"The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes."

ATOM PROBE TOMOGRAPHY: A TUTORIAL

M. K. Miller

Metals and Ceramics Division, Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6376

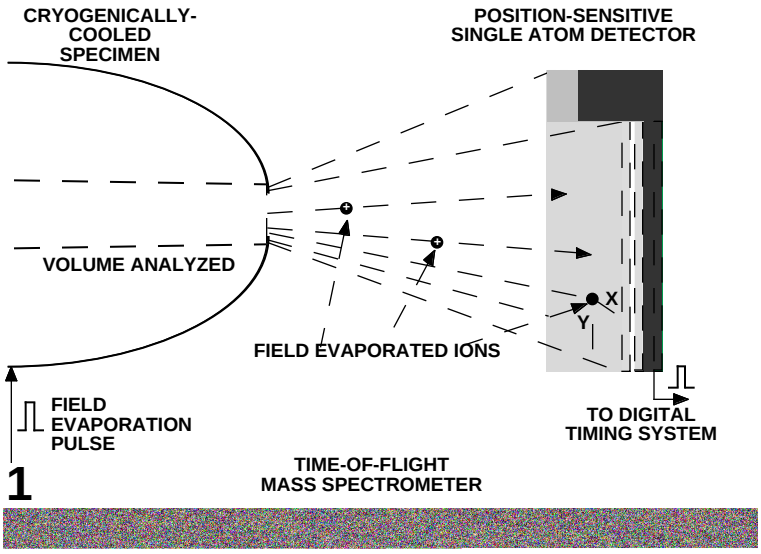
Atom probe tomography (APT) is an ultrahigh resolution microanalytical technique that enables the spatial coordinates and elemental identities of the atoms in a small volume of material to be determined.[1] The specimen volume that may be analyzed typically has an area of ~10 to 20 nm by 10 to 20 nm and is ~100 to 250 nm deep, and contains up to ~1 million atoms. The distribution of the solute atoms within this volume may then be reconstructed from these data. The compositions of small volumes are determined by simply counting the number of atoms of each type within that volume, and thus the technique provides a fundamental measure of local concentrations. Atom probe tomography requires that the specimen has some electrical conductivity and may be applied to almost all metals and alloys, many semiconductors, and some electrically conducting ceramics. The sharp needle-shaped specimens may be fabricated from bulk and thin film materials with the use of electropolishing, chemical or ion milling methods.

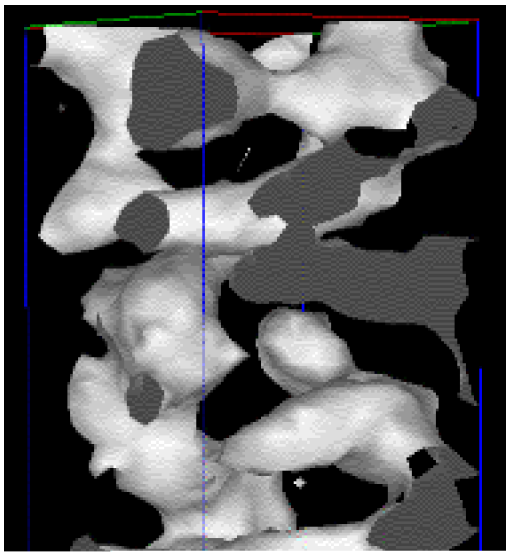
The instrument, known as a three-dimensional atom probe (3DAP), consists of an extremely sharp, needle-shaped specimen and a time-of-flight mass spectrometer equipped with a position-sensitive detector that is able to detect single atoms, Fig. 1. Several different types of position-sensitive detectors have been used, including CCD cameras, wedge-and-strip anodes, multianodes, and combinations thereof. The surface atoms of a cryogenically cooled specimen are individually ionized by the superposition of a short (10 ns), high-voltage pulse onto the standing voltage on the specimen. These ions are then radially projected from the specimen toward a position-sensitive detector. The specimen surface is magnified by a factor of ~5 million at the single-atom detector. The position-sensitive detector provides the x and y coordinates of each field evaporated atom. The z coordinate is determined from the order in which the atoms are detected. This sequential atom-by-atom evaporation results in a volume of analysis. The identities of the field evaporated ions are determined from their flight times in the mass spectrometer. The mass spectrometer has sufficient mass-resolving power to distinguish the individual isotopes of all elements.

The three-dimensional data acquired in the three-dimensional atom probe may be visualized and analyzed by a variety of techniques. The simplest representation is to use a dot or sphere to represent the positions of selected solute atoms, as shown in Fig. 2. This method is effective in cases where the matrix has a relatively low solute content and features in the analyzed volume such as precipitates, interfaces and boundaries exhibit significantly higher solute levels. An impressive example of boron segregation to a dislocation in FeAl has recently been published.[2,3] Another common visualization method is to construct isoconcentration surfaces through the three-dimensional data. Isoconcentration surfaces are created by dividing (or gridding) the analyzed volume into small volume elements, Fig. 3, and estimating the solute concentration of each volume element from the number of ions of each type in that volume element. Surfaces are then constructed through a selected solute concentration, as shown in Fig. 4. Both of these visualization methods permit parameters such as the size, shape and number density of the features present in the analyzed volume to be estimated. An alternative method to examine the three-dimensional data for features such as small precipitates is to locate all atoms of one or more species that are within a selected distance of another atom of that species. As the positions of all the atoms in the precipitates are experimentally evaluated, parameters such as the center of mass, radius of gyration and Guinier radius of each precipitate may be estimated. Once features are located, linear and radial composition profiles through the features may be constructed, as shown in Fig. 5.

The information in the volume elements may also be used to statistically analyze the three-dimensional data. For example, it may be use to create autocorrelations to estimate the size of features, as shown in Fig. 6. In addition, this information may be accumulated into frequency distributions, which may be statistically analyzed to estimate parameters such as the compositions of coexisting phases. Topological parameters such as handle density may also be estimated from the three-dimensional data.[4]

1. M. K. Miller, Atom Probe Tomography, Kluwer Academic/Plenum Publishers, New York, 2000.
2. D. Blavette, E. Cadel, A. Fraczkiewicz and A. Menand, *Science*, 286 (1999) 2317.
3. M. K. Miller, *Science*, 286 (1999) 2285.
4. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.





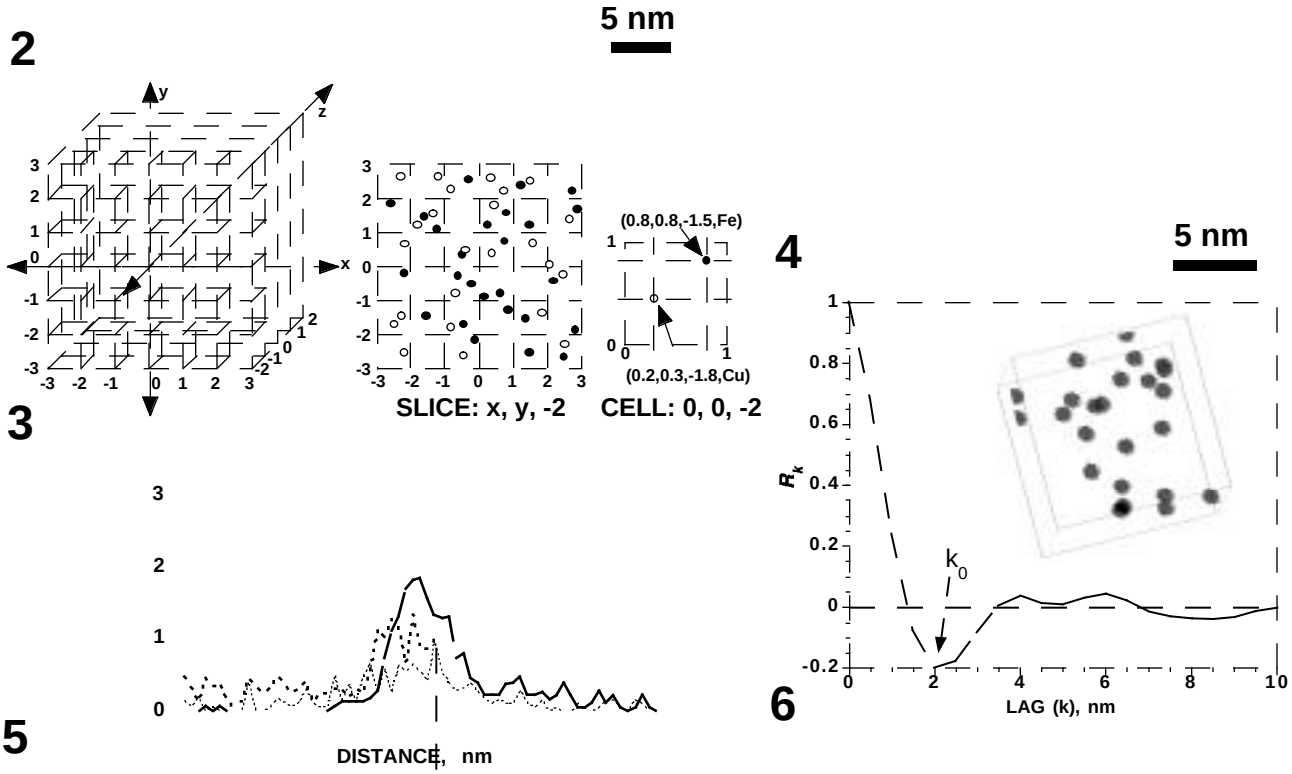


FIG. 1. Schematic diagram of a three-dimensional atom probe.
FIG. 2. Atom map of a volume in an irradiated pressure vessel steel. Each dot represents an individual atom.
FIG. 3. Method used to bin the three-dimensional data into volume elements.
FIG. 4. Chromium isoconcentration surface depicting the α′ phase in a spinodally decomposed Fe-32% Cr alloy.
FIG. 5. Composition profiles perpendicular to the grain boundary for the box shown in Fig. 2.
FIG. 6. Autocorrellogram of a volume containing small spherical precipitates. k_0 estimates precipitate diameter.