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Published in:
Data in Brief

Link to article, DOI:
10.1016/j.dib.2021.106868

Publication date:
2021

Document Version
Peer reviewed version

Citation (APA):
Mikkelsen, L. P., Fæster, S., Goutianos, S., & Sørensen, B. F. (2021). Scanning electron microscopy datasets for local fibre volume fraction determination in non-crimp glass-fibre reinforced composites. Data in Brief, 35, [106868]. https://doi.org/10.1016/j.dib.2021.106868
Article Title
Scanning electron microscopy datasets for local fibre volume fraction determination in non-crimp glass-fibre reinforced composites.

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Abstract
The fatigue damage evolution depends on the local fiber volume fraction as observed in the co-submitted publication [1]. Conventionally, fiber volume fractions are determined as an averaged overall fiber volume fraction determined from small cuts of the laminate. Alternatively, automatically stitching of scanning electron microscopy (SEM) images can make high-resolution scans of large cross-section area with large contrast between the polymer and glass-fiber phase. Therefore, local distribution of the fiber volume fraction can be characterized automatically using such scan-data. The two datasets presented here cover two large Field of Views scanning electron microscopy (SEM) images. The two images is generated from between 1200 to 1800 high-resolution scan pictures which have been stitched into two high-resolution tif-files. The resolution corresponds to between 700-5000 pixels covering each fibre. The datasets are coming from two different non-crimp fabric glass fibre reinforced epoxy composites typically used in the wind turbine industry. Depending on the regions analyzed, fibre volume fraction in the range of 50-85% is found. The maximum local fibre volume fraction is found averaging the local fibre volume fraction over 5x5 fibre diameter (80x80 µm²) areas. The local fibre volume fraction has been used in the analysis performed in [1].

Keywords
Bundle segmentation, SEM, Fatigue damage evolution, wind turbine blades.

Specifications Table

| Subject       | Material Science |
| Specific subject area | Composites, Scanning electron microscopy, fibre volume fraction |
|-----------------------|---------------------------------------------------------------|
| Type of data          | Image (Scanning electron microscopy images) MetaData (hdr-files with metadata for the SEM data) Scripts (Matlab scripts for local fibre volume fraction determination) |
| How data were acquired| VEGA3 SBU tabletop microscope (SEM) |
| Data format           | Raw: SEM individual micrographs in ".bmp" format zipped together Processed: SEM stitched micrographs in ".tif" format, Mat-files with the segmented areas. Analyzed: Matlab scripts for analysing the SEM data |
| Parameters for data collection | The 2D micrographs were obtained using scanning electron microscope with an accelerating voltage of 20kV. |
| Description of data collection | Cuts of two composites test samples embedded and prepared by standard grinding and polishing followed by SEM scanning. |
| Data source location  | DTU Wind Energy, Roskilde, Denmark, Latitude: 55.695343, Longitude: 12.08921 |
| Data accessibility    | Repository name: Zenodo.org Data identification number: 4064835 Direct URL to data: http://doi.org/10.5281/zenodo.4064835 |
| Related research article | B.F. Sørensen, S. Goutianos, L.P. Mikkelsen, S. Fæster, Fatigue damage growth and fatigue life of unidirectional composites. Composite Science and Technology, Submitted. |

**Value of the Data**

- The high-resolution large field of view SEM scanning data is used to determine the local fibre volume fraction distribution in two different non-crimp fabric based glass-fibre composites. The data is used to characterize local fibre volume fraction in conventional non-crimp fabrics, which values subsequently is used in reference [1].
The industry and academia can use the provided datasets for studying fibre volume fraction variations in non-crimp fabric-based composites. The two datasets can be used as a benchmark dataset for developing segmentation and analysis tools for local fibre volume fraction and fibre diameter determination. The datasets can also be used for investigating variation in the fibre volume fractions at different locations in the fabric, e.g., close to the backing fibre bundle.

Data Description

For each of the two cases, five files are made available at the Zenodo repository, see [4]. Those two file-set contains:

- Tif-file: The stitched SEM scanned image which is used in the fiber volume fraction analysis.
- Hdr-file: Meta-data about the stitched SEM scanned image.
- M-file: The Matlab script used for analyzing the tif-file
- Mat-file: Matlab mask data for a selection of the bundles used in the fiber volume fraction analysis
- Zip-file: Collection of the individual SEM scanned images and meta-data files behind the stitched SEM image.

The full cross-section and a zoom-in on the scanning electron microscope images for the two cases are shown in Figures 1 and 2. The images were acquired using a Tescan VEGA3 SEM with the settings as listed in Table 1. The composite samples were cut orthogonal to the dominating fibre orientation, with the cutting surface, subsequently polished and applied with an approximately 10nm thin layer of carbon using a Bal-Tec SCD 005 Sputter Coater. The material samples are cut from fatigue test samples used in reference [2] and [3]. The two cases will here be denoted as Cases 1 and Case 2, respectively. The images were taken with a pixel size of 390.63nm and 195.31nm for Figures 1 and 2, respectively, using a source magnification of 692x at a high tension of 15 or 20 kV using a backscatter (BSE) detector.

Table 1: SEM data setting

| Parameter               | Case 1   | Case 2   |
|-------------------------|----------|----------|
| Pixel size scan [nm]    | 390.63   | 195.31   |
| Pixel size stitched [nm]| 527.25   | 195.31   |
| Magnification           | 692      | 692      |
| Parameter              | Detector BSE | BSE |
|------------------------|--------------|-----|
| Acc. voltage [kV]      | 15           | 20  |
| Scan speed             | 6            | 6   |
| Exposer time [µs/pxl]  | 32           | 32  |
| Number of images       | 1764         | 1200|
| Overlap [%]            | 10           | 10  |
| Total scan time [h]    | 15           | 6   |

Figure 1: Scanning electron microscope images of Case 1 scanned with a VEGA3 SBU. With a pixel size of 527.25 nm, the fibre diameter (16 µm) will be cover 30 pixels over the diameter and 724 pixels in area.
Experimental Design, Materials and Methods

The scan parameter for the scanning electron microscopy (SEM) images, see Table 1, were carefully selected to generate the best possible contrast difference between fibres and resin. The SEM images were acquired and afterward stitched together with the “image snapper” function in the Tescan software VegaTC to generate SEM images of large regions with high resolution. The SEM images were processed with a MATLAB script, where the data were loaded with the function `imread` and binarized using an Otsu threshold value determined by the Matlab-function `otsuthresh` based on a histogram of a central 1000×1000 pixel subsection as shown in figure 3 and 4 for Cases 1 and 2, respectively.

The binarization shown to the left in figures 3 and 4 can now be used for calculating the fiber volume fraction simply by finding the ratio between the number of binarized pixels above the Otsu threshold (fibers) over the total amount of pixels inside the region. For the total scanned area shown in figures 1 and 2, the overall fibre volume fraction is found to be $V_f = 0.598$ and $V_f = 0.525$, respectively. A value that in Table 2 is compared with the values measured by a back-calculated or burn-of experiment reported in the two references [2] and [3], respectively. For Case 1, it should be noted that not the total layer of the lower biax ply is included in the SEM scan, which may result in the slightly larger overall fiber volume fraction found compared to the value reported in reference [2].
Figures 5 and 6 show manually segmented UD fiber bundles from inside the SEM scanned region. In Case 1, 8 different unidirectional bundles are segmented, while it for Case 2 includes a major part of two unidirectional bundles. The unidirectional bundles were manually segmented using the Region of Interest drawing tool in the Image Segmenter toolbox in Matlab. Inside each segmented region, the fiber volume fraction was calculated in the same way as for the overall fiber volume fraction. The values are reported in Table 2 together with their standard deviations.

Table 2: Fiber volume fractions

|               | Overall $V_f$ | Bundle $V_f$ | Max $V_f$ |
|---------------|---------------|--------------|-----------|

Figure 3: Otsu threshold for Case 1

Figure 4: Otsu threshold for Case 2
| Case   | Reference | From ref. | SEM          | $5D_f \times 5D_f$ |
|--------|-----------|-----------|--------------|--------------------|
| Case 1 | [2]       | 0.57      | 0.598        | 0.668±0.020        | ≈0.85              |
| Case 2 | [3]       | 0.53      | 0.525        | 0.606±0.042        | ≈0.85              |

Figure 5: Local fiber volume fraction of 8 UD bundles from a zoomed region of Case 1
Figure 7 and 8 show the variation of the fiber volume fraction calculated by using a moving area averaging of a $5 \times 5$ fiber diameters sized area which for a fiber diameter equal to $D_f = 16 \mu m$ corresponds to approximately $80 \mu m \times 80 \mu m$. This was done using the Matlab function \texttt{conv2} and was applied over the full scanned region as shown in figures 7 and 8. From this, a local fibre volume fraction is determined. In addition to the full scanned region, an image of a region with regions of high fibre volume fraction is presented. From the contour plots, regions with local fibre volume fraction up to around $V_f \approx 0.85$ for both cases were identified.
Figure 7: Averaging over a 5x5 fibre diameters moving area for Case 1

Figure 8: Averaging over a 5x5 fibre diameters moving area for Case 2

CRediT author statement

Lars P. Mikkelsen: Conceptualization, Software, Visualization, Writing - Original Draft, Writing - Review & Editing. Søren Fæster: Methodology, Visualization, Writing - Review & Editing. Stergios Goutianos: Writing - Review & Editing. Bent F. Sørensen: Conceptualization, Funding acquisition, Writing - review & editing.

Acknowledgments

This research was partly supported by the project DACOMAT (Damage Controlled Composite Materials), funded by the European Union’s Horizon 2020 research and innovation programme under GA No. 761072. Thanks to Jesper Asgaard Bøtner (Rockwell Int. A/S) for assisting with the SEM measurements. Thanks to Samuli Korkiakoski and Povl Brøndsted for letting us analysing their test specimens from [3].

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

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