INDUSTRIAL PILOT PROJECT

X-ray photoemission electron microscopy as an analytical tool for spatially resolved chemical states of lignin-based graphene

THE INDUSTRIAL CHALLENGE

Bright Day Graphene has developed a method for continuously producing bio-based graphene from a biomass-derived polymer called lignin. The properties of the produced graphene can vary based on the lignin source and conversion parameters. For further optimization of the production process and controlling the properties of the product, it is critical to understand the properties from a structure-processing relationship perspective.

WHY USING A LARGE SCALE FACILITY

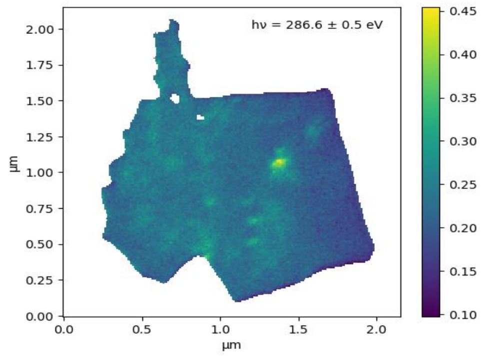
The X-ray photoemission electron microscopy (XPEEM) at synchrotron radiation facilities uses signals of soft X-ray spectroscopies, such as X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy, as imaging contrast for full-field high-resolution imaging. This detects the graphene's spatially resolved empty orbital structures, which are closely related to the local chemical structure. The XPEEM image complements lab-based scanning electron microscopy and scanning Raman spectro-microscopy with element-specified chemical sensitivity, e.g., targeted on C and O respectively, and high spatial resolution. For the MAXPEEM beamline at the MAX IV, the resolution is down to 50 nm. Also, it is possible to map the core-level/valence band electronic structure by XPEEM, which is reserved for future studies.

HOW THE WORK WAS DONE

The bio-based graphene produced by Bright Day Graphene was dispersed in acetone solution, drop-casted on Au-coated Silicon chips (10×10 mm^2), and analyzed at the MAXPEEM beamline under ultrahigh vacuum (~10^{-10} Torr). After proper outgassing of the specimen by heating above 120 ˚C in the vacuum, the specimen was first evaluated against the radiation damage by recording a series of consecutive PEEM images at a fixed photon energy as a function of time. Once the dose limitation was set, the low energy electron microscopy (LEEM) integrated into PEEM was used to find a fresh measurement spot containing an isolated particle. The spectro-micrographs were collected on the identified particle around the C and O resonant transitions, known as K-edge. In total, six specimens prepared via different processing were characterized. Dr. Yuran Niu and Dr. Lin Zhu at MAX IV are acknowledged for performing the operations, including measurements and sample transfer. The data analysis was carried out via customer-programmed Python scripts in the JupyterHub server provided by the MAX IV laboratory.

THE RESULTS AND EXPECTED IMPACT

By measuring the C and O K-edge X-ray absorption signal with 50 nm spatial resolutions, we could map the chemical states distribution and correlate it to the processing conditions. The figure shows a typical XPEEM image that corresponds to the C=O (π*) impurity contribution in the empty molecular orbitals within a single flake of lignin-derived graphene. The brighter colors show where the impurity accumulated.



The results allow Bright Day Graphene to better understand how the processing parameters modify the chemical structure of the products.

"Through analyzing our material at the MAXPEEM beamline we have gotten detailed information that we could not otherwise have received. Thanks to this project we are much better equipped to know which market our graphene is best suited for as well as how we can improve our quality further." ***/Anna Carlsson, CTO, Bright Day Graphene AB***

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