

MICROSCOPY

X-ray nanovision

Eric D. Isaacs

Startling three-dimensional images of nanoparticles have been obtained with an X-ray microscope, showing crystal deformation in unprecedented detail. The trick is not to focus the X-rays, but to diffract them.

Imagine an imaging device with resolution more than 1,000 times better than an optical microscope and capable of seeing deep inside objects. Such is the promise of an X-ray microscope, which has been the dream of scientists since X-rays were discovered over a century ago. On page 63 of this issue¹, Ian Robinson and colleagues present a significant step towards realizing this dream, using the technique of coherent X-ray diffraction imaging. Their results demonstrate the power of X-ray microscopy to create three-dimensional images at the nanoscale, and indicate a clear path towards the ultimate X-ray microscope with atomic-scale resolution.

There is an acute need in nanotechnology for images with atomic-scale resolution. Because nanoparticles consist of relatively few atoms, their mechanical, electrical and thermodynamic behaviours are mainly determined by the particles' surfaces and interfaces with other materials, by defects such as those caused by atomic vacancies or non-native atoms, and by deformation of the atomic crystal lattice, known as strain. For example, nanoparticles of gold, which consist of just thousands of atoms, are excellent catalysts for certain chemical reactions, even though bulk gold is essentially inert². Semiconductor nanocrystals, known as quantum dots, have electronic and optical behaviour that can be tuned by binding a single molecule to their surface³. To develop materials with useful properties, we must first understand how these properties relate to atomic structure and surface chemistry.

Electron microscopes are ubiquitous in materials science, but X-ray microscopes are emerging as an attractive alternative, for two reasons. First, the penetrating power of X-rays into materials allows us to quantitatively determine the full three-dimensional structure of nanoparticles, usually non-destructively. High-resolution electron microscopy often requires samples to be thinned to several atomic layers. This is technically challenging for many materials and can alter the properties being studied. Embedded nanostructures can best be studied with X-rays.

Second, bright synchrotron sources of 'hard' X-rays — those with a wavelength shorter than 0.1 nm — have become readily available. This has enabled the development of X-ray optics with improved resolving power. So far, microscopes based on Fresnel zone plates (which focus light by diffraction) or mirror optics

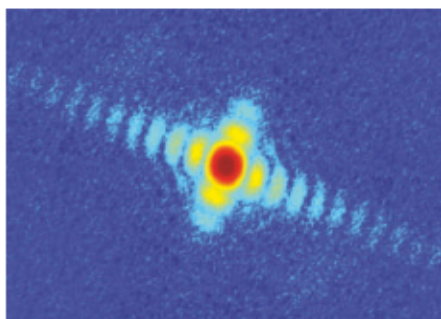


Figure 1 | Coherent X-ray diffraction. The pattern created by the diffraction of coherent X-rays from a 160-nm cube of silver^{10,11}. This may be mathematically 'inverted' to produce an image of the cube.

have produced the highest resolution (about 40-nm) hard X-ray images. But the optics used by these methods are technologically challenging to make^{4,5}. Coherent X-ray imaging, which uses in-phase X-rays, offers a 'lens-less' alternative.

When coherent X-rays irradiate an object, the scattered radiation forms a pattern. In contrast to the scattering produced with incoherent X-rays, the coherent pattern contains not only information about the amplitude of the scattered radiation, but also information about the phase of that radiation, provided that the pattern is sufficiently 'oversampled'⁶. Oversampling is a well-established technique used to ensure proper retrieval of phase information from the scattered radiation. Crucially, the phase information allows the pattern to be mathematically 'inverted' to recover an image of the charge-density distribution of the object. One significant feature of coherent-diffraction imaging is its high sensitivity to crystallographic strain — that is, to deviations of atoms from their nominal bulk positions.

Coherent X-ray imaging was proposed in the early 1980s as a means to visualize the electron-density distribution in non-crystalline materials⁷, and was first demonstrated in 1999 (ref. 8). Two years later, the technique was used by Robinson *et al.*⁹ on a crystalline system. Figure 1 shows an example of the coherent diffraction pattern from a 160-nm silver cube, observed in previous work by Robinson and colleagues^{10,11}. The four symmetric streaks are signatures of the cubic facets. These streaks are produced by sharply terminated surfaces and could be formed with incoherent X-rays. Coherence is, however, required to produce the periodic modulation of the streaks.

The modulation is an interference pattern created by X-rays coherently scattered from opposing sides of the cubes, much like the interference pattern in the classic two-slit experiment of Thomas Young. This pattern was inverted to create an image of the cube^{10,11}.

Robinson and colleagues¹ have now applied their technique to more complicated structures. They collected the coherent diffraction pattern from 750-nm hemispherical lead nanoparticles that had small faceted faces. The pattern was then inverted by applying computational techniques developed by the authors, to obtain a full three-dimensional image of the electron-density distribution in the particles.

A truly remarkable aspect of this image is the clear articulation of a deformation of the lattice, known as a strain field (see Figs 3 and 4 of ref. 1). When contour maps of the magnitude of the strain are plotted, it becomes apparent that the strain field originates from a point just outside the lead particle, in the region of contact with the glass substrate upon which the particle was grown. This strongly supports the authors' claim that the strain field is caused by interfacial contact forces between the particle and the substrate. These forces can be associated with a single defect that may have been the nucleation point for the growth of the nanocrystal.

By creating a lens-less image of the strain fields in a lead nanoparticle with 40-nm spatial resolution, Robinson and co-workers have taken a substantial step forward in realizing the potential of X-ray microscopes. One can envisage even greater uses, for instance imaging the strain field near a single atomic defect inside a semiconductor quantum dot. Such applications will require significant improvements in detectors and a much higher flux of coherent X-rays, which will become available with future generations of hard X-ray sources, such as the X-ray Free Electron Laser. With these advances, the dream of a microscope capable of imaging the position and type of every atom in a nanocrystal or macromolecular assembly could become a reality. ■

Eric D. Isaacs is in the Argonne National Laboratory, Center for Nanoscale Materials, Argonne, Illinois 60439, USA, and the James Franck Institute, University of Chicago. e-mail: isaacs@anl.gov

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