

# 3D INSIGHTS



## **THREE-DIMENSIONAL CHARACTERIZATION OF FIBER ORIENTATION IN A POLYMER MATRIX COMPOSITE**

We analyzed the porosity, volume percentage of fibers and matrix of a polymer matrix composite (PMC) using Robo-Met.3D®. Without acid etching, the Robo-Met.3D system revealed the desired features in the PMC and was successfully used in this case.

# 3D Characterization of Fiber Orientation in a Polymer Matrix Composite

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## Material Background

The structure and properties of polymer are becoming increasingly important as they are used in structural applications, both on their own and in polymer matrix composites (PMCs). Three-dimensional characterization of microstructural features such as size, shape, and distribution of matrix materials, strengthening fibers, and porosity provides us with the geometrical measurements of features directly. Robo-Met.3D is a fully automated serial sectioning system that generates two-dimensional optical microstructural data for three dimensional reconstructions. In this application note we describe the serial sectioning analysis of a PMC with a high temperature thermosetting resin matrix and a imide-based fiber reinforcement.

## Automated Serial Sectioning Analysis Using Robo-Met.3D®

Common applications of Robo-Met.3D include studying additively manufactured components<sup>1</sup>, analysis of welds and thermal barrier coatings<sup>2</sup>, and fiber orientation effects in ceramic matrix composites<sup>3</sup>.

A fiber reinforced PMC sample was selected for analysis. The sample was mounted conventionally for metallography, so as to be gripped in the polishing head (Fig. 1a). As-polished sections were used for this exploratory study, without etching, as the features of interest were pores (Fig. 1b). The material was polished with a 1 micron diamond suspension and finished with 0.05 micron colloidal alumina.

Optical images were automatically acquired with the Zeiss Axio Observer microscope built into the Robo-Met.3D system, as a magnification of 100x. The resultant spatial resolution was 1.08 Microns along X and Y axes. Sections were taken at 3 micron thickness.

## Image Processing

Forty (40) slices of 25 image tiles (a 5x5 montage) each were collected for 3D reconstruction. The image pixel sizes from this collection (after stitching and alignment) were 1500 microns along X; 1500 microns along Y, after images were cropped to exclude unwanted regions. The 2D image tiles from each layer were stitched into montages (or mosaics) and then registered with the images from the next layer using Fiji and Image J software. Figure 2 shows a sample conventional 2D image.

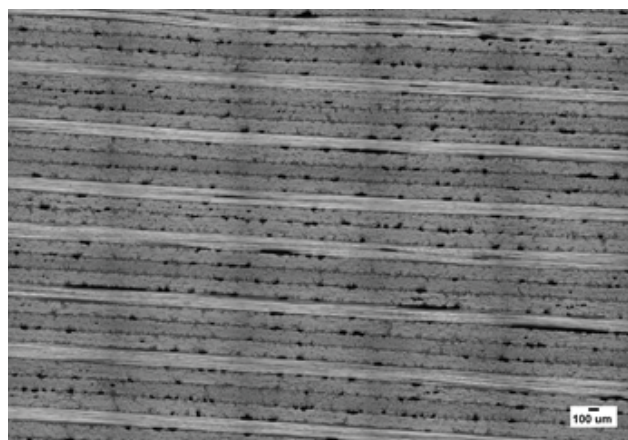
*Fig. 1a: Sample Mounted in Polishing Head*

*Fig. 1b: Polishing, Etching, and Cleaning Stations*

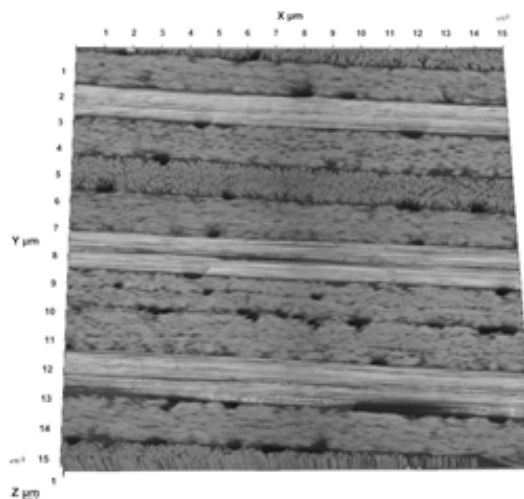
## 3D Analysis

3D analysis was performed using Image-Pro Premier 3D v.9.2.2 (Media Cybernetics, Inc., Washington, USA). For 3D analysis, loaded z-stack of 40 slices was processed with full resolution along X, Y, and Z directions. A 3D isosurface was created without any filtering to extract the volume distribution of the three phases (porosity, matrix, and fiber). An image histogram was used to threshold the porosity, filler, and fiber phases. Based on intensity values and the intensity, the range for porosity is set to 0-45, matrix to 46-94 and fiber phase to 95-220.

*Fig. 2: 2D Post-Processed (raw) image of the PMC Sample using Robo-Met.3D*



*Fig. 3: 3D Image (stack of 40 slices)*



The results of segmentation are shown overlaid (Figs. 3a-d). Quantification: size threshold feature was set to 5 microns along width, height, and depth; volume parameters have been extracted. The segmented images were analyzed using routines built into Image-Pro, to analyze the percentage of pores, matrix, and fibers. The volumetric conclusions (with ~ 1% error) are presented in the table below.

Color Label	Feature	Volume Percentage
Blue	Porosity	3%
Yellow	Matrix	17%
Red	Fiber	79%

From this analysis, we were able to conclude that identification, segmentation, and quantification of the defect such as porosity, matrix content, fiber content, and orientation was possible using Robo-Met.3D aiding in structural analysis of the PMC. Orientation and wave of the fiber are clearly visible in the 3D analyses.

Fig. 3a: 3D Image (Stack of 40 slices)

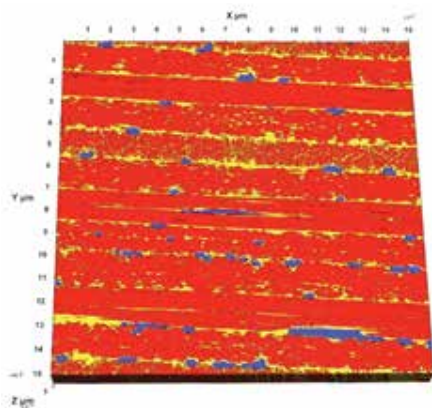


Fig. 3b: 3D Image (Segmented Pores)

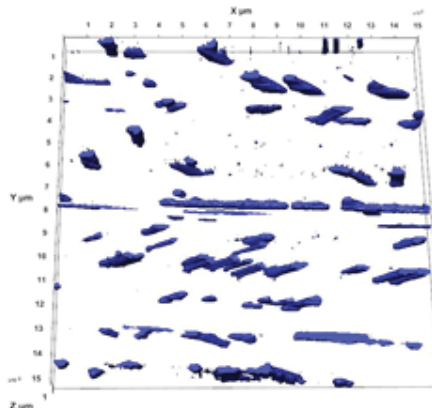


Fig. 3c: 3D Image (Segmented PMC-Matrix)

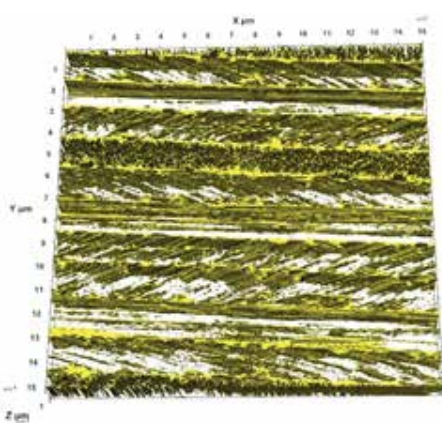
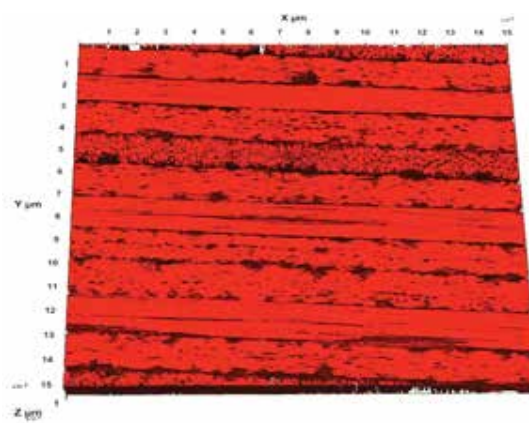


Fig. 3d: 3D Image (Segmented PMC - Fiber)



## Contributors

Bryan Turner (UES), and Satya Ganti (UES) performed the image collection and post-processing. We are grateful to Dr. Hilmar Koerner (AFRL) for the sample.

Animated views of the data are available at <http://www.ues.com/forums/forum/applications/>.

## References

- <sup>1</sup> Seifi, M., Ghamarian, I., Samimi, P., Collins, P. C., & Lewandowski, J. J. (2016). Microstructure and mechanical properties of Ti-48Al-2Cr-2Nb manufactured via electron beam melting. In *Ti-2015: the 13th World Conference on Titanium*.
- <sup>2</sup> Madison, J. D., Huffman, E. M., Poulter, G. A., & Kilgo, A. C. (2015). *R3D at Sandia National Laboratories - A User Update* (No. SAND2015-7665PE). Sandia National Laboratories (SNL-NM), Albuquerque, NM (United States).
- <sup>3</sup> Bricker, S., Simmons, J. P., Przybyla, C., & Hardie, R. (2015, March). Anomaly detection of microstructural defects in continuous fiber reinforced composites. In *SPIE / IS&T Electronic Imaging* (pp. 94010A-94010A). International Society for Optics and Photonics.