MICRO-SCALE STRAIN MAPPING IN NANO-ENGINEERED FIBER-REINFORCED COMPOSITES

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Abstract
Full-field strain measurement in materials that exhibit mechanical heterogeneity at the micro-scale are of great interest. This is particularly important in the emerging field of nano-engineered fiber-reinforced composites, where the effects of nano-modifications are as-yet uncharacterized. Formerly, the authors proposed a methodology to analyze deformations in conventional laminated composites at the micro-scale via digital image correlation (DIC). In the present study, this technique is applied to capture strain heterogeneity in microfiber-reinforced laminated composites that are additionally modified with aligned carbon nanotubes (CNTs), grown radially on microfibers as aligned forests. The composite is loaded in transverse three-point bending inside an environmental scanning electron microscope. The paper describes acquisition of 2D displacement and strain maps, and investigates the effect of CNTs on the deformation of these hierarchical architectures. As a prerequisite for micro-DIC, a high-quality nano-scale random speckle pattern of alumina particles is deposited on the surface. A finite element model of the micro- and nano-structure geometry, with boundary conditions obtained from microscopy and DIC analysis. A good correlation between experimental and modeling results was obtained, indicating that the micro-scale DIC is a promising technique to study deformation in nano-engineered composites.

1. Introduction

Digital image correlation (DIC) is an optical method to track the changes in a set of consecutive images of a deforming body. Using the random speckle pattern on the surface of the material, DIC creates displacement and strain maps for each step of deformation [1]. The scale of DIC is determined by the length scale of the input images; hence, if micrographs are to be studied, the analysis is micro-DIC, which is of great interest for materials with heterogeneous microstructure. Micro-DIC confronts challenges such as creation of a high-quality and small-scale random speckle pattern [2]. It was shown that micro-DIC is a useful technique in the deformation analysis of fiber-reinforced composites [3, 4]. This methodology is also promising in the investigation of an emerging class of materials, nano-

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engineered fiber reinforced composites. These materials hold a high level of structural heterogeneity at the micro-scale. The effect of nano-reinforcement on deformation and damage development at the micro-scale can be determined by means of micro-DIC.

In the present study, the applicability of micro-DIC to the nano-engineered fiber-reinforced composites for identification of micro-features and micro-phenomena is explored. An alumina fiber/epoxy composite with carbon nanotube (CNT) grafted fibers is speckled with a nano-powder and loaded under transverse 3-point bending inside an environmental scanning electron microscope (ESEM). The micrographs of the deforming composite are correlated, resulting in the identification of micro-features such as fibers, CNT forests, and debonding sites at the fiber/matrix interface in the strain maps. A finite element (FE) model of the area of interest is developed, simulating the CNTs grown on fibers and prescribing the boundary conditions from the experiment. The results of both methods are presented and discussed.

2. Materials and methods

2.1. Composite material

The study is performed on a nano-engineered composite. The reinforcement is alumina micro-fibers with aligned CNTs grafted on them. The Alumina micro-fibers are in a plain weave configuration *(Cotronics Ultra-Temp 391*). The CNTs are grown on fibers by aid of *Fe* catalyst, and acquire a length of 17-19 μm. After CNT growth, a stack of five fabric plies was infused with the *RTM-6* resin (*Hexcel*) using vacuum assisted infusion. The laminate was then cured and post-cured.

2.2. Micro-DIC methodology

The micro-DIC methodology includes several steps: specimen preparation, speckle patterning, mechanical loading, image acquisition, optimization of DIC parameters, application of DIC, error analysis, and validation versus finite element analysis (FEA) [3]. A 58.3 mm × 8.2 mm × 3.1 mm specimen is cut from the laminate and one of the two cross-sections is grinded and polished. For creation of a well dispersed and distributed random speckle pattern, a 0.05 wt.% water-based suspension of alumina particles (*TM-DAR series of TAIMICRON*), containing 0.005 wt.% *DARVAN-C-N*, is prepared using ultrasonication and magnetic stirring. The suspension is applied twice to the polished surface of the specimen. The speckled surface is coated with a thin film of gold for eliminating the charging effect during microscopy.
The speckled specimen is then mounted in an *in-situ* micro-test stage (*Deben UK Limited*), installed inside an ESEM chamber (*FEI/Philips XL30 ESEM FEG*) for 3-point bending. A suitable microscopic field of view including almost isolated fibers with radially aligned CNT forests on them is found in the middle of the tensile edge of the specimen (Figure 1a). Transverse 3-point bending is applied to the specimen with a rate of 0.2 mm/min. The loading is stopped every 30 seconds for image acquisition. In total, 11 micrographs are captured with the resolution of 1424 × 968 pixel^2 (103 × 70 μm^2) at 1200× magnification. The final global load is 79.9 N, corresponding to a 1.2 mm maximum deflection.

The subset, step, and filter sizes in DIC influence the accuracy of the results. Therefore, they are selected such as to obtain the highest resolution with the lowest error. A so-called *strain deviation analysis*, explained in [3], is used for optimization of the DIC parameters: the length of the reference micrograph is increased virtually in *MATLAB* for 12 pixels, which is equivalent to a virtual deformation of 0.008462 for the horizontal component of the Lagrangian strain. DIC with different sets of parameters (called collectively "correlation parameters") is performed on the resized image in *VIC-2D 2009* software (*Correlated Solutions*). The examined subset sizes are 41, 61, 81, and 101 pixels, step sizes are 1, 2, 5, and 7 pixels, and filter sizes are 5, 15, and 25 data points. The resulting horizontal strain average is close to the virtually applied strain (0.008462). It is observed that with increase of the correlation parameters

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the standard deviation decreases, which means reduction in measurement error. At the same time, the increase in the correlation parameter decreases the analysis resolution. Therefore, a trade-off should be considered. Based on the results of the strain deviation analysis, the optimized values for subset, step, and filter sizes are 61 pixels (4.42 µm), 2 pixels (0.14 µm), and 15 data points (2.17 µm), respectively.

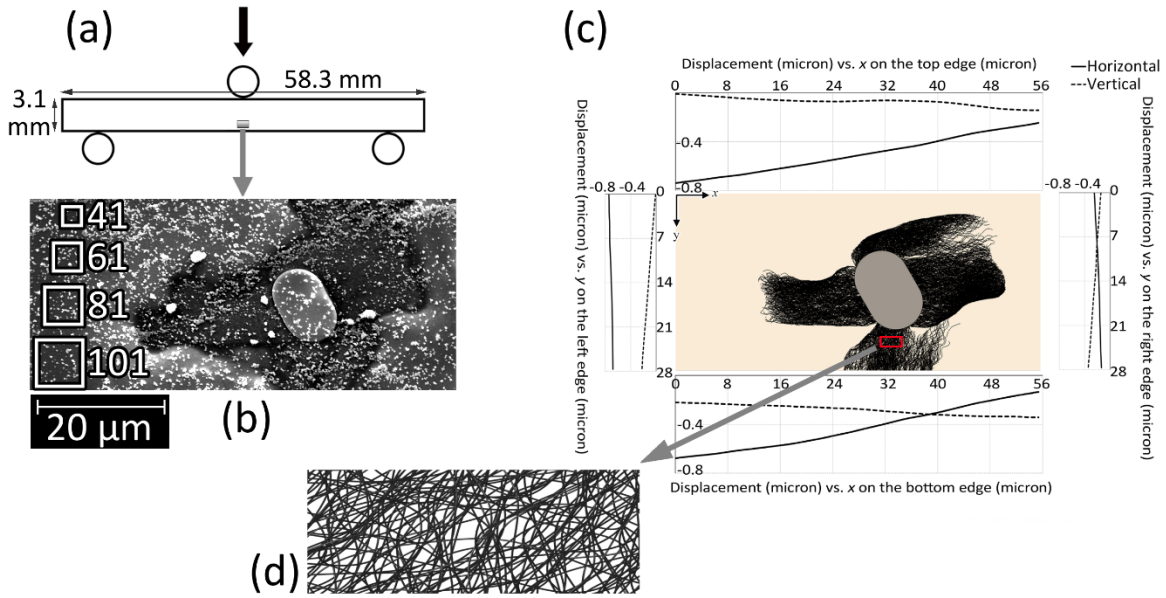

Figure 1. The study zone chosen for strain mapping, shown (a) schematically on the specimen in three-point bending and (b) at the working magnification (1200x) with the examined subsets on it; (c) FE model of the study zone simulating CNTs grown on the surface of the fiber, with the DIC-measured displacements profiles on each edge of the study zone (d) zoom-in configuration of a CNT forest region, marked with the red window in (c).

The 2D DIC can be disturbed by two types of errors: physical- and correlation-based [5]. In the present study, physical-based errors are referred to as microscopy error and estimated through DIC analysis of rigid-body micrographs. This analysis revealed that the maximum values for mean strain, standard deviation, and local strain resulting from microscopy error are 0.000299, 0.000626, and 0.001820, respectively. On the other hand, the correlation-based errors are referred to as measurement error and are estimated with the standard deviation analysis performed above for parameter optimization. It was concluded that the difference between the calculated mean strain and the applied strain is 0.000014, the resulting standard deviation is 0.000091, and the maximum local strain deviation from the applied strain is 0.000600, corresponding to the measurement error. The low values for microscopy and measurement errors ensure a high quality DIC analysis in this study. For DIC, a study zone (55.5 × 27.6 µm^2) containing one fiber with grafted CNTs is selected as the area of interest (Figure 1b). DIC with the optimized parameters is applied to the 11 micrographs.

3. Numerical model

To explore the accuracy of micro-DIC in identification of micro-features and micro-phenomena, a FEA was conducted. A model with the geometry of the study zone microstructure is developed (55.5 × 27.6 × 0.5 µm^3). The two constituents are modeled as isotropic bodies with linear elastic behavior. CNTs are modeled as hollow curved cylindrical shells with the orientation, spatial distribution, and curviness extracted from high- magnification micrographs. Centerlines of nanotubes were generated as sequences of vertices forming quasi-helical curves with multiple perversions. Then, cylindrical hollow CNTs were

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created by performing a shell sweep of a circle with radius of 9 nm along these curves. To discretize nanotubes, 4-node general-purpose conventional shell elements are chosen with the outer surface of the nanotube used as a reference surface. Matrix is discretized with 8-node brick elements with the size of the matrix element equal to $5R_{CNT}$, in the zone, where nanotubes are located. R_{CNT} is the outer radius of a nanotube. The FE model and a zoom-in view of the simulated CNTs are displayed in Figure 1c and d.

According to the experimental evidence, the matrix/fiber interface of the analyzed fiber was initially debonded. Partial debonding on the right and left side of the fiber was modeled as traction free regions. Length of the debonding was fixed during simulations and its propagation was not considered. For the remaining interface, a perfect bonding was assumed between the matrix and the fiber. The model is based on the two-scale approach proposed in [6]. The embedded element technique is used to constrain the translational degrees of freedom of the nanotube nodes to the interpolated values of the corresponding degrees of freedom of the appropriate matrix elements. The 3D FE continuum model was subjected to horizontal and vertical displacements measured by micro-DIC on the outer edges of the study zone (Figure 1c). In the thickness direction, periodical boundary conditions were applied.

4. Results and discussion

In this section, only the results regarding the last (10th) deformation step are presented. In the DIC maps, the edges of the alumina fibers and CNT forests are marked respectively by solid and dashed lines. The resulting displacement maps from DIC and FEA are plotted in Figure 2a and b. It is observed in the DIC map that the study zone is horizontally stretched. The displacement fronts are distorted by the fiber due to its different stiffness. The CNT forests also influence the displacement fronts. Comparing the DIC and FEA maps, DIC captures well the micro-scale heterogeneity of the material through the displacement.

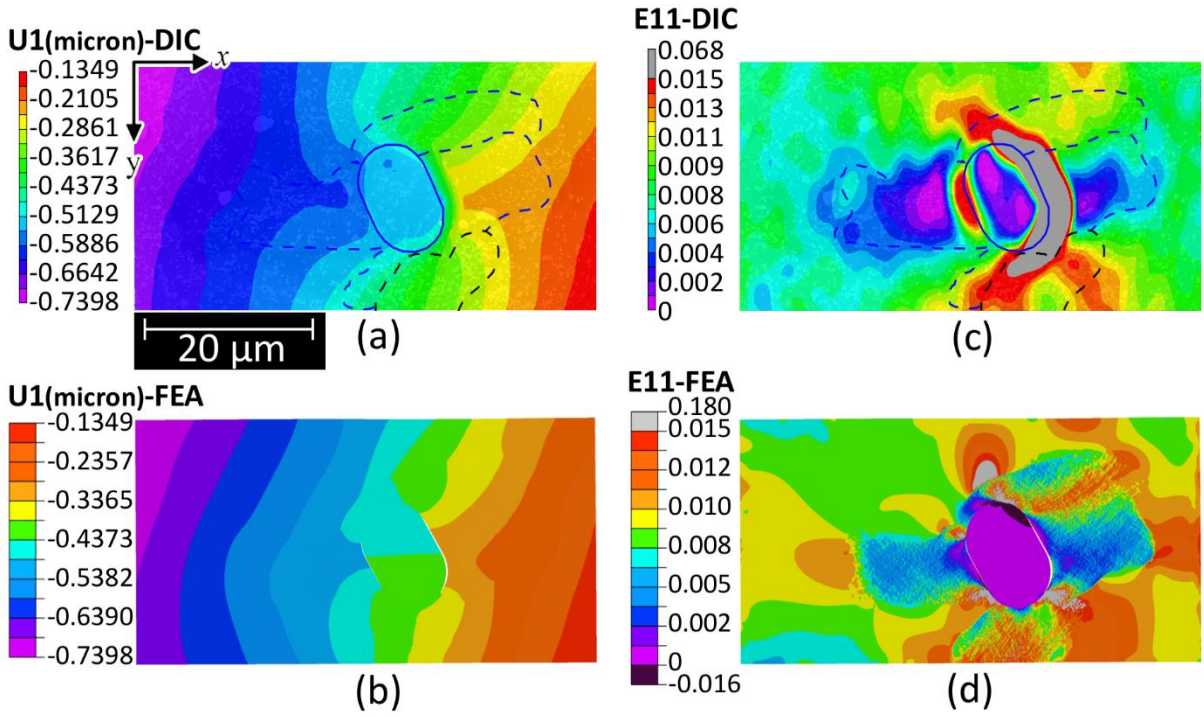

Figure 2. displacement (a,b) and strain (c,d) maps measured by DIC and predicted by FEA.

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Strain maps resulting from DIC and FEA are displayed in Figure 2c and d. High values of strain are observed around the fiber, corresponding to fiber/matrix interfacial debonding. To reveal other microstructure features, the upper limit of the strain scale is reduced to 0.015. The strains above this limit, corresponding to the debonding, are shown in gray color. The horizontal strain is positive allover the matrix, as it should be under horizontal tension (Figure 2c). Fibers experience lower strains because of their higher stiffness. CNT forests that are aligned in the loading direction also show strains lower than the strains in the matrix. This is because CNTs constrain tensile deformation in the longitudinal direction. However, CNT forests that are aligned perpendicular to the loading direction do not constrain the strain since the transverse properties of CNT forests are dominated by the matrix behavior. There is a general agreement between the strain maps resulted from DIC and FEA.

5. Conclusions

The need for a tool to analyze the deformation of nano-engineered fiber-reinforced composites has existed since the effects of nano-modifications became a point of interest. Micro-DIC proved to be a promising technique fulfilling this need. It requires application of a high quality nano-scale random speckle pattern, optimum DIC parameters, and proper microscopy setting. We have demonstrated on an example of alumina/epoxy composite with aligned CNTs grafted to the fibers and under transverse 3-point bending, that micro-DIC could identify the micro-scale features such as fiber, CNT forests, and fiber/matrix debonding. The results are in a good agreement with those of a finite element model, simulating the microstructure incorporating the CNTs.

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