SPATIAL RESOLUTION IN THE ATOM PROBE FIELD ION MICROSCOPE

M. K. Miller

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

The atom probe field ion microscope can resolve and identify individual atoms. This ability is demonstrated in a pair of field ion micrographs of an Ni_3Al specimen, Fig. 1, in which the individual atoms on the close packed (111) plane are clearly resolved. Comparison of these two micrographs reveals that an individual atom was field evaporated between the micrographs. Due to the hemispherical nature of the specimen, the ability to resolve this two dimensional atomic arrangement is only possible on low index plane facets. The spatial resolution in field ion images is determined by a number of factors including specimen temperature, material, microstructural features, specimen geometry, and crystallographic location.¹

The spatial resolution of the data obtained in atom probe and 3 dimensional atom probe compositional analyses can be evaluated with the use of field evaporation or field desorption images. The field evaporation images are formed from the surface atoms with the use of a single atom sensitive detector whereas the field ion image is formed from the projection of a continuous supply of ionized image gas atoms. Field ion and field evaporation micrographs of a grain boundary in Ni_3Al are shown in Fig. 2. The bright band in the field evaporation micrograph, Fig. 2b, indicates that significant trajectory aberrations are present within ~ 1 nm of the boundary. In some cases, both bright and dark regions are observed along a boundary indicating that there is both compression and dilation of the ion trajectories in adjacent regions. In pure elements, bright and dark lines are often observed along the zone lines in field evaporation patterns. However, these lines are not generally observed in alloys that contain significant levels of solutes. There is also a slight difference from the positions of the atoms in field ion image and the field evaporation image. These "aiming errors" are mainly due to the difference in position where image gas and the specimen ions originate and how their trajectories are affected as they leave the specimen, as shown in Fig. 3. If different phases are present in the specimen, slight differences in their field evaporation behavior leads to differences in their local magnification with a resulting compression or dilation of the ion trajectories, as shown schematically in Fig. 4. The spatial resolution associated with an atom probe analysis is defined in terms of lateral and depth components of the volume collected. The lateral component is defined primarily by local magnification effects, trajectory aberrations and in a probe aperture type of atom probe, the effective size of the probe aperture (<1 nm to ~ 8 nm). These factors limit the lateral spatial resolution to ~ 1 nm.

The spatial resolution in the third orthogonal dimension is significantly better than the lateral resolution. For example, the true atomic depth resolution of the technique in this dimension may be demonstrated in a composition profile in which the probe aperture was positioned near the center of an (001) pole in an L1_2 -ordered Ni_3Al specimen. The demarcation between the pure nickel and the mixed nickel plus aluminum planes is clearly evident in Fig. 5. The slight variation in composition between each similar layer is simply a result of the counting statistics due to the small number of atoms collected on each plane. The atomic planes are also evident in a 3 dimensional atom map obtained from a nickel-based superalloy in the tomographic atom probe, as shown in Fig. 6. However, planes are only evident in these reconstructions if their normals are within $\sim 10^\circ$ of the detector normal and the plane has a relatively low index. This indicates that small ion trajectory aberrations are present.²

1. M. K. Miller, A. Cerezo, M. G. Hetherington and G. D. W. Smith, *Atom Probe Field Ion Microscopy*, Oxford University Press, Oxford, 1996.
2. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at Oak Ridge National Laboratory and the tomographic atom probe at the University of Rouen.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

dg

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

FIG. 1. Field ion micrographs of Ni_3Al showing individual atoms on the 111 plane.
 FIG. 2. a) Field ion and b) field desorption image showing trajectory aberrations at an interface.
 FIG. 3. Differences in trajectories for image gas and surface atoms.
 FIG. 4. Schematic diagram showing the origin of local magnification differences between phases.
 FIG. 5. Composition profile along an [001] direction in Ni_3Al revealing the alternating Ni and Ni+Al planes
 FIG. 6. (001) atomic planes resolved in a nickel-based superalloy in the tomographic atom probe. Each sphere represents an individual atom. (Data courtesy D. Blavette and F. Danoix).

SPATIAL RESOLUTION IN THE ATOM PROBE FIELD ION MICROSCOPE

M. K. Miller

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

The atom probe field ion microscope can resolve and identify individual atoms. This ability is demonstrated in a pair of field ion micrographs of an Ni_3Al specimen, Fig. 1, in which the individual atoms on the close packed (111) plane are clearly resolved. Comparison of these two micrographs reveals that an individual atom was field evaporated between the micrographs. Due to the hemispherical nature of the specimen, the ability to resolve this two dimensional atomic arrangement is only possible on low index plane facets. The spatial resolution in field ion images is determined by a number of factors including specimen temperature, material, microstructural features, specimen geometry, and crystallographic location.¹

The spatial resolution of the data obtained in atom probe and 3 dimensional atom probe compositional analyses can be evaluated with the use of field evaporation or field desorption images. The field evaporation images are formed from the surface atoms with the use of a single atom sensitive detector whereas the field ion image is formed from the projection of a continuous supply of ionized image gas atoms. Field ion and field evaporation micrographs of a grain boundary in Ni_3Al are shown in Fig. 2. The bright band in the field evaporation micrograph, Fig. 2b, indicates that significant trajectory aberrations are present within ~ 1 nm of the boundary. In some cases, both bright and dark regions are observed along a boundary indicating that there is both compression and dilation of the ion trajectories in adjacent regions. In pure elements, bright and dark lines are often observed along the zone lines in field evaporation patterns. However, these lines are not generally observed in alloys that contain significant levels of solutes. There is also a slight difference from the positions of the atoms in field ion image and the field evaporation image. These "aiming errors" are mainly due to the difference in position where image gas and the specimen ions originate and how their trajectories are affected as they leave the specimen, as shown in Fig. 3. If different phases are present in the specimen, slight differences in their field evaporation behavior leads to differences in their local magnification with a resulting compression or dilation of the ion trajectories, as shown schematically in Fig. 4. The spatial resolution associated with an atom probe analysis is defined in terms of lateral and depth components of the volume collected. The lateral component is defined primarily by local magnification effects, trajectory aberrations and in a probe aperture type of atom probe, the effective size of the probe aperture (<1 nm to ~ 8 nm). These factors limit the lateral spatial resolution to ~ 1 nm.

The spatial resolution in the third orthogonal dimension is significantly better than the lateral resolution. For example, the true atomic depth resolution of the technique in this dimension may be demonstrated in a composition profile in which the probe aperture was positioned near the center of an (001) pole in an L1_2 -ordered Ni_3Al specimen. The demarcation between the pure nickel and the mixed nickel plus aluminum planes is clearly evident in Fig. 5. The slight variation in composition between each similar layer is simply a result of the counting statistics due to the small number of atoms collected on each plane. The atomic planes are also evident in a 3 dimensional atom map obtained from a nickel-based superalloy in the tomographic atom probe, as shown in Fig. 6. However, planes are only evident in these reconstructions if their normals are within $\sim 10^\circ$ of the detector normal and the plane has a relatively low index. This indicates that small ion trajectory aberrations are present.²

1. M. K. Miller, A. Cerezo, M. G. Hetherington and G. D. W. Smith, *Atom Probe Field Ion Microscopy*, Oxford University Press, Oxford, 1996.
2. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at Oak Ridge National Laboratory and the tomographic atom probe at the University of Rouen.

3

4

5

6

FIG. 1. Field ion micrographs of Ni_3Al showing individual atoms on the 111 plane.
 FIG. 2. a) Field ion and b) field desorption image showing trajectory aberrations at an interface.
 FIG. 3. Differences in trajectories for image gas and surface atoms.
 FIG. 4. Schematic diagram showing the origin of local magnification differences between phases.
 FIG. 5. Composition profile along an [001] direction in Ni_3Al revealing the alternating Ni and Ni+Al planes
 FIG. 6. (001) atomic planes resolved in a nickel-based superalloy in the tomographic atom probe. Each sphere represents an individual atom. (Data courtesy D. Blavette and F. Danoix).