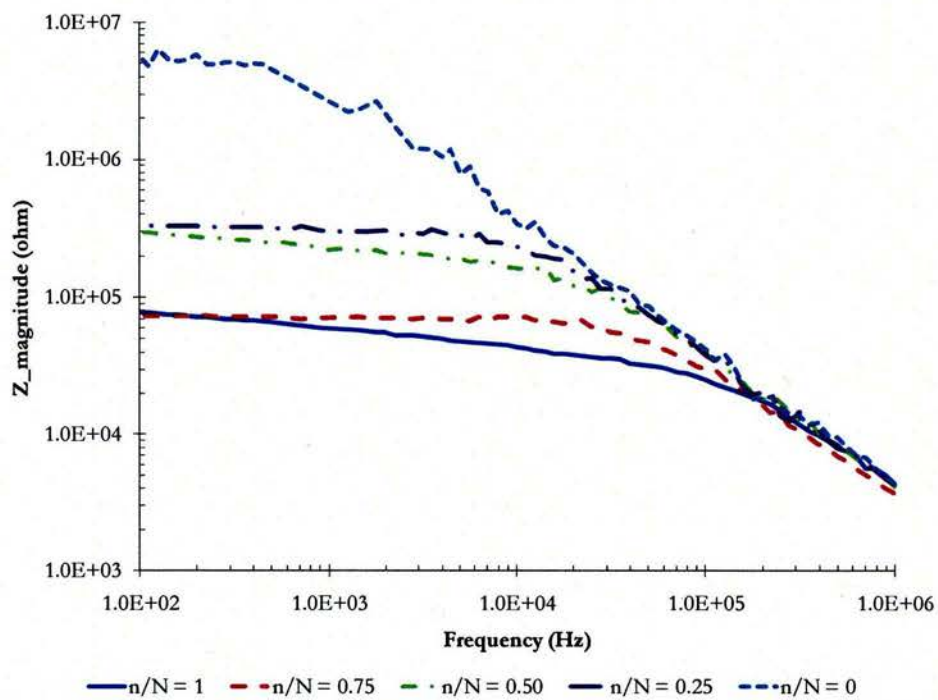


Figure 4: Impedance magnitude vs. frequency – 90°

Several features of the results shown in Figures 3 and 4 are especially notable. Both the real and imaginary parts of the AC impedance are greatly altered by the presence of progressive fatigue damage in these thin glass/epoxy specimens. Indeed, even though the area (and volume) of damage in the bent specimens was restricted to an inch or so of the length of the specimen (near the center), the changes in the impedance were remarkably distinct, consistent, and large. A second feature of interest is that the shapes of the curves are quite different as progressive damage develops, suggesting that there is a lot of information available about what specific material state changes are occurring during the damage development process. And finally, the sensitivity of the method is striking. The largest single change occurs between the undamaged and the 25% fraction of life level. Similar EIS trends were recorded for the 0° orientation.

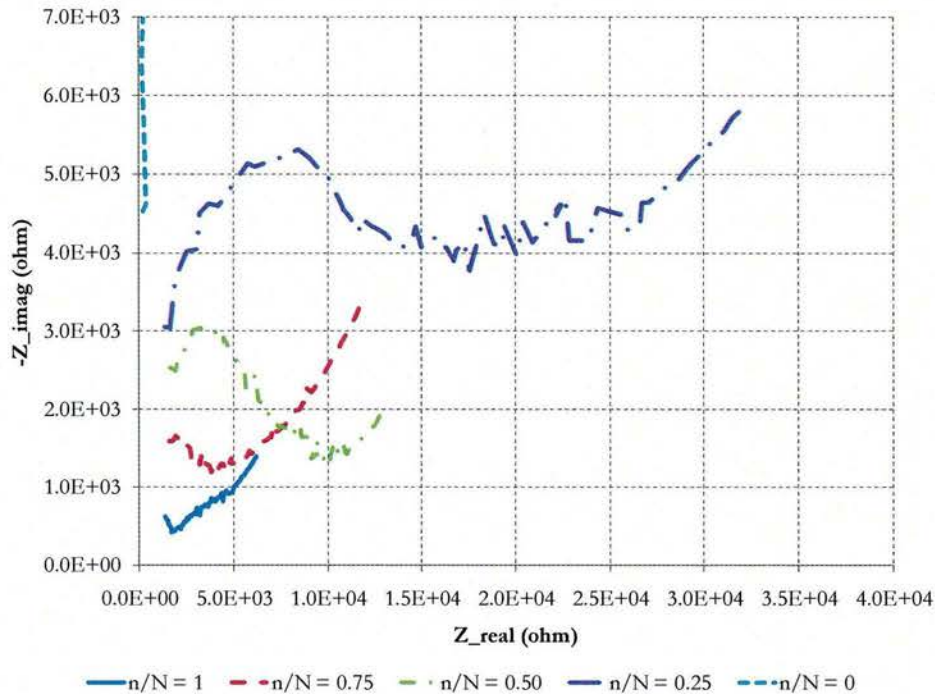


Figure 5: Nyquist plot – 45°

Figure 5 presents EIS results for the 45° orientation. Whereas the 0° and 90° orientations show similar features, the 45° orientation presents new and different traits. The most obvious of those is the significant decrease in overall impedance magnitude, over two orders of magnitude lower than those observed for the on-axis cases. This can be attributed to the significant increase in damage experienced for off-axis specimens, which supports the hypothesis earlier stated. The matrix dominated behavior leads to more matrix cracking and hence more hydration sites. The second observable feature is the change in impedance at high frequencies. For the on-axis cases, the impedance converges at high frequencies and differences in the magnitude are most observable when the frequency falls below 10,000 Hz. For the 45° orientation however there is a constant change throughout the frequency range indicating significant changes in both capacitance as well as resistance. This new change in resistance not observed for the on-axis cases indicates that continuous conduction paths are being formed through the thickness of the specimen.

- Established a relationship between impedance at different frequencies of excitation and the development of damage in the material (residual strength and visual area fraction of damage).

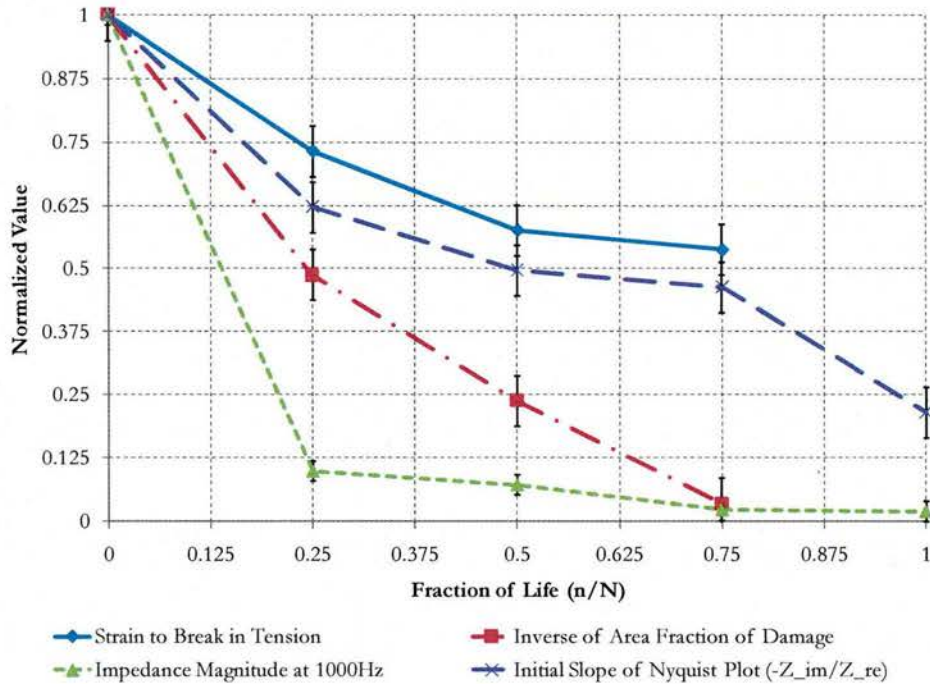


Figure 6: Summary of damage metrics - 90° orientation

From Figure 7 it can be seen that as a specimen is increasingly fatigued and its area fraction of damage increases (inverse of red line), all of the other metrics decrease. Based on these results it can be concluded that EIS can be used as a means of measuring damage in the structure of a composite material. The rate of change in the initial ratio of imaginary to real impedance (Nyquist plot) is similar to that of the breaking strain in tension. In addition, the magnitude of the impedance is by far the most sensitive metric observed, showing a drastic decrease at only 25% of the fraction of life. Interestingly, the EIS curve for the magnitude of impedance at 1,000 Hz lies below the damage area and change in residual properties curve which again shows the sensitivity of this method to capture the underlying mechanisms of damage.

- 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.

In summary, EIS is a promising tool for characterizing progressive damage in a composite material and has the potential to provide information about material state changes with remarkable sensitivity that is not seen when measuring change in other physical properties or visual damage.

Elastica and Plastica Formulation of Out of Plane Bending:

End-loaded bending is a method of conducting bending tests on composite laminates which provides several advantages over conventional three or four point bend configurations. Experimental results show that off-axis woven composites can have significant out of plane plastic deformation even very early in the stress-strain relations predicted by data from tension behavior. The plastic deformation is large at the first loading cycle and continues to grow over each loading-unloading cycle. Using combined elastica-plastica theory, many response variables can be predicted which eliminates the

need to experimentally measure those quantities. Capability to obtain the deformed shape theoretically with a minimum of experimental data is an attractive feature which will help future development of constitutive relations for woven composites in end-loaded bending. This is an ongoing work and the authors expect to use as a foundation for more complete formulations of out of plane constitutive behavior of woven laminates.

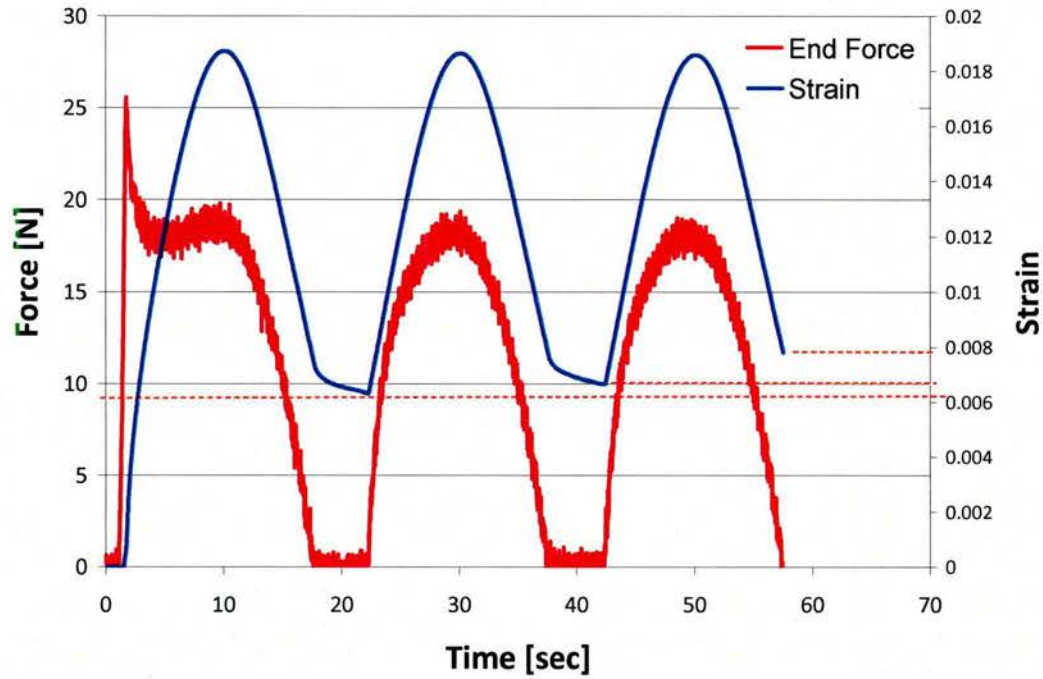


Figure 7 Variation of End force and strain with time over three loading cycle (45deg woven)

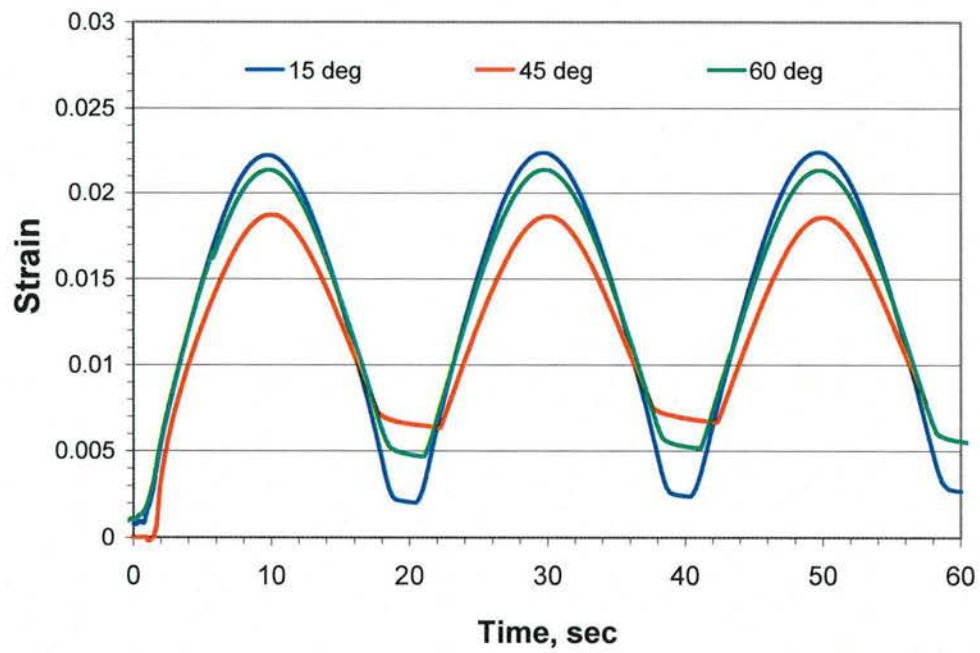


Figure 8 Comparison of plastic strain for different off-axis specimens

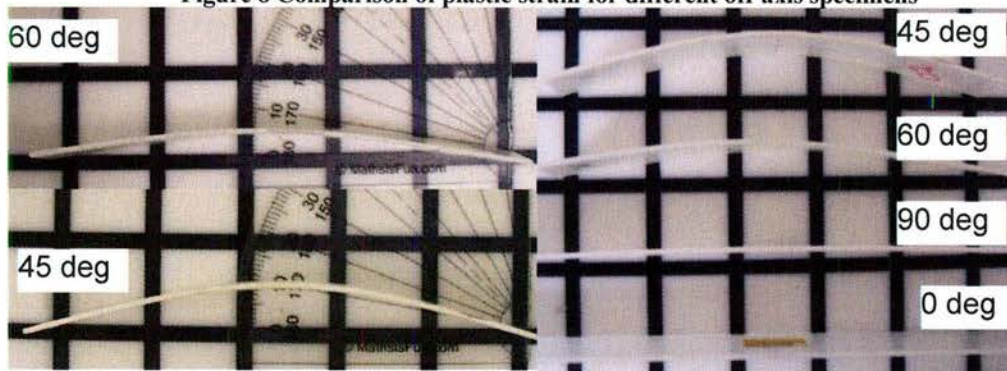


Figure 9 Deformed shape of off-axis specimen after unloading

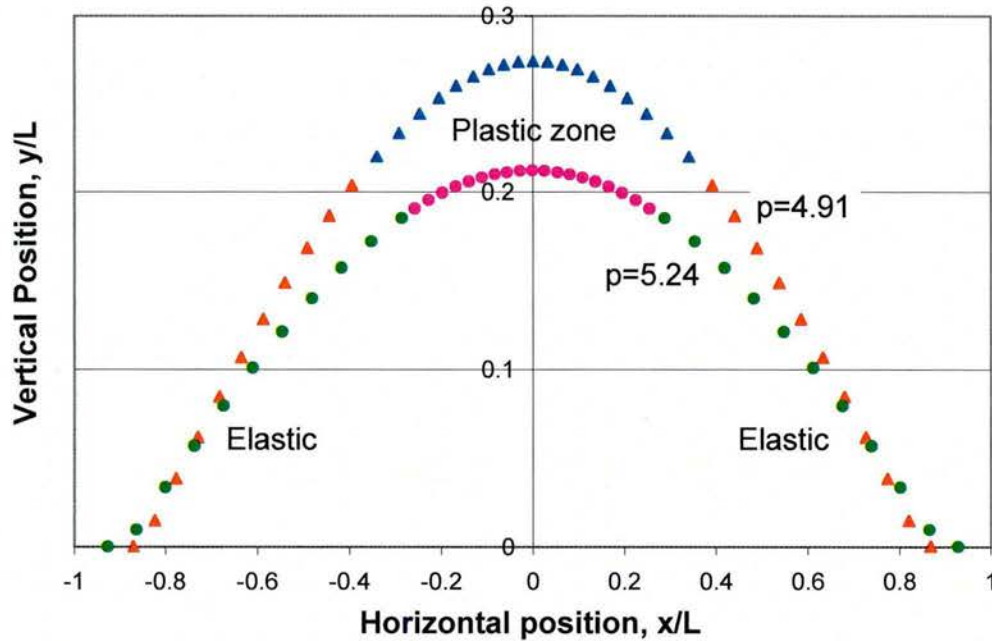


Figure 10 Predicted deformed shape at different load level

FUTURE WORK:

Future extension of this research may focus on identifying individual damage modes associated with the captured EIS response and on extracting specific information about material state changes. Such correlations will facilitate development of life prediction schemes with minimum dependency on empirical relationships. Also, completion of the constitutive formulation and multi-physics modeling will be at the heart of future work.

PERSONNEL SUPPORTED:

- Paul Fazzino – MS student in the Department of Mechanical Engineering, University of South Carolina.
- Prasun Majumdar – Postdoctoral Fellow in the Department of Mechanical Engineering, University of South Carolina.

LIST OF PUBLICATIONS WITHIN THIS PERIOD

Refereed Journal Articles:

- Fazzino, P., and Reifsnider, K. L. "Electrochemical Impedance Spectroscopy Detection of Damage in Out of Plane Fatigued Fiber Reinforced Composite Materials," *Applied Composite Materials*, (2008), Volume 15, Number 3, pp127-138.
- Fazzino, P., Reifsnider, K., Majumdar, P. "Impedance Spectroscopy for Progressive Damage Analysis in Woven Composites," Accepted for publication in *Composite Science and Technology*, (2009).
- Kenneth Reifsnider, Paul Fazzino and Liqun Xing. "Material State Changes as a Basis for Prognosis in Aeronautical Structures". Submitted for publication in the *RAeS Aeronautical Journal*. (In review)

Conference Proceedings and Presentations:

- Majumdar, P, Fazzino, P., Reifsnider, K. Behavior of Woven Fabric Composites in Off-axis End-loaded Bending. Accepted for publication in the SAMPE Conference Proceedings. May 18-21, 2009, Baltimore, MD.
- Fazzino, P., Reifsnider, K., Majumdar, P. Impedance Spectroscopy of Fabric Reinforced Composites. Accepted for publication in the SAMPE Conference Proceedings. May 18-21, 2009, Baltimore, MD.

Thesis:

- Fazzino, P. "Predictive Methods for Large-Scale Progressive Damage in Structural Composites for Aircraft Applications." University of South Carolina, MS Thesis (2008)

NEW DISCOVERIES:

We have discovered a method for relating Potentiostatic Electrochemical Impedance Spectroscopy (EIS) measurements to progressive damage in fiber reinforced composite materials based on first principles multiphysics interpretations of material state change. Comparison with residual strength degradation due to cyclic loading illustrates that the EIS measurement is indicative of real material state change, and capable of providing much higher sensitivity than conventional damage metrics.

HONORS AND AWARDS:

Dr. Ken Reifsnider is a member of the National Academy of Engineering. He is currently Director of the Future Fuels Initiative at the University of South Carolina, and Director of the Solid Oxide Fuel Cell Program there. He is past Editor in Chief of the *International Journal of Fatigue* (1998-2008), Associate Editor of the *ASME International Journal of Fuel Cell Science and Technology*, and North American Editor of the *International Journal of Applied Composites*.

CONCLUSIONS

The present research develops a philosophy and methodology for the prognosis of future performance of aeronautical composite structures based on history of use and current material state. The methodology is based on the general concept that local values of stiffness, strength, and conductivity (for example) are altered by material degradation to create “property fields” that replace the global constants, and introduce time and history into the governing equations for performance and response. The methodology includes three major elements: nonlinear constitutive equations for large strains (up to and including the break strain) that support fully nonlinear stress-strain analysis throughout structural life, in the presence of progressive damage; electrochemical impedance spectroscopy methods of following the local, mechanistic details of the development of damage and the consequent changes in material state, in real time, with multi-physics interpretations of the data in terms of micro-damage development and conduction path comparisons to fracture path development; and prognosis of future performance based on simulations of response based on current material state and projected state changes as a function of projected mission. The first of these elements is well in hand. An extensive data base and a group of published work by the authors confirm that a robust progressive damage constitutive methodology has been constructed. The second element has reached the level of proof of concept. Experiments confirm that EIS data provide a remarkably sensitive and extremely robust experimental space of information that relates directly to the development and accumulation of micro-damage in the materials considered. There is every reason to believe that the physics of conductivity in the presence of fragmented

conduction path development in heterogeneous materials can provide mechanistic interpretations of these data as a result of future work. Up to this point, only a phenomenological interpretation has been suggested. The third element of evolution rate equations, based on either phenomenological or mechanistic methods, is largely conjectural at this point.

However, the development of EIS as an assessment method for material state changes has opened a new door of opportunity to make the material itself the sensor, and the material state (as represented by the material constants in multi-physics equations – like the field equations for complex conductivity of heterogeneous materials) the metric for performance and prognosis. Much is yet to be done, but the results of this current effort point to many opportunities for progress.

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