ATHLETIC COMPOSITES: A STRUCTURE-PROPERTY CASE STUDY USING 3D CORRELATIVE MICROSCOPY

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Summary: Composite materials are widely used in a variety of applications; however, it remains challenging to characterize their properties and performance characteristics using conventional means. Here, we present the technique of correlative microscopy for composite analysis, using data from optical microscopy, XRM, SEM, and EDS to build a multi-scale model of the material microstructure and composition. These results were then loaded into a computational simulation environment, which was used to predict the mechanical properties of the specimen under investigation.

1. INTRODUCTION

Athletic materials are generally engineered with a particular application field in mind. Such a material may need to be anisotropically stiff, in order to provide flex in one direction but rigidity in others, or light weight and impact resistant, depending on the needs of the intended service environment. Monolithic materials often provide limited flexibility in the design, as strong materials may be heavy and stiff materials may lack toughness. In many modern athletic applications, composite materials have become commonplace, as they utilize the most advantageous properties of their components. Composites are typically composed of a dispersed phase, such as reinforcing fibers, bound together by a matrix phase, such as a resin, and may be precisely engineered to provide valuable properties for a particular field. [1]

Characterizing composite behavior, however, may be a challenging task. While a rich history and characterization infrastructure exists using mechanical sectioning and optical microscopy [2], these techniques are limited to studying features along the polished/exposed surface. Failures often nucleate inside the material, which are difficult or impossible to see using conventional 2D optical imaging techniques. To properly understand how a composite material behaves and to predict its performance properties, a more robust approach is needed.

In the context of the present study, a carbon fiber composite hockey stick was investigated using a multi-dimensional, multi-modal correlative microscopy workflow in tandem with computational approaches to digital material testing. The microscopic data collection resulted in a multi-scale model of the material, incorporating information ranging from its overall dimensions to the finest details of the composition and void content. This model was then used as input to a computational simulation protocol, which predicted the mechanical properties of the bulk material. Implementing this technique thus resulted in a model which could be virtually modified, tested, and procedurally iterated, thus developing a structure-property digital testing environment to streamline future designs of new materials. Furthermore, the incorporation of non-destructive XRM in the workflow allows for future specimens to be formed, measured, processed, and measured again, paving the way for "4D" studies on the evolution of the structure-property relationship.

2. EXPERIMENTAL METHOD

In the present study, a correlative microscopy approach was employed, integrating results from optical microscopy, X-ray microscopy (XRM), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS). Specimens from a commercially-sourced hockey stick were sectioned, embedded, and polished, in accordance with ASTM E3-11 [2], to prepare them for both optical microscopy and SEM, while also permitting 3D X-ray imaging. Some additional embedding material was then removed to facilitate X-ray

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transmission in the subsequent XRM investigations without disturbing the polished surface. One prepared specimen was imaged with the ZEISS Axio Imager 2 high-resolution optical microscope, from which a 2D analysis yielded the solid volume fractions (carbon fibers & matrix), fiber area distribution, and fiber shape distribution (feret min/max). The specimen was then transferred to the ZEISS Xradia 520 Versa XRM, using a correlative alignment routine in the ZEISS Atlas 5 software to ensure that the 3D X-ray investigations were performed on the same region as the optical microscopy. A multi-scale approach incorporating high-resolution interior tomography was employed, which showed the solid volume fractions, void content, and fiber size & shape distributions in 3D. Finally, the specimen was transferred to the ZEISS Crossbeam 540 SEM equipped with an Oxford Instruments EDS detector, again aligning to the same region of interest using the Atlas 5 software. In this final step of data acquisition, correlative SEM micrographs and EDS maps were collected to elucidate nano-scale features of the microstructure and the local chemical composition.

When all data had been curated, the results were processed with Object Research Systems Visual Si Advanced and, subsequently, loaded into Math2Market GeoDict 2015 for digital material testing and property prediction. A solid volume fraction analysis was performed on the 3D data, as well as a simulation of the von Mises strain under a transverse load (simulating "typical" service conditions) and, ultimately, the elastic moduli were predicted.

3. RESULTS

The optical microscopy results showed several features along the exposed face and resulted in a measurement of 54% carbon fibers and 46% resin (by area) within a localized region of interest. The addition of correlative XRM and EDS refined this measurement, yielding 50% carbon fibers, 35% matrix, and 15% glass fibers (with total void content <1%, by volume). From the von Mises strain analysis, it was clear that the highest strain under transverse load existed at the fiber-matrix interfaces, which indicated those locations to be the most likely defect nucleation sites under stressful service conditions. Finally, the analysis routine predicted a Young's modulus of 16.8 GPa, Poisson ratio of 0.25, shear modulus of 6.7, and bulk modulus of 11.0. Comparing these results to what may be considered a typical range for various carbon fiber composites, the predicted results are in reasonable agreement with the tabulated values, which served as a validation data point to verify the accuracy of the results [3].

Acknowledgements

References

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

[3] ASM Handbook, Volume 21 – Composites, by Committee, ASM International Handbook, 2001.

200 µm   5 mm   100 µm   30 µm

Light microscope   *X-ray microscope*   *Scanning Electron microscope*
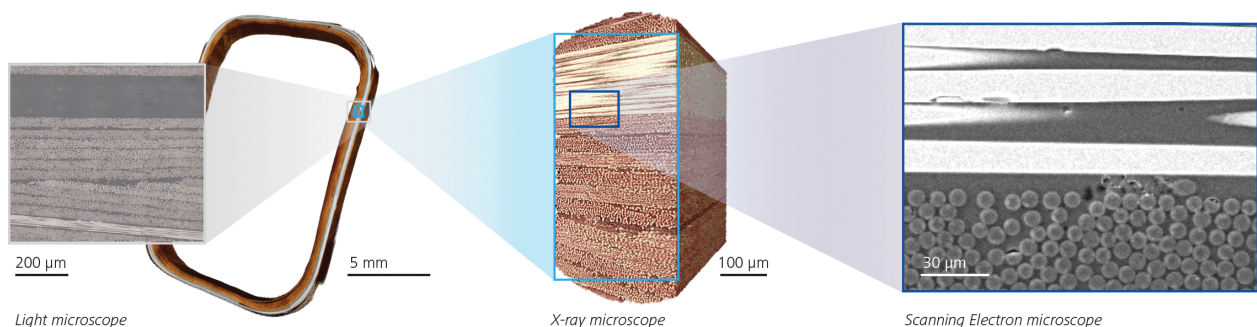
Figure 1: Multi-scale model of the hockey stick microstructure, incorporating optical (visible light) microscopy, X-ray microscopy, and SEM.