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Fiber Morphology Analysis for Directed-Energy Deposition Manufacturing Process

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Abstract

The structural and mechanical properties of fibers manufactured from a batch-wise directed-energy deposition process is related to the fiber morphology. Mechanical property data of a batch is determined by testing a random sample of fibers. Metrics such as edge roughness are utilized to quantify the variations in the outer edges of a fiber whose morphology is expected to be uniform. The amount of edge roughness present in a batch of fibers affects the mechanical properties of the batch. A correlation between reduced mechanical properties and edge roughness was possible through calculating edge roughness metrics programmatically for each batch of fibers. In order to automate the analysis of batches of fibers, a software tool was leveraged to gain understanding of the morphologies.

Introduction

The fiber manufacturing process highlighted in this paper allows for the manufacture of low density, temperature resistant material with superior mechanical properties to other commercially available alternatives. Material properties are determined by down-selecting a sample of fibers from a batch, which currently contains thousands of fibers but will expand to include orders of magnitude more. Optical images are captured for the down-selected batch. Mechanical and morphological properties are then calculated from optical images. Additional characterization and analysis is performed on an as-needed basis using scanning-electron microscopy.

Variations in material properties were observed in several batched and were correlated to fiber morphology. Within these batches distinct fiber morphologies were identified. Straight fibers yield acceptable mechanical properties. In this context, straight is defined as uniform in diameter and linear in shape. An example of a fiber is classified as straight is shown in Figure 1.

Figure 1. *Straight morphology. Diameter: 12.37 +/- 0.51 µm UTS: 3.36 GPa*

Figure 2. *Non-uniform morphology. Diameter: 12.93 +/- 0.76 µm UTS: 1.57 GPa*

Non-uniform morphologies yield lesser mechanical properties than straight fibers. An example of a fiber with decreased mechanical properties and a change in morphology is shown in Figure 2. After qualitatively classifying different fiber morphologies, a tool was created to quantify the variations in morphology in order to better aid in root-cause assessments of process variations.

Qualitative Assessment of Fiber Morphologies

In order to reverse-engineer the factor or factors in the manufacturing process that caused the change from straight fibers to non-uniform fibers, there was need to classify the kinds of defects in the fibers that were being observed. Classifying the different defects that deviate from a straight fiber was a qualitative process. This was done by comparing images from different batches with varying mechanical properties.

Jogs

Changes in axis direction along a length of a fiber can be defined as a jog. Figure 3 illustrates a representative example of a jogged fiber with arrows to highlight the changes in axes.

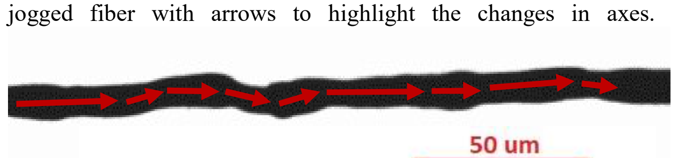

Figure 3. *Jogged fiber with white arrows indicating changes in axes.*

Fluctuations

Repeated and irregular changes in diameter where top and bottom edges of the fibers are anti-correlated can be defined as fluctuations. Figure 4 shows an example of fluctuating fibers with arrows to highlight the changes in diameter and correlating edges.

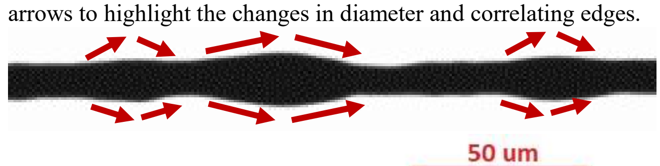

Figure 4. *Fluctuating fiber with red arrows indicating anti-correlated edges.*

Quantifying Changes in Morphologies

The ability to automate the analysis of images and test data of fibers is key to quantifying the mechanical properties and morphologies of a batch of fibers. By using software to process batch-wise data, historical information about batch morphology variations can be utilized to better understand potential process control pathways for the production of consistent, high-quality material.

Automated Analysis

The fiberMeasurement module is a program designed to calculate fiber diameter statics and ultimate tensile strength for individual fibers and for batches of fibers. The module was written using Python3 and is designed to analyze optical images given a pixel to micrometer conversion ratio. With the addition of edge roughness metrics the functionality expands to include morphology characterization.

Morphology Metrics

In order to better quantify variation in fiber morphology, the following metrics were added to fiberMeasurement module (1):

Edge Roughness Metrics

Line edge roughness (LER)	The sum of the absolute value of the residual edge positions divided by the number of positions.
Line width roughness (LWR)	The sum of the absolute values of the residual width positions divided by the number of positions.
Total Edge Roughness (TER)	The sum of the mean square roughness for both edges of a fiber.
Correlation coefficient (C)	LWR mean square roughness subtracted from the TER divided by the product of 2 and the square root of mean square roughness for both the top and bottom edge of the fiber. This metric allows for understanding if the fibers are not not correlated (straight) or either directly correlated or anti-correlated (oscillating). A value between -1 and 1.

The basis of the above metrics is the calculation of edge and width residuals from the top and bottom edge positions a fiber, given N edge positions. Edge residuals must be calculated for both the top and bottom edges of a fiber. To calculate the edge residuals, x_i, subtract the local edge positions, X_i, from the line of best fit:

$$x_i = X_i - (ai - b) \tag{1}$$

To calculate width residuals, w_i, the local width, W_i, is subtracted, from the mean width, $\bar{W}$:

$$w_i = W_i - \bar{W} \tag{2}$$

Mean Roughness

LER and LWR are measures of mean roughness, based on their respective residual values:

$$LER = \frac{1}{N}\sum_{i-0}^{N-1} |x_i| \tag{3}$$

$$LWR = \frac{1}{N}\sum_{i-0}^{N-1} |w_i| \tag{4}$$

Mean Square Roughness

Mean square roughness metrics provides understanding for the quadratic effects on roughness. TER is based on the sum of the mean square roughness of the top and bottom edge, $R^2{}_E$:

$$R^2{}_E = \frac{1}{N-2}\sum_{i-0}^{N-1} {x_i}^2 \tag{5}$$

$$TER = R^2{}_{E\_top} + R^2{}_{E\_bottom}$$

Width square roughness is calculated similarly to edge square roughness:

$$R^2{}_W = \frac{1}{N-1}\sum_{i-0}^{N-1} {w_i}^2 \tag{6}$$

The correlation coefficient is a metric that describes how each edge is related to the other.

$$C = \frac{TER - R^2{}_W}{2R_{E\_top}R_{E\_bottom}} \tag{7}$$

Morphology Data Analysis

After applying the updated fiberMeasurement module on all past collected data, system operators could use the mean C, LWR and TER values of a batch to quantify the batch morphologies at a glance without scrutiny of individual fibers. Both displaying the mean values of the metrics as well as histograms of the values over a batch, allowed for a deeper understanding of the morphologies over the sample and the batch as a whole.

Straight Fibers

The C value is used to indicate if the edges are correlated in movement. For straight fibers, a mean correlation coefficient of 0 indicates no dominating features or shape defects. In the present instantiation of the tool the image resolution is 0.91 µm per pixel. Therefore, any value less than 0.91 for LWR, or TER is outside of the resolution that can be quantified. Any roughness below 0.91 is less than one pixel indicates the fibers are acceptably straight based on the current body of data. Example images from a batch of straight fibers along with property data is presented in Figure 5 and morphology metrics are summarized in the table below.

Figure 5. Batch of fibers with straight morphology. Mean Batch Diameter: 12.39 +/- 0.48 µm Mean Batch UTS: 3.05 +/- 0.48GPa

Batch of Fibers with Straight Morphology Edge Roughness Metrics

Mean C	0.00
Mean LWR	0.54 µm
Mean TER	0.76 µm

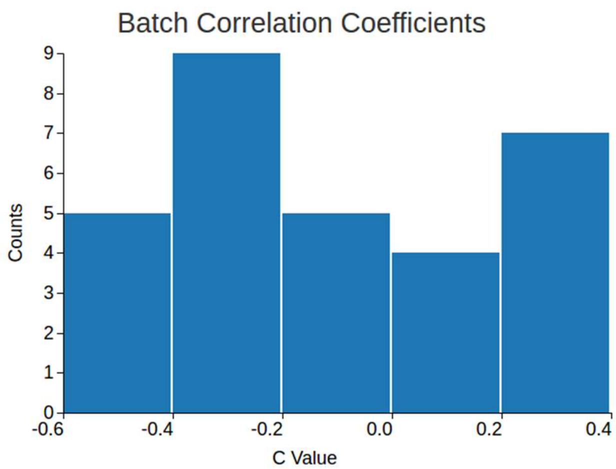

***Figure 6.** Histogram of batch with straight morphology correlation coefficients.*

Straight fibers have the most desirable mechanical properties. The above histogram in Figure 6 shows a range of C values that center around 0, but the range is not normally distributed. This indicates a range in slightly different morphologies throughout a batch that results in a generally straight batch with desired mechanical properties.

Jogged Fibers

For jogged fibers, an mean positive C value indicate the top and bottom edges of the fiber move in the same direction and are correlated. The mean LWR, and TER values will be one pixel or more (higher than the pixel to micron ratio), significantly larger than the straight fibers. Example images from a batch of jogged fibers are shown in Figure 7 and the morphology metrics are summarized in the subsequent table.

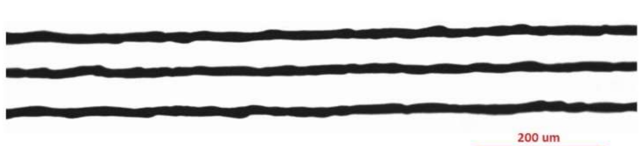

***Figure 7.** Batch of fibers with jogged morphology. Mean Batch Diameter: 13.52 +/- 0.95 µm Note: Resulting poor structural properties preventing mechanical testing.*

Batch of Fibers with Jogged Morphology Edge Roughness Metrics

Mean C	0.55
Mean LWR	0.98 µm
Mean TER	4.57 µm

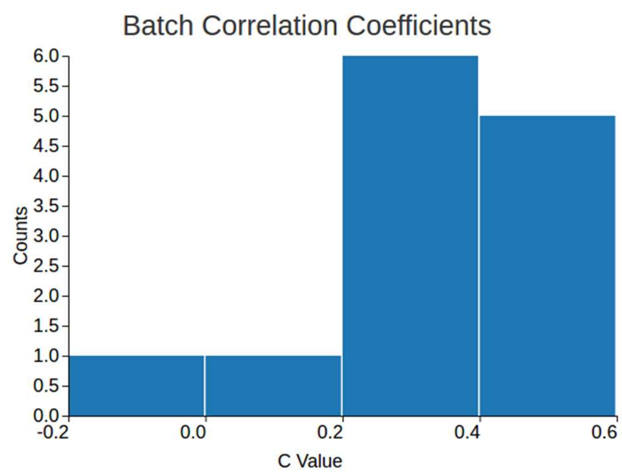

***Figure 8.** Histogram of batch with jogged morphology correlation coefficients.*

Jogged fibers were determined to have the least desirable mechanical properties. In this example, the fibers were not able to be handled to be tested due to breaking. The histogram for these fibers (Figure 8) shows a heavy skew towards positive correlation between the two edges, which is expected in a jogged fiber.

Fluctuating Fibers

For fluctuating fibers, a negative mean correlation coefficient indicates the top and bottom edges of the fibers are anti-correlated. The mean LWR and TER are larger than one pixel, also significantly larger than the straight fibers. Representative images from a batch of fluctuating fibers are presented in Figure 9. The results from the morphology analysis tool are summarized in the subsequent table.

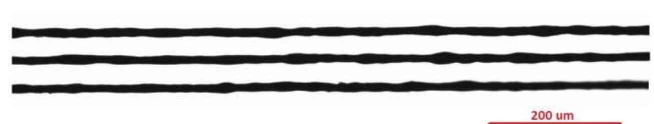

***Figure 9.** Batch of fibers with fluctuating morphology. Mean Batch Diameter: 11.93 +/- 0.58 µm Mean Batch UTS: 0.64 +/- 0.44 GPa*

Mean C	-0.56
Mean LWR	1.22 µm
Mean TER	1.98 µm

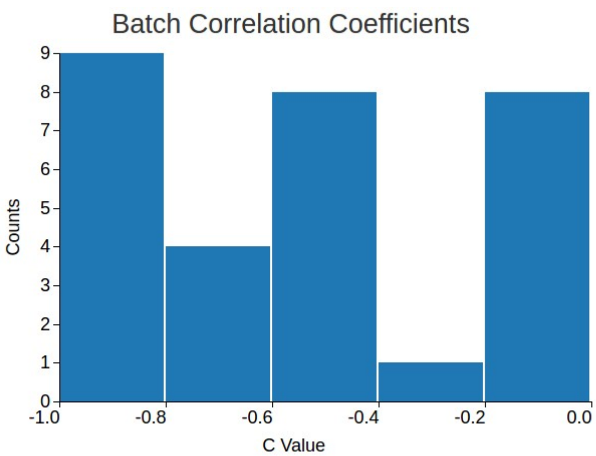

Figure 10. *Histogram of batch with fluctuating morphology correlation coefficients.*

Fluctuating fibers have properties that are not desirable, but are strong enough to be mechanically tested. The correlation coefficients in this batch of fibers skew towards a negative correlation between the two fiber edges (Figure 10). This is expected of fluctuating fibers because the edges are anti-correlated.

Conclusions

The addition of the edge roughness metrics allows for the comparison of different fiber morphologies. With the updates to the fiberMeasurement module, metrics for the uniformity of the fibers are made available at a glance. The fiberMeasurement module is currently being used during post-process batch testing for processing optical images of the fibers. Going forward, this functionality will be explored for expansion to monitor the morphology in-situ – providing real-time feedback to the process control software. This evolutionary step could lead to reduced process development cycle times and a more stable manufacturing process overall.

Future Work

Refinement of the fiberMeasurement tool will continue in order to facilitate the continued development of the fiber manufacturing process. One of the challenges of taking edge roughness metrics is random error introduced by noise in images (2). To reduce random error and increase the accuracy, the software tool will read images with higher resolution at a higher magnification.

The addition of the metrics to the fiberMeasurement post-process analysis of fibers could potentially be expanded to monitor fiber morphology during the manufacturing process in situ. With this feedback, factors that affect the fiber shape could be adjusted in real-time as a part of the process control software in order to reduct product variability.

References

[1] Benjamin D. Bunday, "Determination of Optimal Parameters for CDSEM Measurement of Line Edge Roughness," Proc. SPIE., vol. 5375, pp. 517-533, 2004

[2] J. S. Villarrubia, "Issues in Line Edge and Linewidth Roughness Metrology," Characterization and Metrology for ULSI Technology, 2005

Author Biography

Cailin Simpson is a PhD student in Systems Engineering at Colorado State University. She is also a full-time software engineer at Dynetics Technical Solutions in Huntsville, AL. Her research is focused on data science and analytics applications for advanced manufacturing systems and sensors.