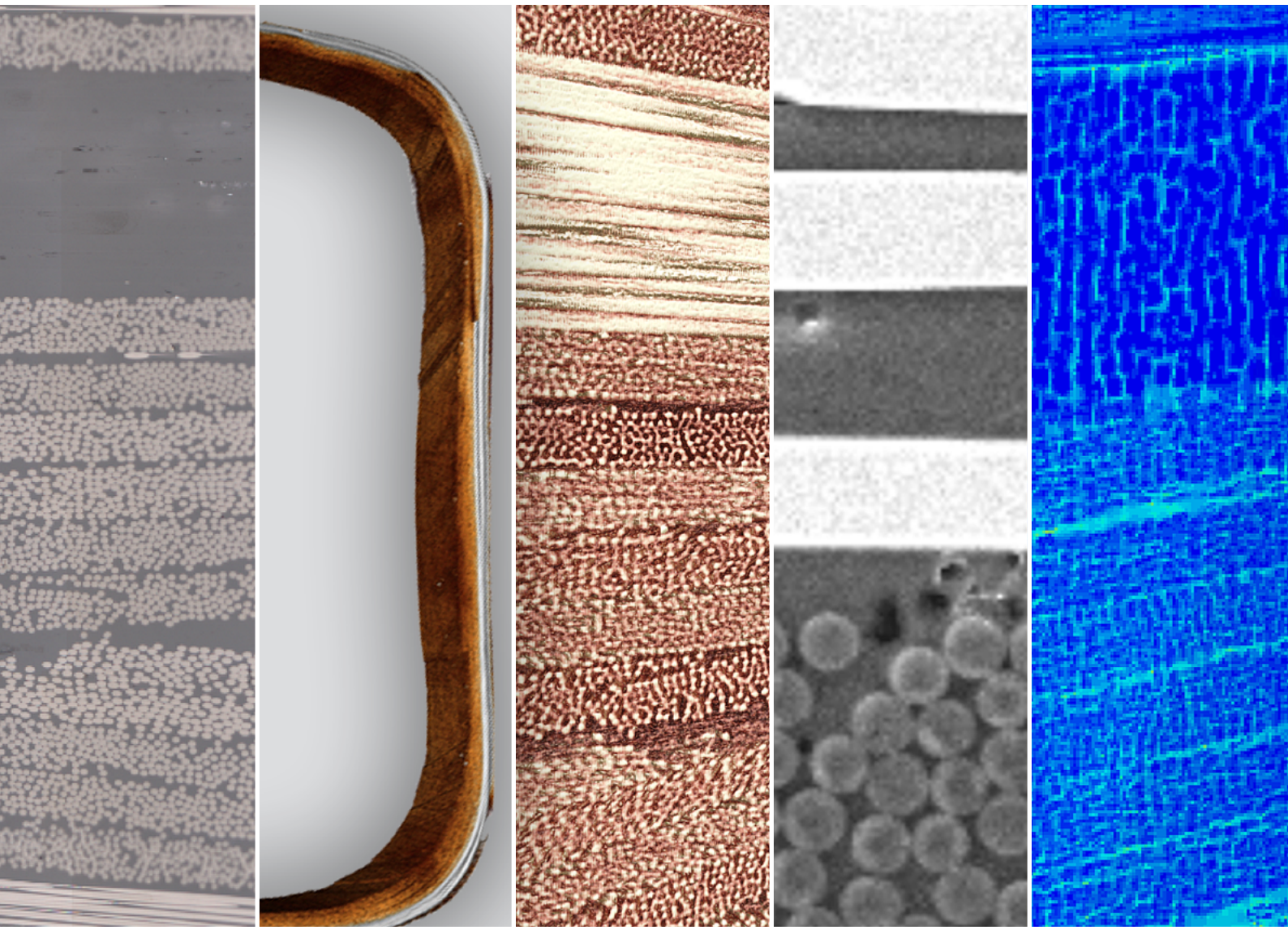
Application Note



Investigating Structure-property Relationships in a Carbon-fiber Composite

ZEISS Correlative Microscopy

Investigating Structure-property Relationships in a Carbon-fiber Composite

ZEISS Correlative Microscopy

Author: Carl Zeiss X-ray Microscopy, Inc.

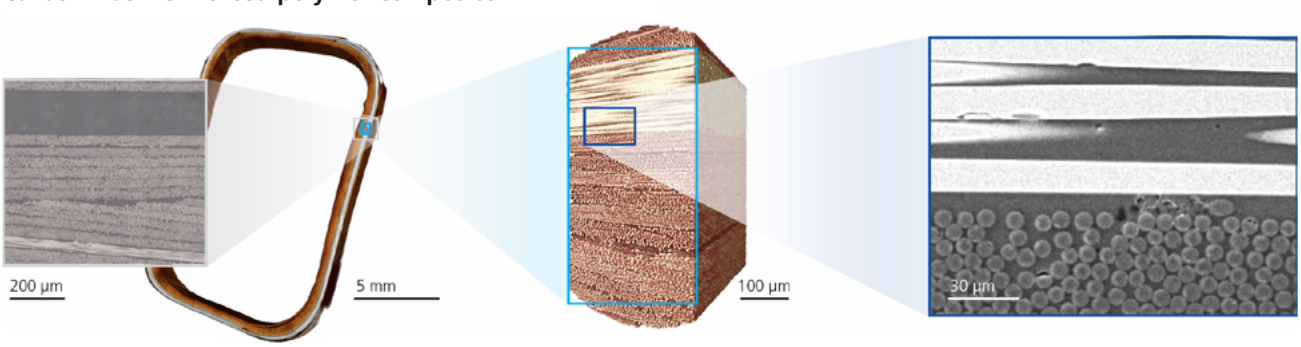
Date: January 2019

In many applications, anisotropy has become the key to designing a successful product [1]. Athletic applications commonly employ composite materials, to impart flexural properties in one load direction, while maintaining a light weight and tough material that resists fracture. The typical service conditions of these devices result in hundreds to thousands of fatigue loads to each material, and thus it is important to understand the nature of fracture under such extreme conditions.

Characterizing composite materials; however, is a challenging task. Failures often nucleate inside the structure and are often unobservable until fracture is reached. Understanding the nucleation processes is critical toward engineering against failure, but traditional bulk testing methods are insufficient to describe this process. Therefore, materials design protocols have typically consisted of an iterative loop between design, testing, and property observation.

To mitigate the challenges in characterization and improve design efficiencies, researchers are increasingly turning toward microscopy as a complimentary technique to bulk mechanical testing. A material may be imaged once as manufactured, from which the mechanical properties can be predicted through simulation (and compared to actual bulk testing results); subsequently, the design may be modified in virtual space until the simulation engine reports the desired results, then the new material may be formed in line with this design. This technique of "digital material testing" has grown in popularity in recent years, as the simulation efficiencies have improved to the point of being able to run on standalone computer workstations. The remaining challenge is that of scale, ensuring that the appropriate information is captured from the micro- to the macro-scale, to build a virtual multi-scale model accurately representing the real material structure for accurate property predictions.

Carbon fiber reinforced polymer composite



Light microscope *X-ray microscope* *Scanning electron microscope*

In the present study, the technique of correlative microscopy is presented as a viable conduit into the digital material testing approach. A carbon fiber reinforced composite hockey stick was used as the subject of the characterization study, though this same technique can apply to any variety of materials, from glass composites to metal matrix composites, as well as to monolithic materials. The traditional approach of optical microscopy is presented in the initial work, which conforms to standard testing approaches and sets the stage for further analysis. The specimen was then transferred into the X-ray microscope (XRM) for non-destructive multi-scale 3D characterization, providing microstructural information from the tens to single micrometers. Finally, the specimen was transferred into the scanning electron microscope (SEM) for nano-scale characterization as well as compositional analysis with energy dispersive X-ray spectrometry (EDS). All of the data were collected in the exact same region of interest (ROI), using the correlative microscopy workspace of ZEISS Atlas 5. The result of this study was, thus, a multi-scale model of the material's structure and composition, which was subsequently used as input into a simulation routine that predicted the material's mechanical properties.

This correlative microscopy approach represents a paradigm shift in material characterization, bridging the physical world into virtual space. Coupling imaging with modeling and simulation is a powerful approach to characterization, providing increased insight into material systems and, in turn, increasing design efficiencies for those who utilize the techniques.

Specimen Preparation

A small section was cut from the bulk product using a diamond saw from Allied High Tech. The section was then embedded in resin and polished using a MetPrep 3 equipped with PH-3 power head, prepared in accordance with ASTM E3-11[2], in order to enable high-resolution optical and electron microscopy. Figure 1 shows a graphical representation of the specimen preparation process. After polishing, some of the resin on the interior of the stick was additionally removed, to facilitate X-ray transmission in the subsequent X-ray microscopy investigations.

Optical Microscopy

Optical inspections were performed using ZEISS Axio Imager 2, as shown in Figure 2 below. A 5X objective was initially used to survey the entire specimen surface (using an automated mosaic imaging & stitching routine), followed by a 50X objective to highlight the smaller features of the material microstructure. Results from the optical inspection are shown in Figure 3, where the reinforcing fibers can be clearly distinguished from the matrix and some small defects (voids) were observed.

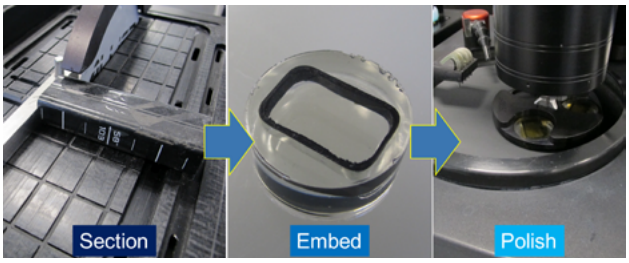


Figure 1 *The preparation procedure began with sectioning a small specimen. This piece was then embedded in resin and polished to a smooth surface finish. Preparation courtesy of George Abraham, Allied High Tech.*

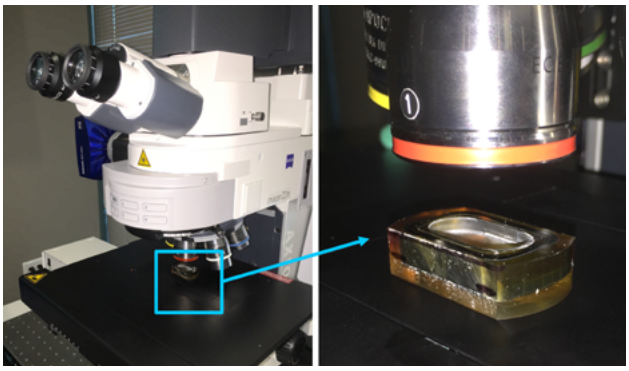


Figure 2 *Imaging setup with ZEISS Axio Imager 2 high-resolution optical microscope.*

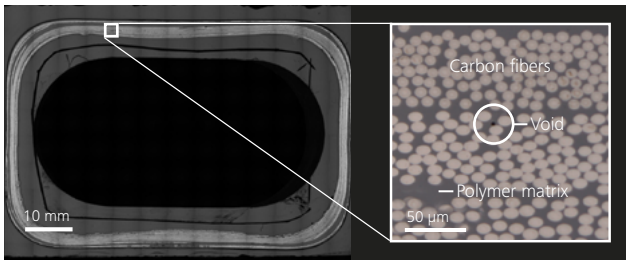


Figure 3 *Optical micrographs, using a 5X (left) and 50X (right) objective. The results provided a detailed view of the 2D microstructure, as well as some observed defects (circled).*

By applying a digital binarization to the optical micrographs with ZEISS ZEN 2 Core, a quantitative analysis of the optical data was realized. The results yielded a 54% areal fraction of carbon fibers (46% resin), as well as fiber shape distributions (Feret min, Feret max, Feret ratio, and roundness) and a fiber cross-sectional area distribution. This data analysis is shown in Figures 4 and 5.

While the data provided an excellent high-resolution view into the specimen, the imaging and data analysis were limited to a single 2D cross-section, which elucidated only one datapoint on the overall material microstructure. One approach to upscaling this into the bulk device would be to take the metrics reported in Figure 4 and use them to build up a phenomenological model representative of the same overall structure as the real material. However, a more rigorous approach is to also incorporate additional imaging techniques, as shown in the following sections, to generate a more direct and explicit conformal structural model of the sample which can then be used for modelling purposes.

X-ray Microscopy

In extending the 2D imaging to 3D, it is important to ensure that the same ROI is imaged to keep the datasets in the appropriate context (as compared to each other). In the present study, ZEISS Xradia Versa was used for 3D X-ray microscopy, and the flat panel extension (FPX detector) was initially employed to survey the bulk microstructure. This result was aligned to the 5X optical micrographs using ZEISS Atlas 5, so that high-resolution interior tomographies could be aligned to the high-resolution optical micrographs. Using this procedure, high-resolution X-ray and optical data could be placed side by side, as in Figure 6, so the X-ray microscopy results effectively became a 3D extension to the initial 2D survey.

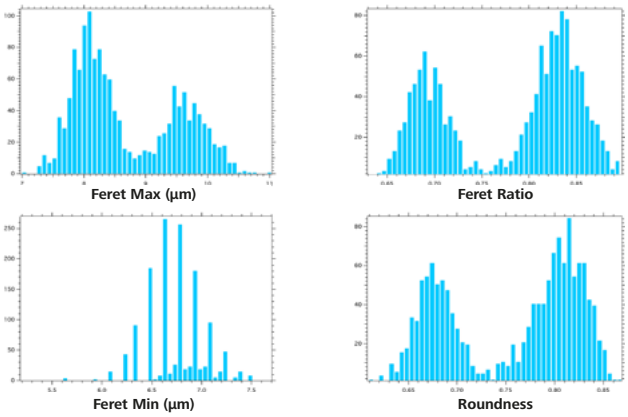


Figure 4 *Fiber shape distribution information, as revealed by optical microscopy with a 50X objective (see Figure 3). The apparent bimodal distribution was believed to be caused by different fiber orientations (producing different cross-sectional faces), which is later confirmed with 3D X-ray microscopy.*

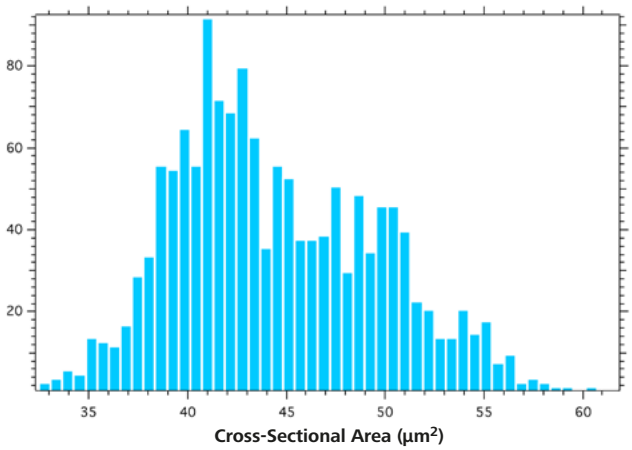


Figure 5 *Fiber size distribution information, as revealed by optical microscopy.*

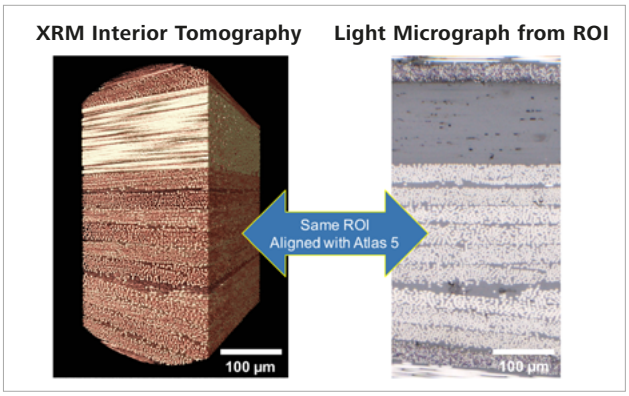


Figure 6 *Comparison of 3D X-ray (left) and 2D optical (right) micrographs of the same region of the same specimen. Correlative microscopy was facilitated by Atlas 5.*

As previously mentioned, X-ray microscopy was performed at two different length scales to build a multi-scale model of the 3D microstructure. The overview imaging was performed using a voxel size of 12 µm, while the high-resolution imaging was performed with a voxel size of 1 µm. All X-ray data was collected non-destructively, so the specimen was not sectioned or otherwise disrupted during the 3D image acquisition, at either the coarse or fine length scale. This preservation of the specimen also potentially allowed for further 4D characterization, for example performing load experiments *in situ*, but those studies were reserved for future investigations. A representation of the "interior tomography" results within a spatially-relevant context is shown in Figure 7.

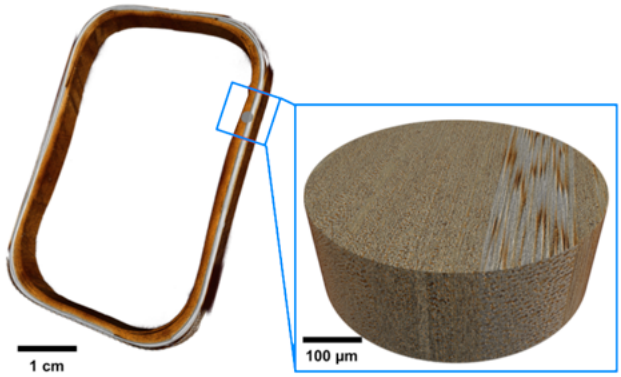


Figure 7: *3D X-ray microscopy is non-destructive, which means that both low- (left) and high- (right) resolution data were collected without sectioning the specimen, preserving the material for future studies. The relationship of the two X-ray datasets to each other is shown here.*

The high-resolution 3D X-ray images confirmed that, indeed, there were multiple layers of fibers in the laminate, with internal voids and delaminations on the order of single to tens of microns. An example virtual slice is shown in Figure 8, where several voids have been measured. It was interesting to note that a bright fibrous region was additionally observed near the specimen's exterior, which was consistent with a localized inorganic composition. Curiosity, as well as the desire for higher-resolution data, were the motivator for further analysis with SEM and EDS.

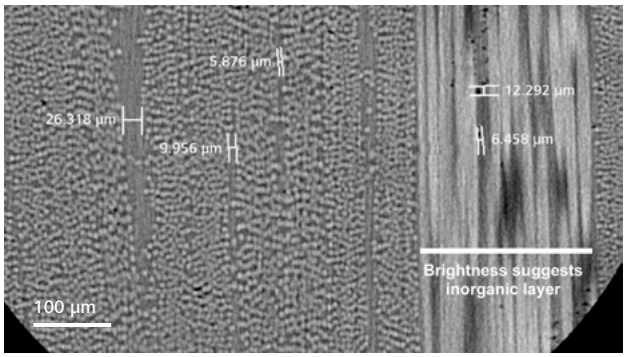


Figure 8: *Example virtual slice from the 3D volume, non-destructively imaged using a voxel size of 1 µm.*

Before proceeding with additional imaging, a digital binarization was applied to the [suspected] carbon-fiber region, to understand the fiber diameter distribution (now independent of fiber orientation). Initial data processing was performed in ORS Dragonfly Pro and the fiber analysis was performed in Math2Market GeoDict. The average fiber diameter, as shown in Figure 9, was found to be 6.5 µm, with a distribution that varied by several microns about the mean.

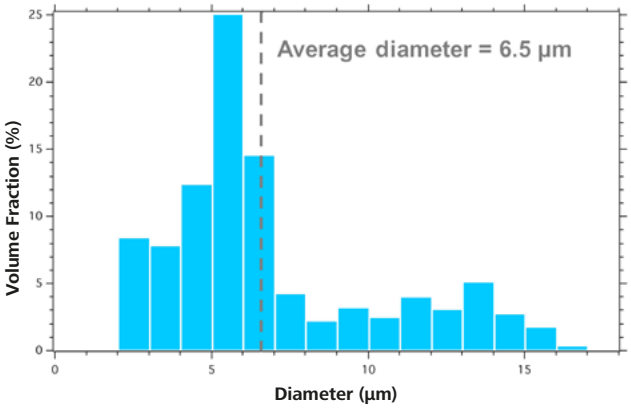


Figure 9: *Carbon fiber diameter distribution, as processed from the 3D X-ray microscopy data.*

Scanning Electron Microscopy

In the final stage of image acquisition, a ZEISS Crossbeam 540 FIB-SEM was used to capture the finest length scale of information, as well as localized composition. ZEISS Atlas 5 was again used to align the microscope to the same ROI as imaged by the optical and X-ray techniques, to retain the context of all data with each other. Figure 10 shows the imaging and analytical results, obtained on the same ROI as imaged with light- and X-ray microscopy. The chemical information, captured by EDS, confirms the presence of two different materials in the fibrous micro-structure, identifying them as carbon and glass fibers.

Correlative Data Analysis and Digital Material Testing

With the identity of the materials now determined and full contextual information of the 2D and 3D microstructure available, a digital analysis was thus possible. This analysis began with a 3D volume fraction computation, as shown in Figure 11, which yielded a measurement of 50% carbon fibers, 35% matrix, and 15% glass fibers (all percentages by volume). Porosity and void fraction were found to account for <1% of the imaged volume, and thus were not reported in the compositional analysis. Furthermore, a slice-by-slice solid volume fraction plot was generated along the planes where optical microscopy was performed, using the 3D approach to indicate what (if any) systematic variations may be introduced through arbitrary sectioning and polishing. These measurements were performed for the imaged volume, which was selected somewhat arbitrarily – for a full characterization of this device, it would be prudent to image other volumes along its length to characterize the larger-scale systematic variations in microstructure. Such a robust characterization was not carried out in the present study since the focus was more on workflow development than understanding one particular device, but researchers implementing this procedure may wish to characterize more regions and repeat this process before reaching a conclusion.

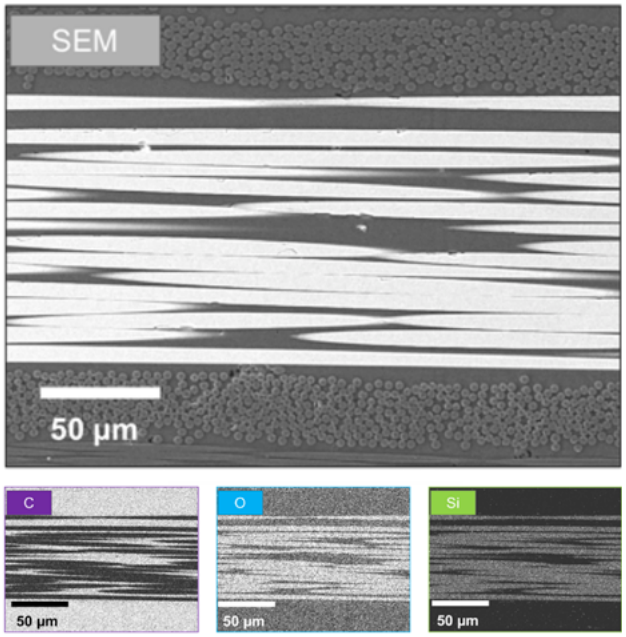


Figure 10: *(top) High-resolution SEM micrograph, confirming the higher-Z layer of fibers amidst the lower-Z fibers. (bottom) EDS maps showing carbon, oxygen, and silicon distributions, indicating a laminated structure of carbon and glass fibers.*

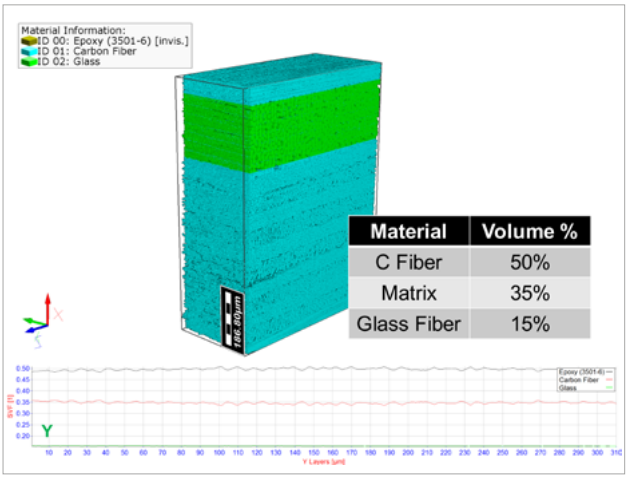


Figure 11: *3D visualization and volume fraction analysis, performed both volumetrically (top) and on a slice-by-slice basis (bottom).*

In analyzing this particular composite material, it was understood *a priori* that the primary failure modes are produced by exceptionally high transverse loads, leading to crack nucleation, propagation, and ultimate fracture. The 3D, correlative dataset was thus loaded with the relevant material properties (obtained via literature values) into the simulation engine of GeoDict and a virtual transverse (XZ) load was applied. From the digital test, the von Mises strain was analyzed in a 3D manner, indicating that the points of highest strain under an extreme "real world" load exist at the fiber-matrix boundaries. Furthermore, the software produced a computational result indicating the mechanical properties of the composite material, including the elastic moduli and Poisson ratio. The results of this analysis are shown in Figure 12, which are in line with the expected properties of a typical carbon fiber composite material [3]. With the properties now calculated for a small region of the material, these results may further be substituted into the larger model (generated by low-resolution XRM) in order to model the flexural behavior of the larger-scale device, but this point will be reserved for future studies.

Summary

In the present study, the technique of correlative microscopy has been effectively used to generate a multi-scale model of a carbon fiber composite material. Optical microscopy provided fast access to microstructural data, showing the fiber and matrix features in both qualitative and quantitative contexts. Correlating these results to those from X-ray microscopy allowed the 2D analysis to extend into 3D, resulting in a 3D model of the microstructure. With the compositional information provided by energy-dispersive X-ray spectroscopy, an accurate virtual depiction of the material was produced, which served as input into the simulation routine for material property predictions.

Correlative microscopy thus enabled a robust imaging-to-simulation workflow, producing a model that is available for further digital modification and analysis. Through implementation of this procedure in a regular basis, material development efficiencies may be enhanced, leading to high-performance products in a reduced amount of time. Furthermore, the results provide a novel insight into the service performance of the material, enhancing understanding of the performance characteristics and failure modes.

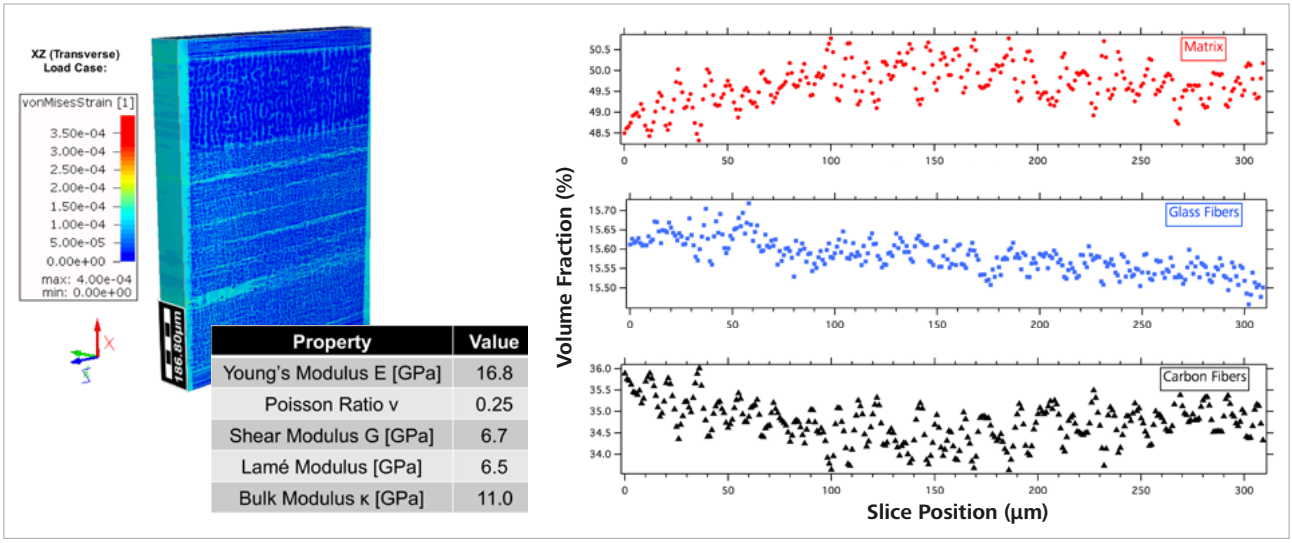


Figure 12: *Analyzing the correlative microscopy results using the GeoDict simulation engine, a von Mises strain within the 3D volume reveals the regions of highest internal strain under a transverse load case. Further simulations performed in the same software package predict the mechanical properties of the composite material.*

References:

[1] Callister, William D. Materials Science and Engineering: An Introduction. 8th ed. New York, NY: John Wiley & Sons, 2010.

[2] ASTM E3-11, Standard Guide for Preparation of Metallographic Specimens, ASTM International, West Conshohocken, PA, 2011, www.astm.org.

[3] ACP Composites. "Mechanical Properties of Carbon Fiber Composite Materials, Fiber / Epoxy Resin." (2014): n. pag. Dec. 2014. Web. Jan. 2017.

Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/materials-science