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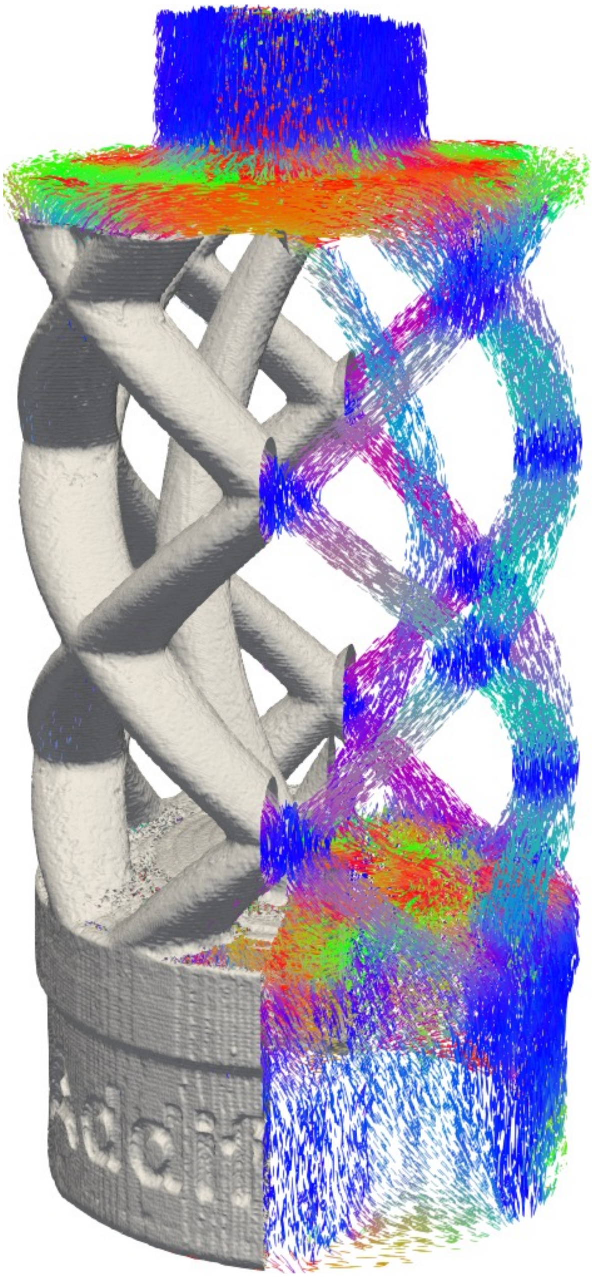

FiberScanner3D^{TM} Laboratory X-ray Tensor Tomography

Unlocking Small-Angle Scattering Information from Fibrous Materials using Laboratory X-ray Microscopy

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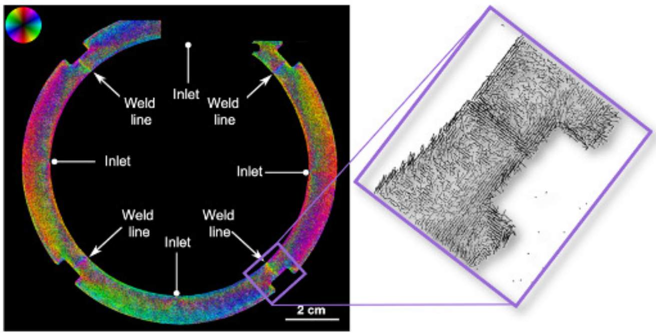
X-ray computed tomography (CT) primarily relies on X-ray absorption contrast to highlight material density variations within samples. However, this method falls short when imaging low-density fibrous materials such as artificial or natural fiber-reinforced polymers and certain biological specimens, where absorption contrast is minimal. Furthermore, the stringent resolution requirements often limit the imaged field of view, posing a challenge, especially in analyzing fiber orientations crucial for defining mechanical properties on a macroscopic scale. To address these limitations, directional dark-field/scattering X-ray tensor tomography (XTT) has emerged as a compelling alternative, offering exceptional efficiency and performance. This technique provides detailed microstructure orientation information of low-density samples in 3D, non-destructively. Now, pushing the boundaries of laboratory X-ray microscopy, we present the capabilities of state-of-the-art laboratory-based XTT on the Exciscope Polaris X-ray microscope platform.

Introduction

Xnovo Technology ApS has extensive experience delivering novel X-ray imaging solutions for academic and industrial R&D labs and quality control. Xnovo has developed and launched FiberScanner3D, an efficient method for fiber orientation mapping in 3D via scattering (dark-field) contrast tensor tomography. In partnership with Exciscope AB, FiberScanner3D is available on Exciscope Polaris, a phase-contrast X-ray microscope.

Fiber orientation characterization is typically conducted using various imaging techniques such as light, electron, and X-ray microscopy [1]. These methods play a crucial role in imaging the microstructure and quantifying fiber orientation distribution in composite materials which is essential for understanding the materials' mechanical properties. However, both light and electron microscopy techniques are limited by their surface-focused nature, making it challenging to accurately evaluate the three-dimensional microstructure [2]. X-ray CT offers a volumetric view of fiber orientation distribution, but its effectiveness depends on several factors, including sample size, density, composition, and structure. Additionally, achieving optimal results requires meeting specific conditions in terms of resolution, contrast, and scan time. For low-density materials with micro-scale structures, such as carbon or natural fiber-reinforced polymers, the resolution requirements can significantly limit the imaged field of view (FOV).

In recent years, there has been a growing interest in scattering contrast X-ray microscopy techniques for orientation analysis of both artificial and natural fiber-reinforced polymers, as well as biological materials with anisotropic structures [3, 4]. These methods offer a unique advantage: mapping anisotropic structures without their direct resolution, thus expanding the FOV. The technology behind FiberScanner3D stands out from its predecessors, all of which encounter throughput challenges, limiting their application in laboratory settings. Namely, scanning small-angle scattering methods entail significant overhead due to 2D raster scanning with a focused X-ray beam while full-field linear grating methods require additional linear and rotational shifts for 2D orientation sensitivity.



Fiber orientation map of an injection molded carbon fiber reinforced component reconstructed using FiberScanner3D. The orientation and alignment of fibers is represented via different colors and their intensities, respectively.

In collaboration with the Paul Scherrer Institute, Xnovo has pioneered a breakthrough grating design featuring circular unit cells [5]. This circular design ensures 2D orientation sensitivity within a single frame and allows for single-shot mapping of orientations and scattering in 2D. The innovative approach with each unit cell offering local scattering information mimics raster scanning but in a more efficient manner. The hereby-obtained high throughput is crucial, especially for applications necessitating full 3D scattering tensor information, which is attainable through X-ray tensor tomography (XTT) [6].

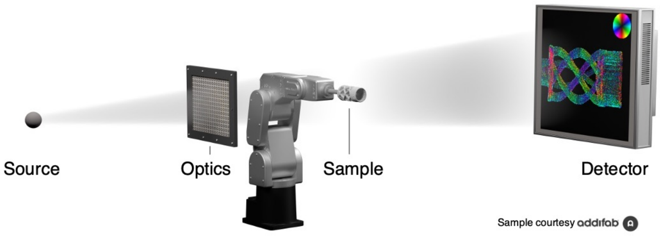


Fig. 1. *Schematic of the FiberScanner3D setup. The sample is illuminated by X-rays from the source. The sample-induced small-angle scattering is recorded with a detection system. Images of the sample at different viewing angles are collected and used in the 3D tomographic/volumetric scattering tensor reconstruction.*

FiberScanner3D: How it works

Here, we introduce a novel methodology for the acquisition, reconstruction, and analysis of fiber orientation and associated information in materials with anisotropic structures, such as those containing fibers, using the FiberScanner3D add-on module on a commercial laboratory X-ray microscope (Exciscope Polaris) equipped with a microfocus metal-jet X-ray source.

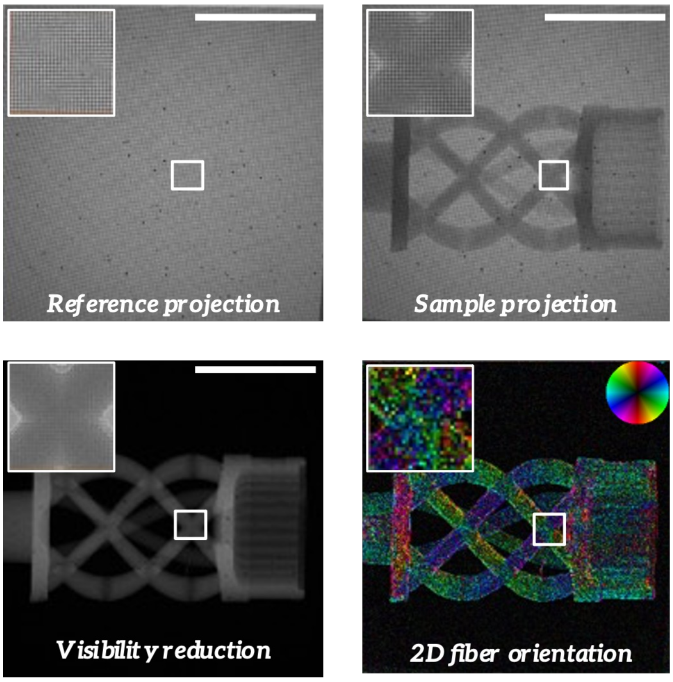


Fig. 2. *Example 2D projections and fiber orientation provided by FiberScanner3D. The reference pattern of the grating optics is measured without the sample followed by a series of sample projections at different orientations/poses. From the sample-induced visibility reduction/small-angle scattering, 2D fiber orientation is extracted. FiberScanner3D provides fast and robust analysis of the small-angle scattering signal without relying on high-precision optics alignment. Scale bars are 10 mm.*

A schematic representation of the FiberScanner3D implementation is shown in Figure 1. The X-ray beam illuminates the sample through specialized grating optics. Small-angle scattering within every unit cell of the grating optics is recorded on the detector. The acquisition of the sample's absorption information is simultaneously performed.

With FiberScanner3D, robot-arm-assisted sample manipulation leads to a series of projections at different sample poses. Figure 2 illustrates a 2D reference pattern of the grating optics as acquired with FiberScanner3D. The sample is imaged through the grating, and the pattern visibility reduction due to small-angle scattering is derived. Directional scattering is then visualized as 2D fiber orientation. Only a few hundred projections are sufficient for a complete tensor tomography data set. Utilizing FiberScanner3D analysis capabilities, spatially resolved 3D directional scattering within the sample can be reconstructed.

Validation and Application Example

A carbon fiber-reinforced polymer (CFRP) sample containing fibers with a diameter of 5 μm produced using a free-form injection-molding process (courtesy of Addifab | Nexa3D) has been explored and used here to demonstrate the results of FiberScanner3D. Successful 3D reconstruction of the scattering tensor yields a completely voxelated scattering tensor representation of the entire sample. This information enables extracting details of the sample's microstructure, such as fiber orientations, fiber alignment, fiber volume fraction, etc. A 3D fiber orientation map of the CFRP sample is shown in Figure 3a, where the fiber orientations are color-coded according to the 3D color sphere. A horizontal slice through the sample is shown in Figure 3b to highlight the complex arrangement of the fiber orientations and degree of fiber alignment.

To help validate the accuracy of the FiberScanner3D (laboratory XTT) technique, comparisons to the synchrotron XTT and absorption X-ray microscopy were carried out to perform independent measurements of the sample microstructures.
The accuracy of the fiber orientations was compared to those obtained using synchrotron XTT. Reconstructed fiber orientation is shown in Figure 4a, along with the histogram of per voxel misorientations determined from the laboratory XTT measurement. The misorientation expressed as the inner product of fiber orientation vectors matches well, with the mean value of 0.95. The parameters of both measurements are shown in Table 1.

Measurement parameter	SyncXTT	LabXTT
Exposure time, s	0.05	15
Detector pixel size, μm	6.5	16
Effective voxel size, μm^3	84.5	80
Field of view, mm^2	16.6x4.3	25x25
Volume size, mm^3	12x12x25	25x25x25
Images per sample pose	8	1
Total number of images	5678	721

Table. 1. *Comparison of the main measurement parameters between synchrotron XTT and FiberScanner3D (laboratory XTT).*

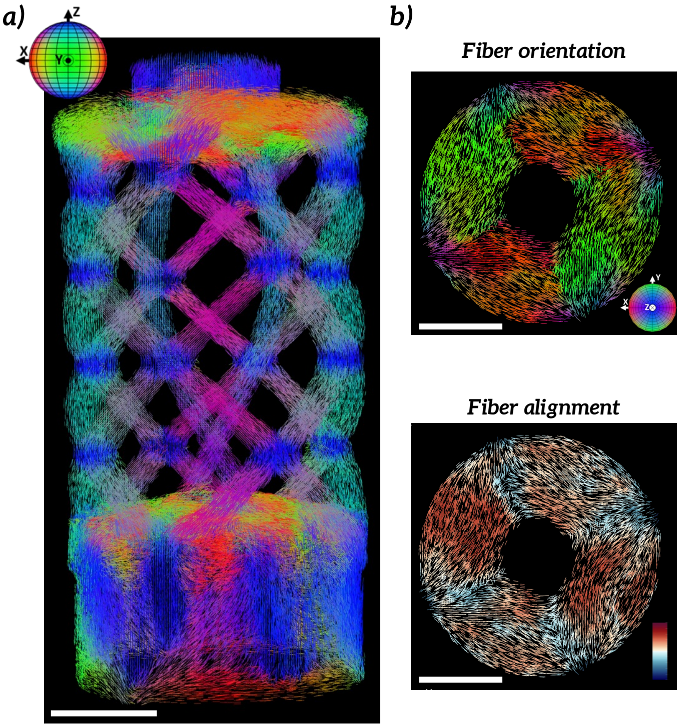


Fig. 3. *a) Visualization of a 3D fiber map of the CFRP provided by FiberScanner3D. Scale bars are 5 mm. The color sphere highlights the fiber orientation information. b) Virtual cross-section through the 3D fiber map in fiber orientation and fiber alignment representations.*

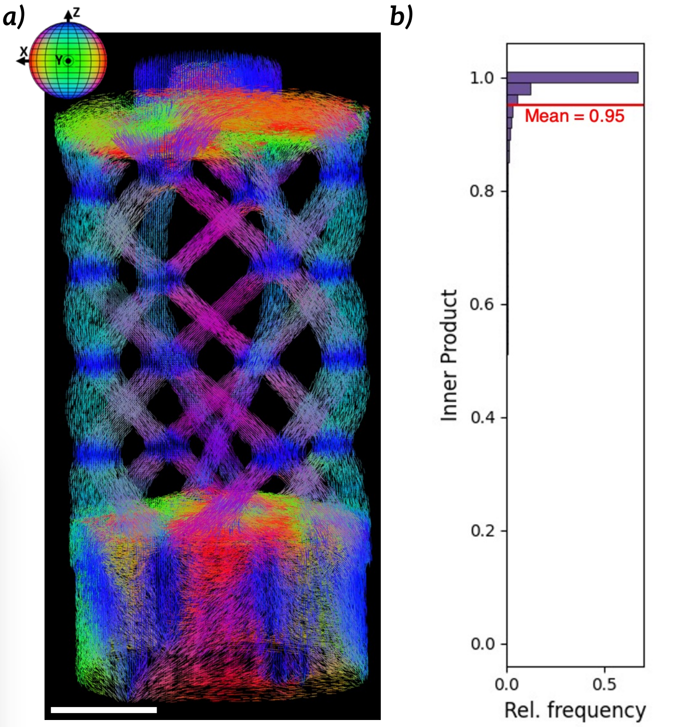


Fig. 4. *a) Visualization of a 3D fiber map of the CFRP provided by synchrotron XTT. Scale bar is 5 mm. b) The histogram illustrates a distribution of the inner product of fiber orientation vectors obtained from synchrotron XTT and FiberScanner3D. A value of 1 signifies ideal alignment between the orientations determined by both methods.*

Due to the slight density variation between carbon fibers and polymer matrix, the fibers can be visualized through high-resolution X-ray absorption CT (XCT) and further analyzed to retrieve fiber orientations [7]. The resolution requirements of the absorption X-ray microscope, however, limit the imaged field of view to a few cubic millimeters. Therefore only a few sub-volumes of the sample were scanned. The measurement parameters are shown in Table 2. Figure 5a shows the location of the scanned sub-volumes (s01a-c) within the CFRP sample. Spatial variations of fiber orientations are shown in Figure 5b on the slice through the stitched reconstruction of s01a-c. Figure 5c shows the comparison of 3D fiber orientations derived from high-resolution XCT and FiberScanner3D. The 3D fiber orientations agree well within the scanned regions between both methods.

Measurement parameter	XCT	LabXTT
Exposure time, s	30	15
Detector pixel size, µm	35	16
Effective voxel size, µm^3	1	80
Field of view, mm^2	2x2	25x25
Volume size, mm^3	2x2x2	25x25x25
Images per sample pose	1	1
Total number of images	1601	721

Table. 2. *Comparison of the main measurement parameters between high-resolution XCT and FiberScanner3D (laboratory XTT).*

Conclusions

We have introduced the principles of scattering tensor tomography and its application to determine fiber orientations and fiber alignment in fiber-reinforced composite samples. This demonstrates the ongoing advancement in laboratory X-ray microscopy, specifically tailored to address the needs for large field-of-view, high-throughput imaging methods, drawing inspiration from synchrotron origins. The unique grating optics design enables directional scattering/dark-field data acquisition at an unprecedented scale. Advanced reconstruction and analysis capabilities of FiberScanner3D software provide high-quality reconstructions. The continued use and application development of this technique will accelerate the way 3D and 4D material science is pursued for the non-destructive study of materials with anisotropic structures.

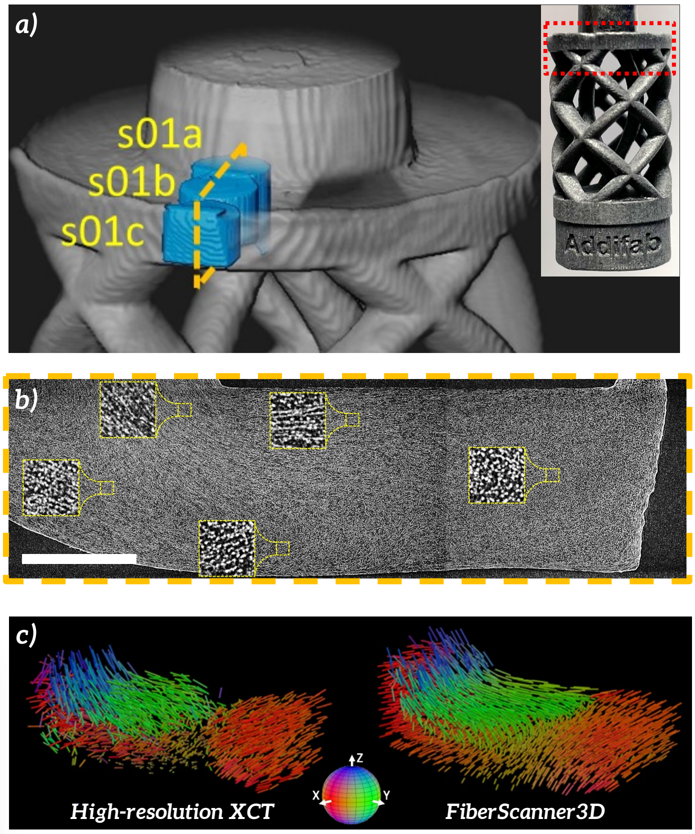


Fig. 5. *a) Visualization of the sample sub-volumes (in blue) scanned with high-resolution XCT. b) Virtual cross-section through the stitched volume of XCT at s01a-c. Insets show zoomed-in images that visualize complex fiber orientations at different locations of the sample. Scale bar is 1 mm. c) Side-to-side comparison of fiber orientation obtained from the stitched XCT volume and the FiberScanner3D reconstruction at the associated part of the sample.*

The FiberScanner3D is available as an add-on module to the Exciscope Polaris phase-contrast X-ray microscope

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Innovative 3D X-ray Imaging Solutions

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