AIAA SciTech
13-17 January 2014, National Harbor, Maryland
55th AIAA/ASMe/ASCE/AHS/SC Structures, Structural Dynamics, and Materials Conference

Effect of Fiber Volume Fraction Variation Across Multiple Length Scales on Composite Stress Variation: The Possibility of Stochastic Multiscale Analysis

Ray S. Fertig, III[1], Eric M. Jensen[2], and Kedar A. Malusare[3],
Department of Mechanical Engineering, University of Wyoming, Laramie,WY, 82071

Prediction of composite failure is of critical importance to the design of composite structures. However, because failure of composite structures is typically sudden and catastrophic, understanding the stochastic behavior of failure is critical to accurate prediction of structural reliability. The research reported here focuses on quantifying the length scales of microstructural variability, specifically variation of fiber volume fraction, and their relationship to stress fluctuations in the bulk material. From this study, a stochastic multiscale progressive failure simulation of a compressive test is developed to demonstrate the feasibility of incorporating realistic stochastic information in a multiscale model. Unlike other stochastic studies that randomize strengths or stiffnesses, this approach randomizes an entire microstructure on the basis of volume fraction distributions computed directly from SEM images. Furthermore, the distributions are specific to mesh size. The predicted failure modes are correct and the scatter in the compressive strength predicted by the simulation is similar to scatter in strengths measured experimentally.

Nomenclature

λ = sampling cell size
ϕ_f = fiber volume fraction

I. Introduction

THE use of a representative volume element (RVE) for multiscale analysis of composite failure is widespread[1]. In particular, localization tensors are often used to extract constituent stresses at specific points or on average[2-10]; these stresses are then used to predict constituent failure. The RVEs used for this approach are almost always idealized: perfect bonding between constituents, constant material properties, and regular fiber packing (typically hexagonal or square). Yet widespread agreement exists that mechanical behavior and reliability of composites are significantly affected by defects (e.g. see reviews[11-16]). The unique challenge in predicting failure in composites is that ultimate failure behavior is complicated by a range of failure mechanisms[17] dictated by several factors including ply orientation, loading state, sample geometry, and constituent material behavior. These failure mechanisms are affected by various factors associated with microstructural variation and defects such as volume fraction, spatial distribution of reinforcement, and voids. Even in the case of simple loading and geometries the failure behavior of composites is complex and stochastic in nature. Despite the fact that microstructural variation and defects strongly influence the reliability of composite structures[18], relatively few studies have quantitatively examined the effect of defects on composite reliability[19], and none account for the fact that how a specific defect may degrade reliability depends on the local loading state. Because of the paucity of experimental research in this area and the challenge of stochastic multiscale modeling, physics-based methods for predicting reliability in structures have not yet been realized. Almost all current composite modeling approaches focus on *average* behavior.

[1] Assistant Professor, Mechanical Engineering Department, Dept. 3295, 1000 E. University Ave, AIAA Member.
[2] Graduate Student, Mechanical Engineering Department, Dept. 3295, 1000 E. University Ave, AIAA Student Member.
[3] Graduate Student, Mechanical Engineering Department, Dept. 3295, 1000 E. University Ave, AIAA Student Member.

The focus of this work is on understanding the role of a fiber volume fraction variation, a particular type of "defect," on the average stress states in a composite across length scales ranging from microns to millimeters. This was accomplished by first fabricating a unidirectional composite and imaging in a scanning electron microscope (SEM) to determine fiber packing morphology. The SEM image was then digitized for subsequent analyses. The deviation of volume fraction from the mean was computed as a function of sampling cell size. The digitized image was then used to create a voxel-based finite element model for the purpose of examining stress variations in the microstructure, again as a function of sampling cell size. Finally, the correlation between deviation in volume fraction and average stresses were computed as a function of sample size to show that this defect is a significant contributor to property variation in the composite. Using date from these microstructural analyses, a stochastic progressive failure finite element simulation was developed on the basis of the distribution of microstructures directly observed via SEM. These data enabled computation of scatter in material properties as well as observation of variations in failure morphology.

II. Experimental Setup

The material studied was a unidirectional carbon prepreg made of Zoltec Panex 35 50K carbon fibers impregnated with Hexcel's M9.7 epoxy resin system. This material was developed specifically for the wind energy industry. A $[0]_4$ laminate plate was laid up and vacuum bagged. Temperature was ramped to 120°C at a rate of 3-6°C/min under vacuum. After the temperature ramp, the pressure was increased to 414 kPa and held for 30 minutes. Then the cured plate was allowed to cool under pressure until it reached a temperature of 50°C. The cured laminate was nominally 2.1 mm thick. An SEM specimen 25 mm x 25 mm was cut from the plate using an abrasive water jet cutter.

To prepare the SEM specimen for imaging an edge was cut with a diamond saw and polished with 1000 grit sandpaper. An ultrasonic cleaner was used to remove loose particles and the clean surface was wiped with methanol. Finally, a light carbon coating was applied to the surface to help prevent charging of the matrix. Images were then acquired using a backscattered electron detector with a voltage of 20 kV, these are shown in Figure 1.

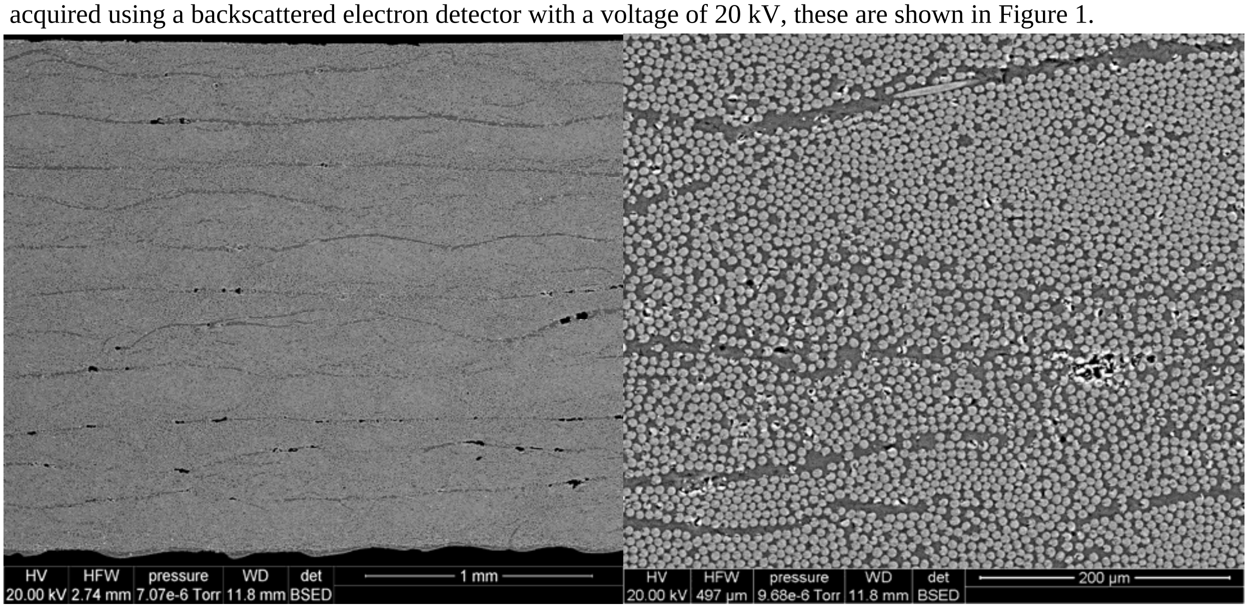

Figure 1. SEM image of a unidirectional laminate cross-section at (left) 55x and (right) 300x.

III. Modeling and Analysis

A. SEM Image Analysis

The SEM images were digitized to give an array with grayscale values ranging from 0 to 255. Histogram plots of these values indicated clear distinction between fiber and matrix. The fiber or matrix constituent was assigned to each pixel based on grayscale value of the image. Using these definitions, an average volume fraction was computed to be 0.593 for both the 55x and 300x image.

The SEM images shown in Fig. 1 illustrate that although the mean volume fraction is the same in both images, fiber volume fraction varies from region to region within these images. Of particular interest in this study was to assess how the volume fraction varied as a function of the microstructure sampling size λ. To assess this effect of length scale on volume fraction variation, square boxes with edge lengths ranging from 6 μm to 1000 μm were used to sample local regions of the microstructure. The volume fraction in each region was computed such that a standard deviation was obtain for each length scale. (The mean fiber volume fraction remains constant in each image with respect to sampling size at 0.593.) Figure 2 shows the effect of sampling cell size on the variation of the fiber volume fraction, represented by the coefficient of variation (COV). Two features in these data stand out. First, the variation in volume fraction decreases with increasing sample size, as expected. Second, typical RVE sizes are on the order of the length scale of a fiber diameter (~10 μm). At this length scale the COV of the fiber volume fraction is nearly 20%. Thus, if the distribution is bounded by two standard deviations, the RVE at any given point could range from 0.36 to 0.84 for a typical average volume fraction of 0.6. This wide variation of volume fraction suggests that perhaps significant stress variations may also occur in the the microstructure such that homogenization of stresses even at the scale of the lamina thickness may not be appropriate.

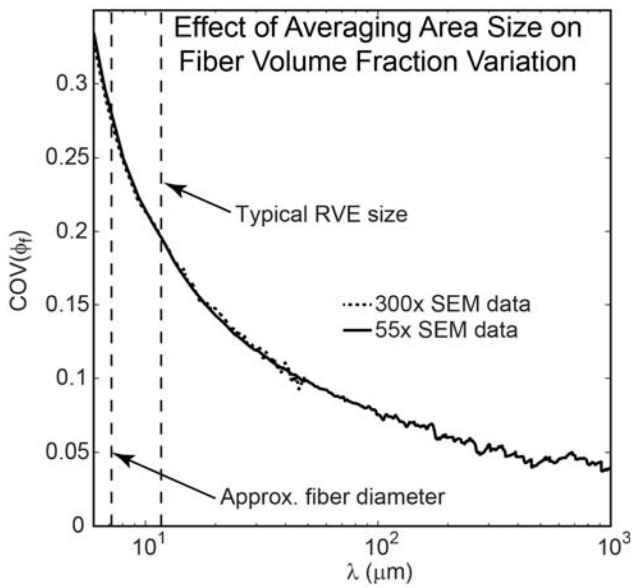

Figure 2. Effect of sampling cell size l on fiber volume fraction variation.

B. Finite Element Analysis of Digitized Microstructure

To investigate the effect of volume fraction variations on composite stresses at multiple length scales, the digitized fiber-matrix grid for the 300x SEM image (Fig. 1 (right)) was translated to a voxel cell finite element model using Abaqus[20]. Nominal properties of epoxy and carbon fiber were selected for the constituent materials. Because this image represents a region of material internal to the lamina, planar boundary conditions were enforced on all faces to ensure tileability of the image (although not true periodicity). A transverse load of 76.8 MPa in the 2-direction was applied to the cell and the resulting stresses were examined. Figure 3 shows the transverse normal stresses in the loading direction. Qualitatively, Figure 3 shows that stresses appear to vary not only from fiber to matrix, but also across much larger regions. Regions of high fiber density show higher stresses than regions of low fiber density.

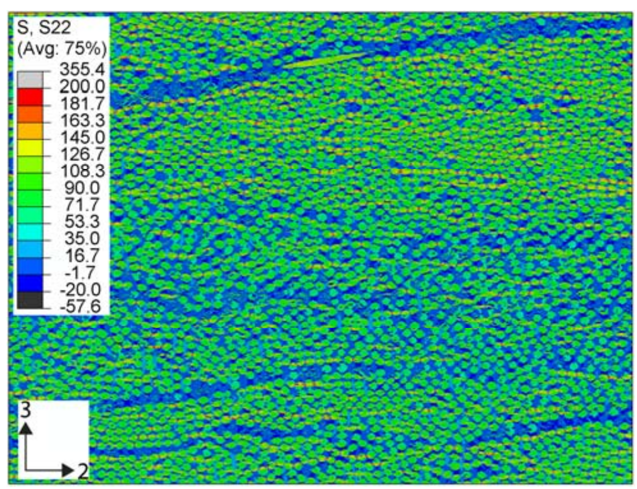

Figure 3. Simulated S22 (transverse) stresses arising from a transverse loading in the 2-direction in the composite.

Of particular interest in this study was whether the deviation from the average stress behaved in a similar manner to the fiber volume fraction shown in Fig. 2. As with the volume fraction, average composite stresses were computed in local square cell regions with varying edge length λ. The standard deviation between cells of a given length is shown via the COV in Figure 4 for the transverse composite stress σ_{22}, which corresponds to the stress in the loading direction. Again, two features are readily apparent. First, at a typical RVE size of 10μm, the transverse stress variation is nearly 15%, which is significant. Second, the general shape of the COV is similar to the volume fraction, which suggests that even at long length scales a homogenized stress state may not be acheived.

The data shown in Figs. 2 and 4 only indicate that variations in volume fraction and variations in stress exist and are strongly dependent on length scale. To investigate whether stress variations are primarily caused by volume fraction variations, the correlation between volume fraction variation and composite stress variation was computed. Figure 5 shows this correlation as a function of length scale for each significant stress component in transverse loading (values of σ_{12} and σ_{13}) were negligible. The most important feature of these data is that correlation of the primary stress component σ_{22} increases with increasing length scale but doesn't reach unity—the correlation approximately saturates at a length scale of about 50 μm and a correlation of about 0.75. Above this critical length scale, volume fraction variation is expected to correlate well with stress. Additionally, the correlation is weakest where the variations in both volume fraction and stress are the largest, namely, at the smallest length scales.

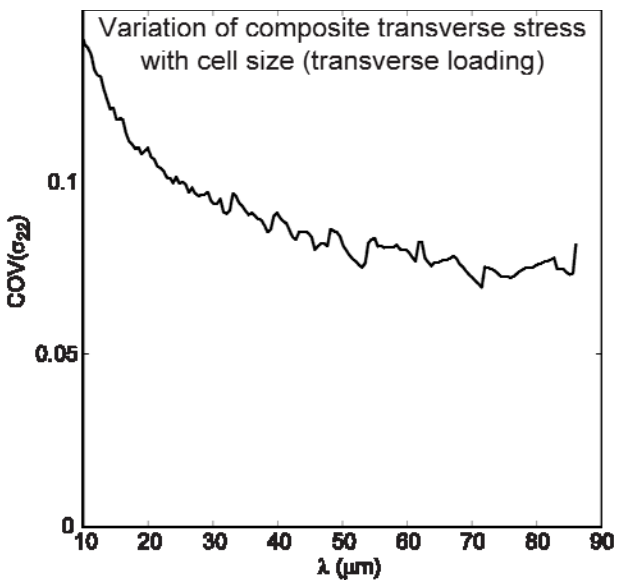

Figure 4. Variation of composite transverse stress with cell size.

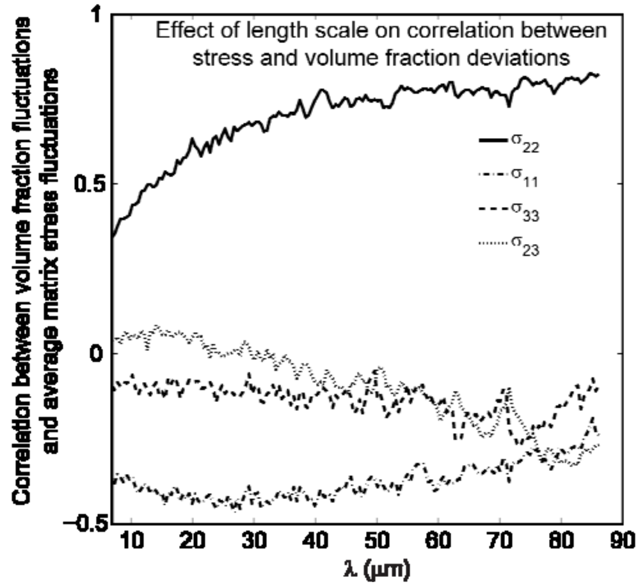

Figure 5. Variation of composite transverse stress with cell size.

C. Stochastic Multiscale Failure Simulation

The results presented in the previous two sections suggest that modeling a structure even as simple as a compressive specimen as a homogeneous material is not correct. But standard approaches of randomly seeding strengths or stiffnesses do not provide realistic insight into any potential scatter in measured structural response. The approach proposed here is to randomly seed an *entire microstructure* with is corresponding stiffnesses and strengths, but to seed it with the consideration of mesh size and volume fraction distributions computed directly from SEM images. Although here, only volume fraction distribution is used to develop the seeded microstructure, this method would enable direct seeding of defects such as voids, stitching points, or alignment fibers. Thus, a defect sensitive multiscale stochastic simulation is proposed. The ultimate goals of such a simulation are to predict variation in failure paths and scatter in material or structural properties, and provide a direct link between microstructural features and reliability of a structure.

To demonstrate this approach, a finite element model of a gage section of a compression specimen was created in Abaqus. Reduced integration linear brick elements (C3D8R) were used to model an internal slice of the gage cross-section with dimensions 2 mm × 9.525 mm × 50 μm. The longest dimension of the specimen corresponded to a transverse direction (2-direction) and the direction perpendicular to the plane of the slice corresponded to the longitudinal (1-direction). All elements were perfect 50 μm cubes, corresponding to the critical length scale computed for correlation between volume fraction and stress. A compressive load was applied in the 2-direction to simulate transverse compression. Surfaces perpendicular to the 3-direction were unconstrained; surfaces perpendicular to the 1-direction were constrained to remain planar.

Based on the SEM analysis discussed above, the fiber volume fraction of the material was selected to be a random variable in the FE model. For this analysis, a mean volume fraction of 0.593 was selected with a standard deviation of 0.0445. The fiber volume fraction was assumed to be normally distributed and this distribution was

Table 1. Material properties for extreme and mean fiber volume fractions

Fiber volume fraction	0.46	0.59	0.73
E_{11} (GPa)	105	134	163
$E_{22} = E_{33}$ (GPa)	7490	9110	11200
$G_{12} = G_{13}$ (GPa)	2880	3870	5560
$\nu_{12} = \nu_{13}$	0.35	0.33	0.31
ν_{23}	0.59	0.54	0.47

discretized into 21 separate equally sized bins. The extreme values for the bins were 0.46 and 0.73, corresponding to three standard deviations from the mean. Each element was assigned a fiber volume fraction based on sampling from this discrete distribution. The properties of the composite corresponding to each volume fraction were computed using a micromechanical model of the fiber and matrix in a periodic hexagonal fiber packing arrangement. The properties from the extreme values and the mean are given in Table 1. Although the strengths would certainly change with changing volume fraction, for the purposes of demonstrating the feasibility and utility of a stochastically seeded microstructure, the strength values for all volume fraction were held constant: $S_{22}^{+} = 45\,\mathrm{MPa}$, $S_{22}^{-} = -145\,\mathrm{MPa}$, $S_{12} = 37\,\mathrm{MPa}$, and $S_{23} = 15\,\mathrm{MPa}$. With the exception of S_{23}, these values were measured experimentally from tests of the material described above. (A typical compression failure is shown in Fig. 6.) Progressive failure was implemented using Autodesk Simulation Composite Analysis[21], an add-on for Abaqus, which uses average constitutive stresses to drive constituent-specific failure theories[2]. When failure of a particular constituent is detected, the constituent stiffness values are degraded discretely. In this study, matrix constituent stiffnesses were isotropically degraded by 90% when failure was determined to occur.

Figure 6. Typical compression failure.

Eight replicates of the distribution were simulated and their failure was examined. Figure 7 shows the normal strain distribution in the loading direction for each of the replicates immediately after failure. Note that each replicate shows a different failure morphology, although all indicate shear failure. Furthermore, these results show that, as expected, strain distribution is not uniform in the sample as a result of volume fraction variation. Although these results are typical of those observed experimentally, they are not typical of a finite element simulation. If homogeneous properties are assigned, the stress distribution is uniform and all elements fail simultaneously, giving no indication of the failure path. Furthermore, in a non-stochastic simulation the failure will always occur in the same way at the exact same loading each time an analysis is run. In contrast, simulation using a stochastic microstructure distribution as shown here shows an essentially infinite spectrum of failure morphologies and shows scatter in predicted strengths. For the simulation reported here, the eight replicates had a mean compressive strength of -46 MPa with a standard deviation of 1.42 MPa (~3.1%). This is in excellent agreement with experimental results from six replicates, which gave a standard deviation of ~3%. The mean strength value for these simulations is artificially low because of the fictitious value for S_{23} that was given—testing is currently underway to quantify this value. Nevertheless, the mode of failure is

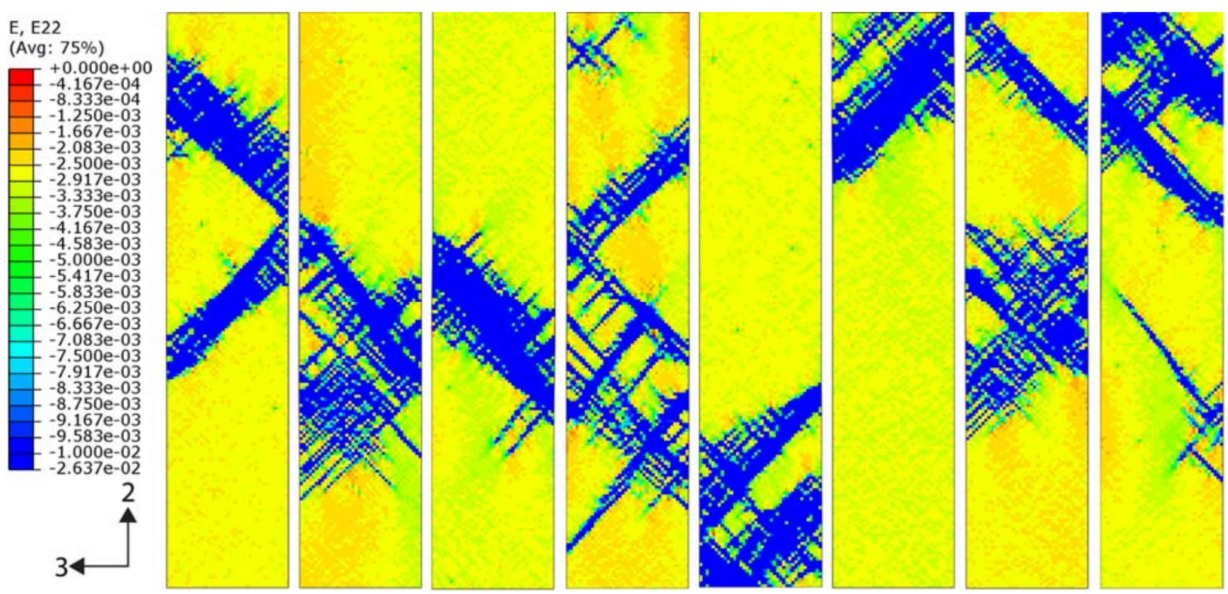

Figure 7. Failure of eight different random replicates with identical fiber volume fraction distributions.

correct, as is shown in Fig. 6, where a typical 45° shear failure is observed.

IV. Discussion

The SEM image analysis has shown that significant volume fraction deviations exist in the microstructure even at length scales on the order of the ply thickness. This result calls into question the efficacy of using an RVE, which has a length scale on the order of 10 μm, to predict behavior in the lamina. Because failure typically initiates at the weakest point in the material, this result also suggests that typical microstructural studies cannot accurately predict composite strength on the basis of bulk constituent properties. The solution proposed in this paper was to *statistically seed microstructures* in individual elements to capture the effect of volume fraction variation on strength properties of a compression coupon. The simulation data categorically gave a lower strength value than the measured value, suggesting that bulk composite strength data does not accurately measure microstructural strengths, which must be considerably higher. However, the stochastic model proposed would enable the relationship between microstructural strength and composite strength to be established.

To the authors' knowledge, this work represents the first effort to link microstructure statistics with stochastic macroscopic response based on random microstructures. The use of volume fraction in this work was illustrative, but the methodology is not limited to microstructures based on volume fraction variations. Microstructures based on known defects such as voids could also be incorporated into the modeling to assess their effect on distribution of structural response. One feature of the data suggests that other variations or defects likely do play a role in failure—namely that correlation between average stress and average volume fraction was ~0.75. Thus, other microstructural variations, such as fiber spacing variation, likely contribute to variation in composite stress. These other variations or defects may be more important than volume fraction alone because they may give rise to sharper stress concentrations in the microstructure that could cause failure to initiate earlier in the loading.

Although these results dramatically expand the potential of the FEA method, a few caveats regarding the modeling conducted in this work are in order. First, the absolute values of the distributions will be dependent on the particular failure theory and progressive damage scheme used locally. In this work, the Autodesk Simulation Composite Analysis defaults were used. These provide a good starting point, but failure initiation and evolution methodologies need to be further developed with microstructural failure mechanisms in mind. Second, microstructure seeding was completely random in this work; no correlations between adjacent regions were considered. These correlations are currently being investigated. Third, a normal distribution was assumed to describe the volume fraction. More SEM images need to be analyzed to assess the validity of this assumption. Finally, the strength values were held constant for all random microstructures, which is not physically realistic. Appropriate incorporation of strength variation would lead to larger scatter in the structural response.

V. Conclusion

This work has shown that microstructural variations, specifically volume fraction variations, lead to significant stress variations in the composite. These variations strongly depend on length scales that range over three orders of magnitude from 1 μm to 1 mm. This work also demonstrated that real microstructural data can be used to develop a stochastic structural simulation that can predict variation and distribution of a macroscopic structural response. These analyses represent the first step towards predicting structural reliability on the basis of the physics of microstructural defects. This would enable virtual experiments to be conducted via stochastic simulation to establish the effect of manufacturing variability in composite materials on application-specific reliability.

Acknowledgments

This work was funded by Wyoming NASA Space Grant Consortium, NASA Grant #NNX10A095H.

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