From the bulk of the space shuttle to the miniature wonder of microprocessors, material science is behind many of today's scientific wonders. Understanding the shape, orientation, and distortion of crystal grains such as those in ceramics, metals, and semiconductors is critical to the next generation of wonders, including the expanding frontier of nanoscience. X-rays have long been the favored tool for scientists interested in 2-D mapping of materials, but point-to-point 3-D mesoscale probes of materials on the order of microns or tenths of microns have been limited to one-shot destructive analyses and best-estimate computer models.

A new technique from researchers at Oak Ridge National Laboratory (ORNL; Oak Ridge, TN) uses knife-edge profiling with a microbeam from the Advanced Photon Source at Argonne National Laboratory (Argonne, IL) to create 3-D images of crystalline structures with submicron resolution. The technique, called differential aperture x-ray microscopy (DAXM), isolates Laue diffraction patterns created by the interaction of x-rays with crystal grains. The distribution of the Laue pattern relates to the shape, orientation, and distortion of the intra- and intergranular crystal-grain relationship—and perhaps more important, how these factors change as outside forces put strain on the sample.

When broadband x-rays (6 keV to 25 keV) impinge on a crystal lattice, the interaction creates Laue diffraction spots for many wavelengths. In the case of incoherent x-ray illumination such as used in this experiment, the diffracted light does not interfere with itself. The Laue spot patterns are typically read by an x-ray-sensitive CCD camera, revealing crystalline structure to depths of tens of microns in dense materials and up to a millimeter in light materials. Unfortunately, the grains in polycrystalline samples create dozens of these patterns, each superimposed on top of the other in a pattern too complex to analyze.

Enter Ben Larson and Gene Ice of ORNL, who adopted a knife-edge profiling technique to create a scanning pinhole camera, or more specifically, a DAXM system. In knife-edge profiling, an absorbing object is stepped across the illumination beam in small, well-defined increments. As the beam is occluded, the radiation drops off in increments. The resultant data not only reveals beam width in metrology applications, but it also can be used to unravel superimposed Laue patterns.

In their system, Larson and Ice step a 50-µm-diameter x-ray-absorbing wire in 0.5-µm increments across the front of a metal or crystalline sample, occluding individual diffraction patterns before they can be read by the camera (see figure). By subtracting the image of the Laue patterns after each step from the previous image and using ray-trace algorithms that take into account the position of the incident beam, the wire, and relevant 20-µm pixels on the CCD detector, the system can determine the point of origin for a particular Laue pattern. If the wire is much closer to the sample than the CCD, one can resolve the crystalline structure to a couple of tenths of a micron.

After the source of individual rays for one position of the microbeam on the sample is isolated, the process is repeated along the beam. Software then reconstructs full Laue patterns from each micron along the beam. By comparing the patterns against stored diffraction patterns of known crystal structures, DAXM constructs a computer image of the size and shape of each grain, its orientation to other grains, and inter- and intragranular distortion or stress.

Although Larson and Ice have used the method to look at grains in aluminum, the applications and materials that could benefit from the procedure are many. “[DAXM] is a novel technique and is considered to be the only ‘nondestructive’ technique capable of measuring strain, orientation, and stress with submicrometer resolution,” says Anter El-Azab at Pacific Northwest National Laboratory (Richland, WA). “The method is general and can be expected to be adaptable to studies of crystalline biological materials such as bone and marine shells, geological materials, etc. In principle, any diffraction information available can be obtained with this resolution, so there will likely be a wide range of applications as the technique matures.”

Further development should yield a decrease in measurement time and an increase in resolution. “[There are] enormous applications in the field of nanoscience and nanotechnology, once the spatial resolution gets down to the 100-nm or less range,” El-Azab says. — Winn Hardin