Assessment of Material State for Predicting the Durability of Composites

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ABSTRACT

The long term behavior of composites have been extensively studied for the last four decades. Given the heterogeneity of these materials, the damage accumulation mechanisms lead to superior fatigue performance of composites compared to metals. However, due to the ‘sudden death’ behavior controlled by defect coupling, the precursor to fracture plane development of these materials, the challenge remains on how to assess the real-time material state and predict when it becomes critical? In the recent past, broadband dielectric spectroscopy (BbDS) has been used successfully to assess the material state and predict the material state change (triggered by defect coupling) for quasi-static loading. In this work, we perform in situ monitoring of material state change for tension-tension fatigue loading (low cycle fatigue) and attempt to capture the material state change for quasi-isotropic laminates. The in situ response can be used to predict the formation of a ‘critical’ material state- the final frontier to predict the durability of composites and discuss the repeatability of the methodology.

INTRODUCTION

The long term behavior of composites have been extensively studied for the last four decades. Several models have been developed that attempt to estimate the residual properties for different loading conditions, different sequence of loading.

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However, all the findings and conclusions are generally specific to certain stacking sequences and/or certain material types and have been reviewed by Paepegem et al. [1-2]. Given the heterogeneity of these materials, the damage accumulation mechanisms lead to superior fatigue performance of composites compared to metals. In general cases, the high cycle fatigue (HCF) studies which run for almost $10^6$ cycles; combined with advanced testing capabilities with a frequency of 10-100 Hz are generally useful for characterization in metals. However, for composites operating at such high frequencies lead to viscous heating effects and completely changes the material behavior [3]. The maximum stress $\sigma_{\text{max}}$ for HCF studies are typically much lesser than 30\% of quasi-static ultimate tensile strength $\sigma_{\text{ult}}$ of the laminate. On the other hand low cycle fatigue (LCF) studies in which $\sigma_{\text{max}}$ is typically in the range of 50-90\% of $\sigma_{\text{ult}}$ are run for typically designed to withstand $10^4$ cycles [4].

Given the heterogeneous nature of these material systems, the damage development in these materials are complex; a technique capable of capturing the local damage events is critical to better understand the ‘state’ of the material system. Electrochemical Impedance Spectroscopy (EIS), Broadband Dielectric Spectroscopy (BbDS) are such techniques to extract the material-level information, including the morphology changes caused by micro-defect generation and the orientation of those defects [5]. Because of the inherent heterogeneity, these materials act as self-sensing systems where changes in microstructure (local) due to cracks/voids/defects alter the global response of the material. In the recent past, several researchers have utilized EIS and BbDS techniques to monitor damage growth in composites under static and fatigue tests.

Reifsnider et al., conducted flexural bending based fatigue testing in GFRP composites, and monitored the damage development using EIS. It was shown that during the damage progression, the real and imaginary parts of the impedance changed due to the conductive paths created in the material. Hence, through this method the degradation of the composites in terms of the damage and durability can be estimated from the EIS data [6]. Seo et al., [7] studied the fatigue damage in CFRP laminates by electrical resistance measurements. The electrical resistance gradually increased as the stiffness reduced and showed very abrupt change when the final fatigue failure in imminent. Both the measurements of the stiffness and resistance showed very similar trends. They also developed an electrical resistance based damage parameter model. Zhao et al., [8] carried out cyclic tensile tests with CNT polymer films as the sensors, to capture the deformations measured from resistivity and strains using electromechanical sensing system. Giurgiuțiu et al., [9] demonstrated that permanently attached PWAS in conjunction with the electromechanical impedance method can be successfully used in structural health monitoring to detect the presence of incipient damage through the examination and classification of the high-frequency E/M impedance spectra. Almuhammadi et al., [10] carried out static and cyclic tests on CFRP composites performing EIS studies. It was observed that the depth probing capability of EIS is highly dependent on the conductivity of the surface along the measurement direction. De Baere et al., [11] investigated the use of electrical resistance technique for monitoring fatigue damage, using four probe method. They observed that the longitudinal resistance is very sensitive to fiber failure.
In the current work, composites are loaded in tension-tension fatigue and the change in dielectric properties are monitored simultaneously to interpret the material ‘state’ change. The observations will be discussed in detail below.

EXPERIMENTAL SETUP

Sample Preparation

In this study, unidirectional glass fiber reinforced polymer (GFRP) composites (Rockwest 250F Epoxy/E-glass fiber (volume fraction 55%)) were manufactured. These have application in different structural fields of automotive bodies, marine sector, sporting goods, medical and industrial manufacturing and exhibit very good mechanical strength and stiffness properties. The laminas were stacked in [-45°/90°/45°/0°]s sequence to obtain a quasi-isotropic laminate behavior. The prepregs were cured out of autoclave using compression molding technique where the temperature and pressure were ramped up from room temperature to 275°F at the rate of 5°F/min and from atmospheric pressure to 40 psi respectively. The prepregs were cured for 90 minutes at 275°F and 40 psi and then cooled at the rate of 5°F/min from 275°F to 100°F while reducing the pressure to atmospheric conditions as per the manufacturer’s recommendations. To prepare specimens for tension-tension fatigue testing, ASTM D3479 has been followed [12]. The cured panels were cut precisely using Protomax Water Jet Cutter, and then sanded at the edges to ensure the absence of any kind of flaws which could cause premature failure. The final specimen dimensions are 10” × 0.75” × 0.063”.

Testing Setup

The samples are loaded in tension-tension fatigue under load control using a MTS hydraulic system with a 50KN load cell. To measure the dielectric properties during loading, the sample is sandwiched between electrode blocks to form a parallel plate capacitor setup as shown below in Figure 1. The electrode blocks are connected to the analyzer of a NOVOCONTROL™ unit which can measure the impedance, capacitance etc. as a function of frequency with high precision. In the current work, a frequency of 100 Hz (to detect interfacial polarization) was used for the dielectric response in order to obtain low frequency response with a high sampling rate which is crucial to capture the change in material state [13].

The samples were loaded in quasi static loading initially to obtain the breaking load. The specimen was loaded using displacement control at rate of 0.3 mm/min. The average breaking load for these specimens was ~11 KN. To perform fatigue, the specimen was initially loaded to ~50% of \(\sigma_{ult} (6 \text{ KN} (\sigma_{mean}))\). An amplitude of 2 KN was applied to cycle between (4 KN (\(\sigma_{min}\)) – 8 KN (\(\sigma_{max}\))). The cycling was performed using a frequency of 2 Hz and the data was acquired at every 0.15 s to capture the peak and valley loads and displacements. The dielectric data was obtained every 3 s in quasi static loading and every 10 s in fatigue loading.
RESULTS

Quasi-Static Loading

The average breaking load of these specimens was ~11 KN. The stress strain response and the variation in dielectric properties are shown below in Figure 2. It can be observed that the dielectric ‘state’ variable (Re(permittivity) $\varepsilon'$) increases in the beginning due to matrix cracking resulting in charge storage at the newly created crack surfaces, followed by saturation in dielectric state variable due to the saturation of the matrix cracks (referred to as ‘characteristic damage state’ (CDS)), followed by an initial decrease of dielectric state variable due to initiation of delaminations followed by steady decrease due to accumulation of delaminations leading to local fiber failures resulting in global fracture [14]. From Figure 2, it can observed that the dielectric response is consistent for different samples indicating the repeatability of this technique.
Fatigue Loading

The stress strain response for one of the specimen is shown below in Figure 3. The coupon failed around 12042 cycles. The rapid increase in strain at the beginning indicates the rapid decrease of modulus in the beginning followed by steady increase of strain correlating to steady decrease of modulus and followed by rapid increase in strain due to rapid interactions of local damage events leading to rapid decrease of modulus [15]. A similar trend can be observed in the insitu dielectric response as well shown below in Figure 4.
From Figure 4, it can be observed that the permittivity increases at a higher rate in the beginning, followed by reduced rate of increase and eventually leading to saturation and starts to decrease at the end stages. The behavior is similar to quasi static behavior. However, in quasi static behavior the changes in the dielectric response have been attributed to specific damage events in the laminate. The increase in the beginning is caused due to accumulation of charges at the newly formed surfaces, saturation of the dielectric response is triggered by the formation of a characteristic damage spacing (CDS), the interaction of damage modes leads to the initial drop in the dielectric response and accumulation of local failures resulting in global fracture triggers the sudden drop in the dielectric response. In fatigue, we believe the changes observed in the dielectric response could be triggered by the rate of damage development which from literature is shown to be significant in the beginning, steady in the middle and significant again in the end stages due to the interactions [15]. To better understand this, the slopes of the dielectric response were calculated; for this the cycles were normalized with respect to maximum cycles the sample experienced, the so called ‘life’ and a fourth order polynomial curve fitting was done as shown below in Figure 5. The slopes of the dielectric response are shown in Figure 6.

![Figure 5. Dielectric response in fatigue](image-url)
From Figure 6, it can be observed that the first slope (which indicates how the dielectric response changes) indicates that up to 90% of life the dielectric response increases and then starts to decrease. This is different in case of quasi static where the slope change occurs around 50-60% of strength [14]. The absolute value of second slope (which indicates the rate at which the dielectric response changes) indicates that the rate of change is significant in the beginning and starts to decrease and saturates at almost 60% of life and starts to increase again. We believe this change in rate is triggered by the interaction of local events, however more number of tests and damage replications are required to better understand the changes in the dielectric behavior and will be discussed along with the repeatability of the technique in subsequent publications.

CONCLUSIONS

In this work, quasi isotropic laminates made of glass fiber reinforced polymer matrix composites were manufactured and tested in quasi static tension and tension-tension fatigue (low cycle fatigue). The quasi static tests were consistent with literature showing different regimes of dielectric behavior, the increase due to formation of micro cracks, saturation due to formation of a characteristic damage spacing (CDS) and initial drop due to initiation of delaminations and rapid decrease in the end due to accumulation of local failures leading to global fracture. In the case of fatigue, the response was similar except that the rates and the triggers for the changes are not straight forward. It was observed that the dielectric response saturates at 90% of life in contrast to 50-60% of strength when loaded in quasi static tension. More number of tests and replicating damage patterns are required to better understand the triggers of the dielectric response and hence we might be able to correlate the changes to events in the life of a specimen.
REFERENCES