

X-ray microscopy techniques for nanostructure analysis.

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X-rays have been used for a long time to characterize nanostructures. Shapes and sizes of nanoparticles are routinely determined by small and large angle X-ray scattering. Elemental and chemical information is gained from X-ray fluorescence and absorption spectroscopy, respectively. These techniques are useful to study ensemble averages of macroscopic quantities of the specimen. Recent advances are starting to make it possible to use X-ray microscopy to study clusters, or even individual nanoparticles in isolation, or as parts of a larger system.

A major advantage of using X-rays is the penetrating power of the radiation. The specimen may be relatively thick (e.g. nanoparticles embedded in a matrix, or a complex structure made up of nanoparticles). In addition, fluorescence and absorption spectra are beginning to be collected from individual nanostructures, extending the elemental and chemical analysis capabilities to the nano-world.

Several approaches to X-ray microscopy of nanostructures are under development. [1]

- Zone plates are used to form nanoprobe for scanning microscopy and spectromicroscopy [2].
- Other forms of X-ray optics, such as Kirkpatrick-Baez mirror systems are rapidly improving in resolution, and offer broad spectral tunability in microprobe applications [1].
- Zone plates are also used as objective lenses in full-field microscopes, and the resolution in this form of imaging has already reached the 15 nm level [3].
- The technique of Diffraction Microscopy, where the diffraction pattern of a non-crystalline specimen is recorded, and the object is reconstructed by an iterative algorithm [4] dispenses with X-ray optics altogether.

The ultimate limitation to the finest spatial resolution one can obtain in X-ray microscopy is either radiation damage [5] or, in the most radiation-hard specimens, it is the wavelength of the X-rays. Where multiple identical copies of the specimen can be obtained, and arrayed in a regular structure as in a crystal, the radiation dose can be shared among the many repeats, and atomic resolution is possible, as demonstrated by crystallographers on a routine basis. But what if the identical objects refuse to crystallize? With a suitably powerful X-ray laser with femtosecond pulse duration, one should still be able to exceed the radiation damage limit, by recording the diffraction pattern before the nanoparticle or molecule explodes. [6] Such a laser, the LCLS, is under construction at the Stanford Linear Accelerator Center [7]. If the diffraction pattern from a single object is not strong enough to allow reconstruction at the desired resolution, a stream of identical objects may be used. In this case the diffraction patterns need to be sorted depending on the orientation of the specimen, much as in electron-cryo-tomography [8]. Alternatively one may consider ways of pre-aligning the identical specimens to simplify the analysis. [9].

Related to this last scheme is the suggestion by Spence and Doak [10]. They point out that if a large collection of identical, non-crystallizable specimens is available these may be aligned using the field of an intense infrared laser. It may then be possible to do “serial crystallography” [10], where the radiation dose to any one specimen is small enough that the diffraction pattern may be collected using a conventional synchrotron X-ray beam as the laser-aligned nanostructures or macromolecules stream by. The first tests of this idea are scheduled for the near future.

Not only is X-ray microscopy suitable for the characterization of nanostructures, nanoparticles are also used as labels or markers in X-ray microscopy and tomography. Nanodots made of heavy metals are particularly useful, since these are easily identified, and can be functionalized to label structures of interest. [11], [12]

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