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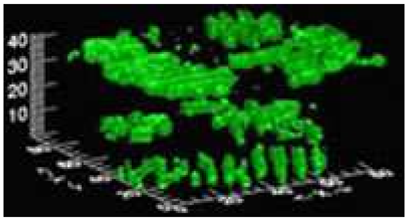
3D X-ray vision



Three-dimensional reconstruction of two identical nickel nanopatterns buried within a sample. The patterns were rotated with respect to each other by an angle of 65° and separated by 1 mm.

X-ray imaging has been used to probe the structure of materials for more than a century. But X-rays are difficult to manipulate and the focusing ability of X-ray lenses is poor, so the resolution of conventional X-ray images is limited. For crystalline materials, X-ray diffraction can be used to accurately determine the atomic structure to sub-ångstrom accuracy, but is less useful for non-crystalline materials and for irregular microstructures. Writing in *Physical Review Letters*, Jianwei Miao and colleagues report a technique that uses X-ray diffraction to generate images of irregular, non-crystalline nanostructures with a resolution down to 8 nm. This method can be used to reconstruct 3D images of nanostructures buried beneath a sample's surface.

Many techniques have been developed over recent years to image structures at the nanometre scale, but all have important limitations. Atomic force, scanning tunnelling and near-field microscopes are limited to imaging only surface structures, and transmission electron microscopes are appropriate only for samples that can be thinned down to less than 50 nm. Because of their small wavelength and ability to penetrate deep within a sample, X-rays are ideal for imaging structures well below a sample's surface.

As radiation of any form passes through a sample it can become scattered by objects, such as atoms, molecules and other internal structures, to form a diffraction pattern. This diffraction pattern represents a 2D projection of the Fourier transform of these objects within sample. One way of producing an image from this diffraction pattern is to focus the scattered radiation on to an image plane using a lens, such as the objective lens of a light or transmission electron microscope. But because of their high energy, small size and weak interaction with matter, focusing X-rays is difficult and the quality of X-ray lenses is relatively poor. As a result, the resolution of X-ray images obtained in this way is limited to around 30–50 nm.

Nonetheless, if researchers collect the diffraction pattern and use a computer to perform the reverse Fourier transform to reconstruct the image instead, they can avoid the resolution-limiting aberrations introduced by X-ray lenses. Such an approach has traditionally been limited to highly periodic crystalline materials. But by using a technique known as 'oversampling', Miao *et al.* are able to form images of irregular nickel nanostructures buried within a sample at a resolution of 8 nm. Moreover, by collecting diffraction patterns over a range of angles, they were able to form a 3D reconstruction of these nanostructures over a range of several micrometres at a resolution of 50 nm (see figure).

The ability to image objects at nanometre resolution and in 3D using X-rays has many important implications for both materials science and biology. Because this technique does not involve any complicated optics, images can therefore be collected over a much larger area than other techniques and it could be used to produce high-resolution images of whole cells or large subcellular structures in cell biology. And with the development of ultrashort intense pulsed X-ray sources, such as X-ray free electron lasers, the authors expect this technique could be used to image single protein molecules to a resolution of 0.25 nm.

High Resolution 3D X-Ray Diffraction Microscopy
JIANWEI MIAO, TETSUYA ISHIKAWA, BART JOHNSON, ERIK H. ANDERSON, BARRY LAI & KEITH O. HODGSON
We have imaged a 2D buried Ni nanostructure at 8 nm resolution using coherent x-ray diffraction and the oversampling phasing method. By employing a 3D imaging reconstruction algorithm, for the first time we have experimentally determined the 3D structure of a noncrystalline nanostructured material at 50 nm resolution. The 2D and 3D imaging resolution is currently limited by the exposure time and the computing power, while the ultimate resolution is limited by the x-ray wavelengths. We believe these results pave the way for the development of atomic resolution 3D x-ray diffraction microscopy.
Physical Review Letters **89,** 088303 (6 August 2002)

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letters to nature
Extending the methodology of X-ray crystallography to allow imaging of micrometre-sized non-crystalline specimens
JIANWEI MIAO, PAMBOS CHARALAMBOUS, JANOS KIRZ & DAVID SAYRE
The contrast and penetrating power afforded by soft X-rays when they interact with matter makes this form of radiation ideal for studying micrometre-sized objects. But although soft X-rays are useful for probing detail too fine for visible light microscopy in specimens too thick for electron microscopy, the highest-resolution applications of X-ray imaging have been traditionally limited to crystalline samples. Here we demonstrate imaging (at 75 nm resolution) of a non-crystalline sample, consisting of an array of gold dots, by measuring the soft X-ray diffraction pattern from which an image can be reconstructed. The crystallographic phase problem — the usually unavoidable loss of phase information in the diffraction intensity — is overcome by oversampling the diffraction pattern, and the image is obtained using an iterative algorithm. Our X-ray microscopy technique requires no high-resolution X-ray optical elements or detectors. We believe that resolutions of 10–20 nm should be achievable; this would provide an imaging resolution about 100 times lower than that attainable with conventional X-ray crystallography, but our method is applicable to structures roughly 100 times larger. This latter feature may facilitate the imaging of small whole cells or large subcellular structures in cell biology.
Nature **400,** 342–344 (22 July 1999)
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