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Grain Boundary Analysis of Inconel 718 Using a Novel Atom Probe Design

Katherine P. Rice, Robert M. Ulfig, Yimeng Chen, Ty J. Prosa and David J. Larson

[1.] CAMECA Instruments Inc, Madison, WI USA

In this work, we present a study of correlative grain boundary segregation analysis for an Inconel 718 superalloy using a newly-designed atom probe. The EIKOS™ atom probe provides a simpler design than the CAMECA's Local Electrode Atom Probe (LEAP™) and is primarily targeted towards metallurgical applications. The specimen carrier uses a wire geometry, which is well-suited for electropolishing methods of sample preparation, but can also accommodate focused ion beam (FIB) prepared liftout specimens. In addition, the EIKOS™ atom probe also offers a novel design where the specimen and counter-electrode are integrated within the same specimen carrier. The specimen-counter electrode assembly is aligned *ex-situ*, allowing for no moving stage parts within the UHV analysis chamber. The specimen assembly is attached directly to a cryogenic cooler during analysis, providing high data quality without the need for complicated cryogenically cooled moving stage parts. A 532 nm green laser can also be used to assist field evaporation of poor electrical conductivity materials such as semiconductors and oxides. Data analysis is performed using CAMECA's Integrated Visualization and Analysis Software (IVAS™).

For this grain boundary study, correlative techniques were used to study segregation behavior in a variety of different grain boundary types of Inconel 718. Electron backscatter diffraction (EBSD) was performed on a highly polished surface. Grain boundaries with coincident site lattice (CSL) values of $\Sigma 3$, $\Sigma 9$, and $\Sigma 27a$ were located on the sample surface from the EBSD analysis, and FIB liftout techniques were used to mount the grain boundary containing specimens to a wire-shaped substrate. Transmission EBSD was used during the sharpening process to position the grain boundary in the final needle-shaped specimen [1]. Figure 1a shows the EBSD map highlighting the $\Sigma 9$ grain boundary. Figures1 b and c show the sharpening process. Figure 1d shows a transmission EBSD map highlighting the $\Sigma 9$ grain boundary position within the final needle-shaped specimen.

Ion distribution maps from atom probe analysis are shown in Figure 2a-c for Inconel 718 [3]. The density map in Figure 2a shows a density discontinuity at the grain boundary position because of differing field evaporation characteristics at the boundary. The density map confirms collection of the grain boundary for $\Sigma 3$ boundary where no segregation was observed. $\Sigma 9$ and $\Sigma 27$a show clear boron segregation on them. Gibbsian interfacial excess values show segregation to be inversely correlated with energy of CSL grain boundaries [2].

References:

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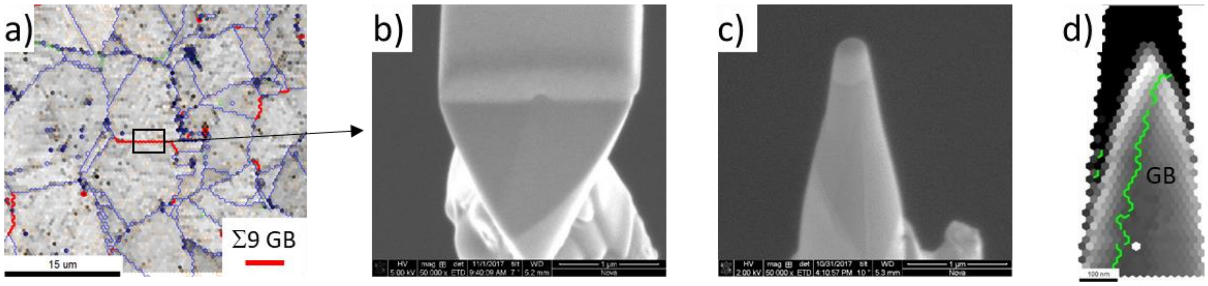

Figure 1. a) Σ9 grain boundary highlighted in reflection EBSD map. b) Specimen wedge mounted to wire post. Grain boundary in mount is visible in cross-sectional view. c) Ion beam sharpening to target the grain boundary. Grain boundary contrast near the apex is lost due to smaller electron interaction volume when sharpening. d) Transmission EBSD map taken from final tip shape, confirming the presence of Σ9 grain boundary close to the tip apex.

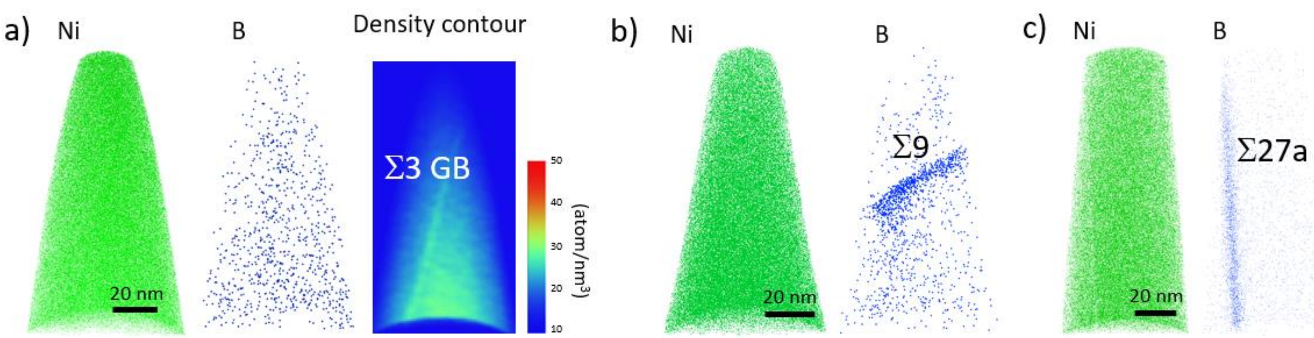

Figure 2. APT results for specimens containing Σ3, Σ9, and Σ27*a* grain boundaries. Sigma 3 shows no segregation to the grain boundary, consistent with the very low energy of formation for the FCC twin boundary type. A 2D density contour map confirms the grain boundary was acquired within the data (density variations along the GB). Both Σ9, and Σ27*a* boundaries show clear solute segregation to the respective grain boundaries[3]