New Argonne Method Greatly Improves X-Ray Nanotomography Resolution

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Using the powerful X-ray beams of the Advanced Photon Source with new computer-driven algorithms, scientists will be able to study batteries and electronics at nanometer scales.

It's been a truth for a long time: if you want to study the movement and behavior of single atoms, electron microscopy can give you what X-rays can't. X-rays are good at penetrating into samples — they allow you to see what happens inside batteries as they charge and discharge, for example — but historically they have not been able to spatially image with the same precision electrons can.

But scientists are working to improve the image resolution of X-ray techniques. One such method is X-ray tomography, which enables non-invasive imaging of the inside of materials. If you want to map the intricacies of a microcircuit, for example, or trace the neurons in a brain without destroying the material you are looking at, you need X-ray tomography, and the better the resolution, the smaller the phenomena you can trace with the X-ray beam.

"We hope this will be a powerful tool for research at smaller and smaller scales." — Viktor Nikitin, Argonne National Laboratory

To that end, a group of scientists led by the U.S. Department of Energy's (DOE) Argonne National Laboratory has created a new method for improving the resolution of hard X-ray nanotomography. (Nanotomography is X-ray imaging on the scale of nanometers. For comparison, an average human hair is 100,000 nanometers wide.) The team constructed a high-resolution X-ray microscope using the powerful X-ray beams of the Advanced Photon Source (APS) and created new computer algorithms to compensate for issues encountered at tiny scales. Using this method, the team achieved a resolution below 10 nanometers.

"We want to be at 10 nanometers or better," said Michael Wojcik, a physicist in the optics group of Argonne's X-ray Science Division (XSD). "We developed this for nanotomography because we can obtain 3D information in the 10-nanometer range faster than other methods, but the optics and algorithm are applicable to other X-ray techniques as well.”

Using the in-house Transmission X-ray Microscope (TXM) at beamline 32-ID of the APS — including special lenses fashioned by Wojcik at the Center for Nanoscale Materials (CNM) — the team was able to use the unique characteristics of X-rays and achieve high-resolution 3D images in about an hour. But even those images were not quite at the desired resolution, so the team devised a new computer-driven technique to improve them further.

The main issues the team sought to correct are sample drift and deformation. At these small scales, if the sample moves within the beam, even by a couple nanometers, or if the X-ray beam causes even the slightest change in the sample itself, the result will be motion artefacts on the 3D image of the sample. This can make subsequent analysis much more difficult.

A sample drift can be caused by all kinds of things at that small a scale, including changes in temperature. To perform tomography, the samples also must be rotated very precisely within the beam, and that can lead to motion errors that look like sample drifts in the data. The Argonne team’s new algorithm works to remove these issues, resulting in a clearer and sharper 3D image.

“We developed an algorithm that compensates for the drift and deformation,” said Viktor Nikitin, research associate in XSD at Argonne. “When applying standard 3D reconstruction methods, we achieved a resolution in the 16 nanometer range, but with the algorithm we got it down to 10 nanometers.”

The research team tested their equipment and technique in several ways. First they captured 2D and 3D images of a tiny plate with 16-nanometer-wide features fabricated by Kenan Li, then of Northwestern University and now at DOE’s SLAC National Accelerator Laboratory. They were able to image tiny defects in the plate’s structure. They then tested it on an actual electrochemical energy storage device, using the X-rays to peer inside and capture high-resolution images.

Vincent de Andrade, a beamline scientist at Argonne at the time of this research, is the lead author on the paper. “Even though these results are outstanding,” he said, “there is still a lot of room for this new technique to get better."

The capabilities of this instrument and technique will improve with a continuing research and development effort on optics and detectors, and will benefit from the in-progress upgrade of the APS. When complete, the upgraded facility will generate high-energy X-ray beams that are up to 500 times brighter than those currently possible, and further advances in X-ray optics will enable even narrower beams with higher resolution.

"After the upgrade, we will push for eight nanometers and below," said Nikitin. "We hope this will be a powerful tool for research at smaller and smaller scales."

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