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Measuring Chemical Segregation at Grain Boundaries by Atom Probe Tomography

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Many of the properties of materials, whether they are metals, ceramics or semiconductors, are governed by the interfaces such as grain boundaries and hetero-phase boundaries that are present within the microstructure. The chemistry of these interfaces is of particular importance as it provides a possible handle to control materials properties, e.g. corrosion resistance, electrical or thermal conductivity, and fracture toughness. Measuring grain boundary segregation has traditionally been addressed using Auger spectroscopy or transmission electron microscopy (TEM). More and more often, atom probe tomography (APT) is employed due to its ability to map solute segregation in three dimensions and its higher sensitivity to light elements over TEM for instance. The development of routine specimen preparation has also made the use of APT for grain boundary a sensible approach.

While APT might offer a unique opportunity to access atom-by-atom information with very high chemical and spatial resolution that no other technique can match [1], challenges remain for multi-component materials and interfaces in terms of chemical and spatial resolution. For instance, the so-called local magnification effect is a well-known artifact arising from differences in evaporation fields of adjacent phases. These differences can lead to ion trajectory overlaps near the interface and loss of spatial resolution [2]. A few studies have been performed to understand the effects of varying evaporation fields on 3D reconstruction of microstructures containing small clusters or multi-layers [3,4].

It is conceivable that the same evaporation artefacts affect the analysis of grain boundaries. Therefore, accurate 3D reconstruction from grain boundaries by APT requires a careful analysis of the evaporation behavior and the possible effects on solute atom positioning. Such an understanding will provide a unique method to estimate concentration accuracy at grain boundaries as well as inversing the APT transfer function to go back to the actual solute distribution at the original grain boundary. We first address this question by investigating the evaporation behavior of a $\Sigma 3$ grain boundary in a simple binary system Fe-15Cr alloy both experimentally and theoretically.

A $\Sigma 3$ boundary is first found by Electron Backscatter Diffraction (EBSD) analysis. APT specimens containing the very same boundary are then prepared by lift-out and focused ion beam (FIB) milling in such a way that the boundary is positioned at different angles with respect to the axes of needle-shaped APT specimen. SEM images and 3-D reconstructions of specimens with the grain boundary at two different orientations (parallel and perpendicular with respect to tip axis) are shown in Figure 1 and 2, respectively. 1-D concentration profiles of Cr atom taken perpendicular to the grain boundary for both orientations are shown in Figure 1c and 2c. The appearance of Cr segregation measured by APT is clearly a function of the orientation of the grain boundary with respect to the tip axis directly indicating reconstruction artefacts.

The evaporation behavior at this model grain boundary is investigated by field evaporation modeling [4]. Comparison between the experimental data and the simulation results will be presented and discussed.

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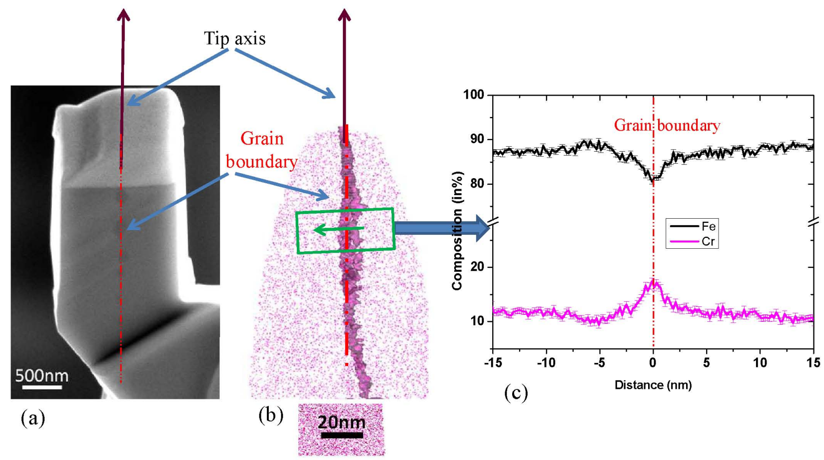

Figure 1. Grain boundary parallel to tip axis: (a) SEM image of lift-out (b) Reconstructed volume analyzed by APT showing Cr segregation and (c) 1 D profile of Fe and Cr atoms across grain boundary

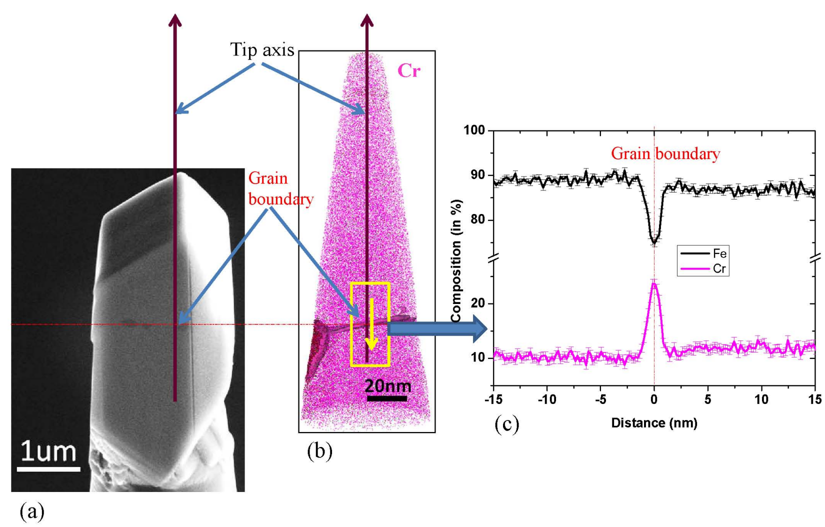

Figure 2. Grain boundary perpendicular to tip axis: (a) SEM image of lift-out (b) Reconstructed volume analyzed by APT showing Cr segregation and (c) 1 D profile of Fe and Cr atoms across grain boundary

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