# Grain Boundary Analysis in Ni-based Superalloys

## APT Methods for Quantitative Characterization of Segregation & Precipitation

Many advancements in high-performance alloys require compositional characterization of interfaces at the (sub-) nanometer scale using atom probe tomography (APT). Understanding the local enrichment and/or depletion of solutes across grain boundaries (GBs) and interfaces in proximity to GBs unlocks the design of advanced processing routes and/or new alloys.

Ni-based superalloys are high-temperature materials used for the most durable components in gas turbine engines. Their high-temperature strength is brought about by their hierarchical microstructure consisting of a γ-matrix, GB-γ' precipitates, carbides, and borides. Micro-alloying with B and C allows achievement of superior high-temperature strength and processability. However, minute variations in such micro-alloying additions have pronounced effects on the types and decorations of interfaces in Ni-based superalloys [1].

For example, the cast & wrought Ni-based superalloy René 41 provides excellent high-temperature strength but is limited in processability due to GB cracking. In collaboration with an Australian manufacturer, researchers at UNSW Sydney studied the detailed impact of B and C micro-alloying additions on GB segregation in the presence of various GB precipitates. Figure 1 provides a reconstruction of co-precipitated GB-γ', $M_6C$, and $M_{23}C_6$ containing the interfaces $M_6C$ / $M_{23}C_6$, GB-γ' / $M_6C$, and GB-γ' / $M_{23}C_6$ in proximity to GBs. Figure 2 highlights how the interfacial excess can be extracted via proximity histograms, concentration differences, and cumulative profiles. 'Interface plots' allow an intuitive comparison between individually decorated interfaces [2].

Advanced understanding of the complex interaction of solute enrichment and/or depletion is essential for process and alloy design, however, changes in solute solubility across phase boundaries often obscure this. We therefore developed APT methods for the quantitative characterization of the interfacial excess of solutes in such complex GB microstructures [1,2].

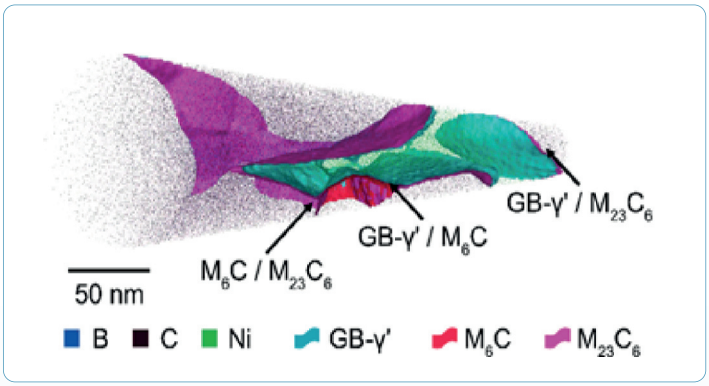


*Figure 1: Reconstruction of GB carbides $M_6C$ and $M_{23}C_6$ in René 41 with B additions exhibiting $M_6C$ / $M_{23}C_6$, B-γ' / $M_6C$, and GB-γ' / $M_{23}C_6$ interfaces.*

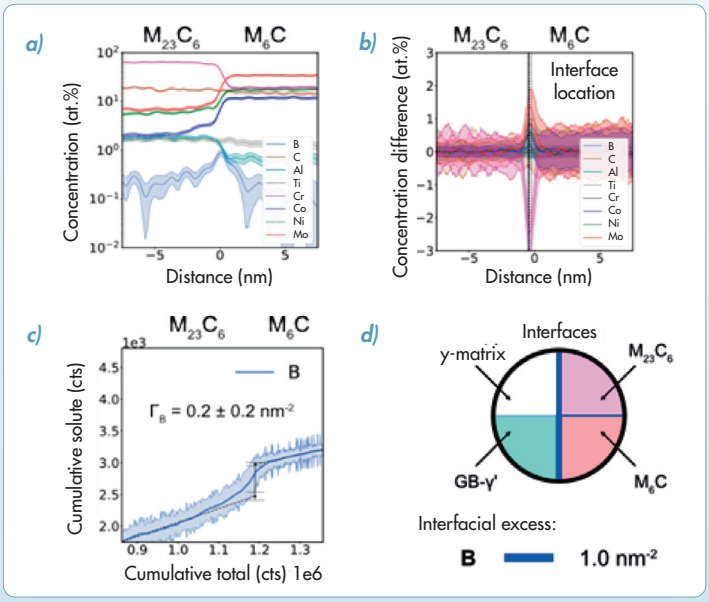


*Figure 2: a) Proximity histogram of the $M_6C$ / $M_{23}C_6$ interface, b) corresponding concentration difference profile, c) cumulative profile to extract the interfacial excess across this phase boundary, and d) 'interface plots' allow intuitive comparison of the interfacial excess between interfaces.*

[1] F. Theska, S.R. Street, M. Lison-Pick, S. Primig, Grain boundary microstructure-property relationships in the cast & wrought Ni-based superalloy René 41 with boron and carbon additions, Acta Materialia. 258 (2023) 119235. https://doi.org/10.1016/j.actamat.2023.119235.
[2] F. Theska, S. Primig, Interfacial excess of solutes across phase boundaries using atom probe microscopy, Ultramicroscopy. (2023) 113885. https://doi.org/10.1016/j.ultramic.2023.113885.

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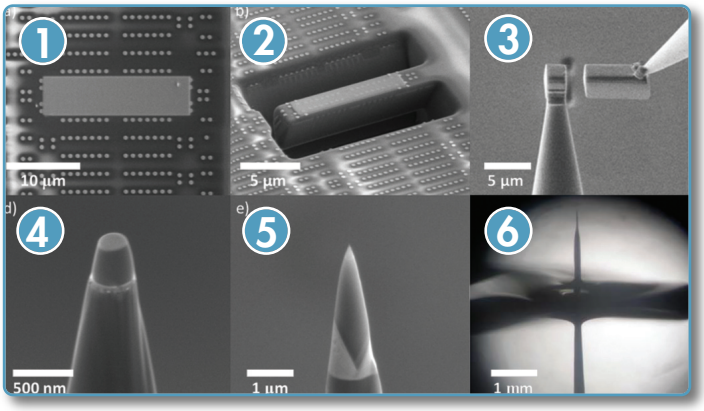
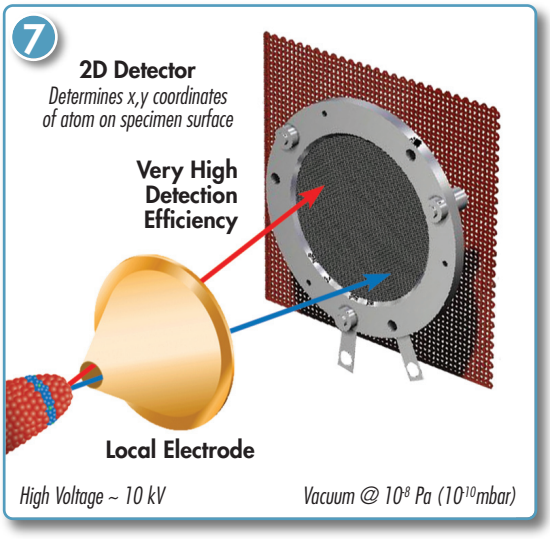
# Three Steps to 3D Nanoscale Analysis

## An Introduction to Atom Probe Tomography

### Step 1: Specimen Preparation

An atom probe specimen usually has a nanoscale region of interest (ROI) requiring both 3D compositional imaging and analysis. The sample is formed into a needle shape containing the ROI. Common APT specimen preparation methods using electropolishing or a Focused Ion Beam system (FIB) are very similar to TEM methods except instead of forming a thin sheet, a needle shaped sample is desired. At the right, standard FIB liftout and mounting of a specimen (figures 1 through 3) and then sharpening the sample with the ROI left at the very apex ( 4 and 5). In 6, a wire geometry sample is being electropolished.




### Step 2: Data Collection

An atom probe produces images by field evaporating atoms from a needle-shaped specimen and projecting the resultant ions onto a detector 7.

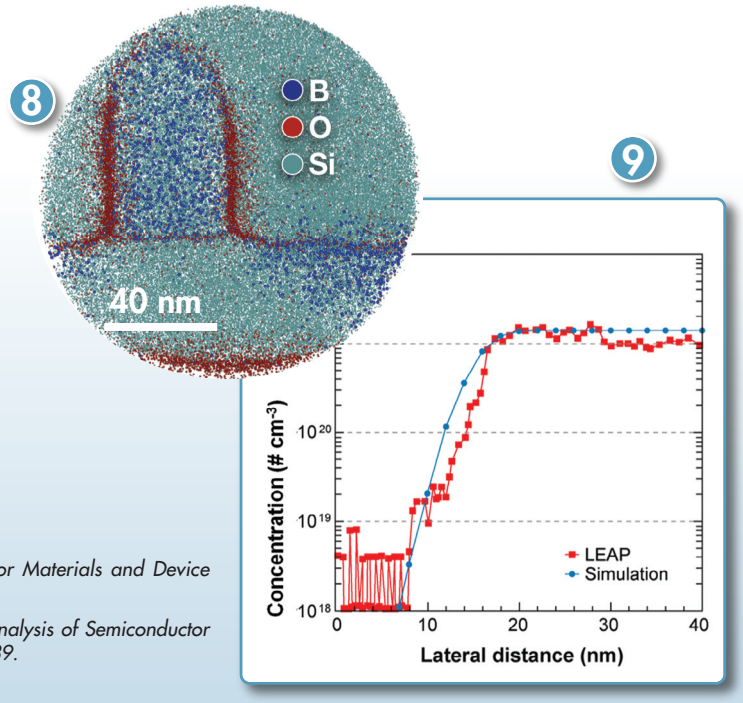
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately $10^6$.

An atom probe identifies atoms by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

The specimen is held at approximately 50K to reduce surface diffusion during the experiment. The high electric field results in 100% ionization and the high speed detector is capable of measuring up to 80% of the collected ions, independent of ion mass.

### Step 3: Data Visualization and Analysis

Examples of data output are illustrated by a slice of a 3D atom map of a transistor† 8, and a dopant composition profile‡ 9. The image shows the positions of individual atoms (oxygen is red and boron is blue) in the transistor with subnanometer resolution. From the reconstructed data set many types of useful analyses are possible. These include 3D visualization, 2D atom mapping 8, 1D depth profiling and line scanning 9, as well as mass spectra and compositional analysis from user-selected volumes.




† *Lauhon, L. J. et al, MRS Bulletin "Atom Probe Tomography of Semiconductor Materials and Device Structures" 34(10) (2009) 738.*

‡ *Moore, J. S.; Jones, K. S.; Kennel, H.; Corcoran, S., Ultramicroscopy "3-D Analysis of Semiconductor Dopant Distributions in a Patterned Structure using LEAP" (2008), 108, 536–539.*

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