

# Scanning WAXS and microtomography for bulk structure characterization of graphene-based bio-composite

## THE INDUSTRIAL CHALLENGE

Biobased composite companies aim to use graphene for new functionalities. The wood fiber composite manufacturer Biofiber Tech (BFT) AB needs to avoid graphene layer stacking into graphite to ensure optimal conductivity in wood fiber composite (WFC) for electromagnetic shielding and 3D printing materials. The pulp manufacturer Holmen AB needs to optimize the graphene stacking thickness as air barrier layer in the nanocellulose/polymer (NFPC) composite. However, distinguishing between crystalline scattering from biobased fiber, polymer, and graphene crystallinity and dispersion to standardize production has been challenging.

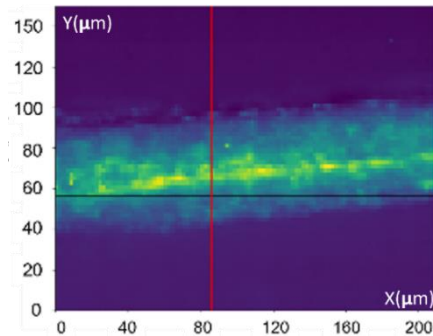
## WHY USING A LARGE SCALE FACILITY

Lab-based X-ray scattering methods and electron microscopy techniques have been inefficient and inflexible for industrial applications, taking long time and often failing to characterize the above-mentioned crystallinity. Synchrotron radiation facilities offer highly coherent X-rays that enable efficient characterization, capable of distinguishing different crystalline scattering at the atomic scale in milliseconds. X-ray tomography achieves 3-dimensional characterization in micrometer scale of bulk microstructures without requiring additional sample preparation to enable versatile characterization for industrial samples as films, injection-molded pieces, or 3D-printed objects.

## HOW THE WORK WAS DONE

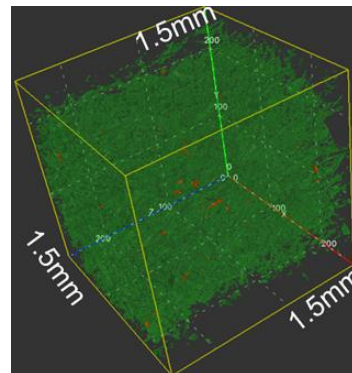
The experiments were conducted at the European Synchrotron Radiation Facility (ESRF) in France, with the assistance of ESRF scientists Dr. Michael Sztucki (ID13) and Dr. Phil Cook (BM05). Scanning wide-angle X-ray scattering (WAXS) was performed at the ID13 beamline to identify the stacking of graphene layers in NFPC films and in WFC injection-molded dumbbell samples. Micro-computed tomography ( $\mu$ CT) at the BM05 beamline was used to obtain bulk information on a 3D-printed kayak.

## THE RESULTS AND EXPECTED IMPACT



The upper figure displays a WAXS image that shows bright yellow, revealing that graphene is stacked in a graphite-like air barrier layer. The graphite flake plane is parallel to the thin NFPC film in a 150 x 210  $\mu$ m measurement area from X-ray orientation analysis. This result will be used as the air barrier layer standardization baseline for Holmen. No graphite scattering from the graphene functionalized WFC was observed, indicating that graphite stacking has been avoided in conductive composite during compounding.

The lower figure shows pore distribution and average fiber size from a 3.4 mm<sup>3</sup>  $\mu$ CT volume of 3D-printed WFC kayak. The data will be used by kayak manufacturers for production standardization.



***“The first time we see the 3D visualized bulk structures of biobased fiber composites. Beneficial to product development and demonstration to our customers.” /Luis Valencia, Biofiber Tech AB***

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