DOI: 10.1017/S1431927605502034
Microsc Microanal 11(Suppl 2), 2005

Three-dimensional Nanoanalysis with the Local Electrode Atom Probe

M.K. Miller*

*Metals and Ceramics Division, Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6136

The commercial introduction of the local electrode atom probe (LEAP®) in 2003 has significantly increased the volume that may be analyzed with atom probe tomography (APT) [1] and dramatically reduced the time required to acquire the data. Data sets from this instrument typically contain between 0.25 and 100 million atoms. APT experiments can now be performed in a few minutes rather than days. The large field of view, typically 50-100 nm, also enables a larger diversity of microstructural features to be investigated: coarse (~50 nm) and fine (~1 nm) features or low number density features such as dislocations in the vicinity of a strengthening precipitate and segregation at boundaries. The fundamental information obtained with this instrument is the atomic coordinates and the mass-to-charge ratio of the atoms in the sampled volume. This three-dimensional (3D) information may be processed in a variety of ways. For example, a carbon atom map of a Cottrell atmosphere at a dislocation in steel is shown in Fig. 1. The lateral extent of the carbon segregation was estimated to be ~7 nm and the local level of carbon was found to vary between 5 and 10%.

An atom map obtained from an Alloy 718 nickel base superalloy showing Al, Ti, Nb, Cr and Fe atoms is shown in Fig. 2. A number of ~11-nm-diameter ellipsoidal precipitates are evident from the non-uniform distribution of the solute atoms in this thin slice. Close examination of the solute distribution within these precipitates reveals that they consist of Al- and Ti-enriched regions characteristic of the $L1_2$-ordered Ni_3(Al,Ti,Nb) γ′ phase and Nb- and Ti-enriched regions characteristic of the $D0_{22}$-ordered Ni_3(Nb,Ti,Al) γ″ phase. The coherent interface between these γ′ and γ″ regions is on the {001} planes. The continuation of (001) planes reveals that these precipitates are coherent with and have a cube-on-cube orientation relationship with the face centered cubic Cr- and Fe-enriched γ matrix.

The 3D morphology and distribution of the phases may be visualized with the use of isoconcentration surfaces. An aluminum isoconcentration surface constructed from a 13 million atom dataset from a crept CMSX-4 superalloy, Fig. 3, reveals a coarse primary γ′ precipitate, an adjacent precipitate-free zone, and secondary γ′ precipitates that increase in size with distance into the central region of γ matrix. A 10-nm-high step is evident on the surface of the primary γ′ precipitate. The solute gradients of all the elements with respect to any of the microstructural features or interfaces may be examined by subsampling the volume. For example, a concentration profile constructed normal to a γ-γ′ interface, Fig. 4, reveals partitioning of Cr, Co, Re and Mo to the γ matrix and Al, Ti and Ta to the γ′ precipitate. In addition, 10 to 20 nm wide solute depleted and solute enriched regions are evident on both sides of the γ-γ′ interface. Some fine secondary γ′ precipitates are also evident in the γ matrix [2].

[1] M. K. Miller, *Atom Probe Tomography*, Kluwer Academic/Plenum Press, New York, 2000.
[2] Research at the SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. LEAP® is a registered trademark of Imago Scientific Instruments Corp.

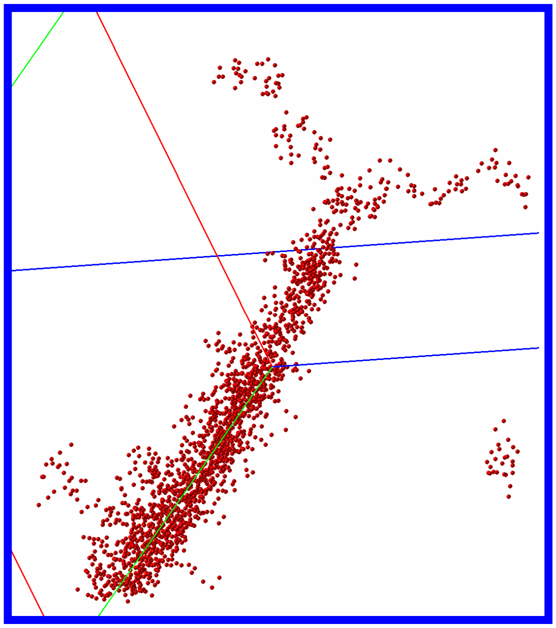

FIG. 1. Carbon atom map of a Cottrell atmosphere (~7 nm diameter, 5-10 at.% C) in steel. Courtesy Prof. E. Pereloma, Monash University.

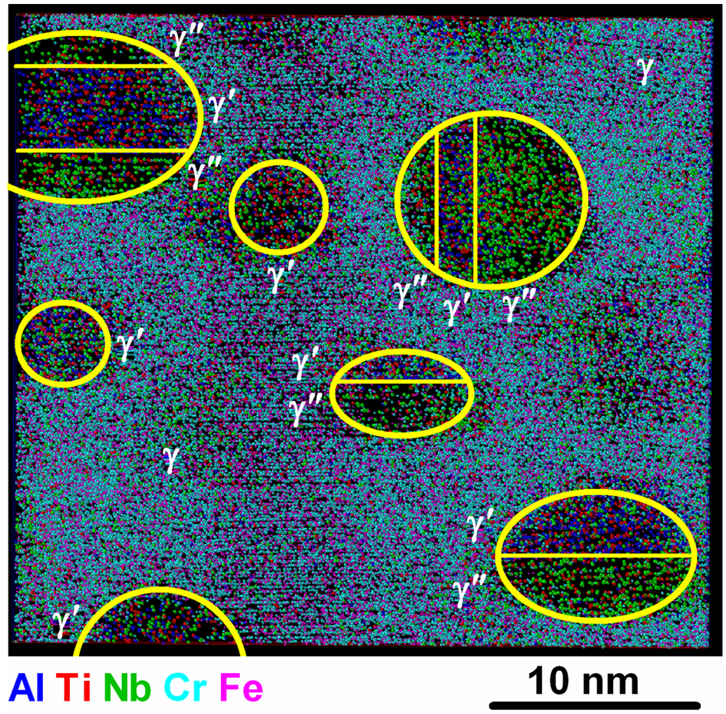

FIG. 2. Atom map of γ′/γ″ precipitates in 718 nickel-base superalloy. The 001 planes are evident.

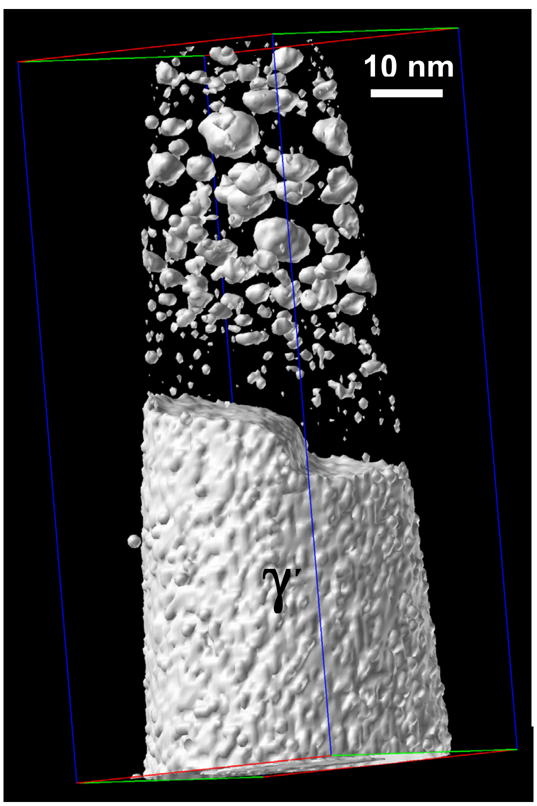

FIG. 3. Al isoconcentration surfaces showing primary and secondary γ′ precipitates in a crept CMSX4 nickel-base superalloy.

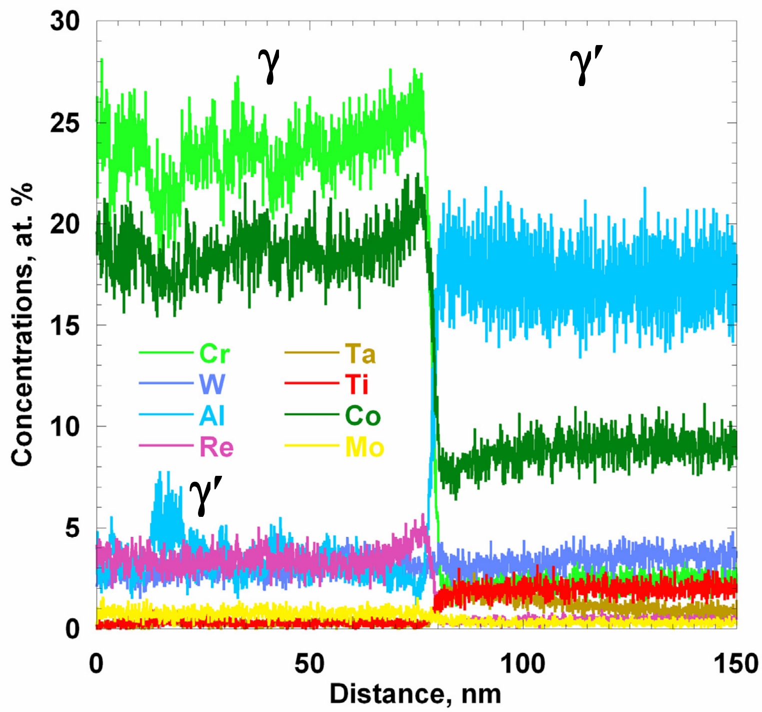

FIG. 4. Concentration profiles constructed from the γ matrix into the primary γ′ precipitates shown in Fig. 3 revealing the partitioning of the alloying elements and narrow concentration gradients near the γ-γ′ interface. Some secondary γ′ precipitates are intersected in the γ matrix.

https://doi.org/10.1017/S1431927605502034 Published online by Cambridge University Press