CAMECA SCIENCE & METROLOGY SOLUTIONS / AMETEK

APPLICATION NOTE #EIK-04

Optimization of Additively Manufactured Materials
APT Analysis of Nanoscale Clustering

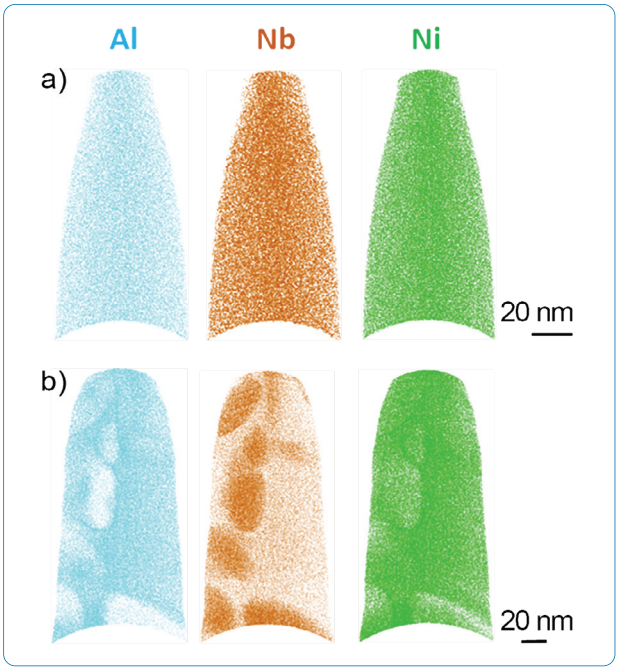
Additively manufactured (AM) materials, also known as 3D printed materials, are a rapidly growing class of materials due to their numerous processing advantages. Initially established for polymeric materials, several techniques have since been developed to allow metal and alloy manufacturing through additive methods. One breakthrough for AM materials is the idea of rapid prototyping of parts or manufacturing complex designs. This enables faster, potentially less expensive manufacturing methods to replace traditional foundry casting followed by complex machining.

In one method of metal AM, a powder bed of the desired material is heated in specific areas through use of a precise laser or electron beam to sinter the powder in areas that will become the final solid part. The initial powder bed composition provides options for the users to customize the material of the finished part.

Atom probe tomography (APT) can provide atomic-scale insight into the local chemical variation and microstructure of AM materials, which impact those materials' ability to be used in load-bearing applications. CAMECA APT microscopes provide useful nanoscale information, such as clustering behavior of different chemical species and grain boundary segregation, that can be used to optimize processing conditions in order to achieve the optimal properties.

Figure 1a shows a uniform elemental distribution in the laser beam-assisted curing method of an Inconel 718 alloy. Each dot represents a detected ion of the element shown.Only a portion of ions are displayed for clarity. Figure 1b shows the same alloy deposited by electron-beam assisted curing. Large niobium-rich precipitates were formed during the electron beam-assisted process, which is attributed to the bed temperature being above the temperature of the gamma prime phase formation. Aluminum is shown to form smaller precipitates on the flat surface of the large disc-shaped Nb precipitates as indicated by the isoconcentration surfaces in Figure 2.

By correlating the microstructure and material performance to processing conditions, additive manufacturing methods can be quickly optimized. Atom probe tomography provides 3D data that can advance the AM field by providing understanding of the chemical composition and microstructure on the atomic level.



Alloy provided by collaborators at The University of North Texas and Monash University.

Figure 1: Elemental distribution in (a) laser beam-assisted AM Inconel; (b) electron beam-assisted AM Inconel [1]

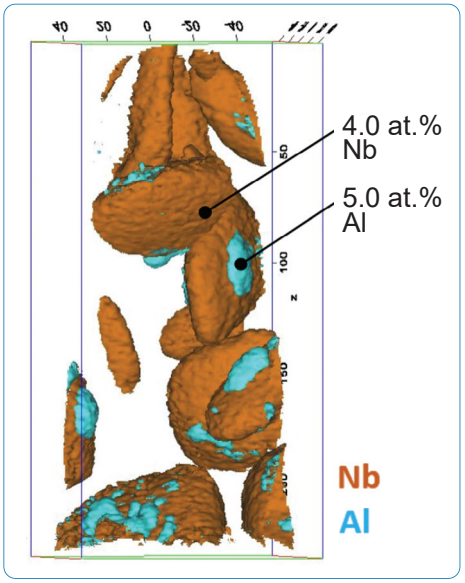


Figure 2: 4 at.% Nb and 5 at.% Al isoconcentration surfaces showing nanoscale clustering behavior in electron beam assisted AM Inconel [1]

[1] Larson, D.J. et al., Microstructural Investigations in Metals Using Atom Probe Tomography with a Novel Specimen-Electrode Geometry, Journal of Metals (Sep. 2018, Vol. 70, Issue 9, pp 1776–1784)

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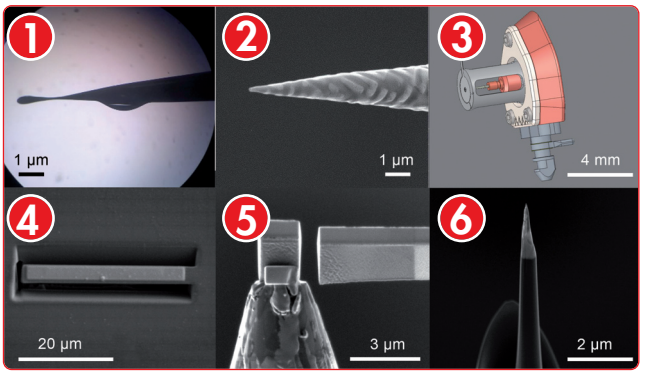
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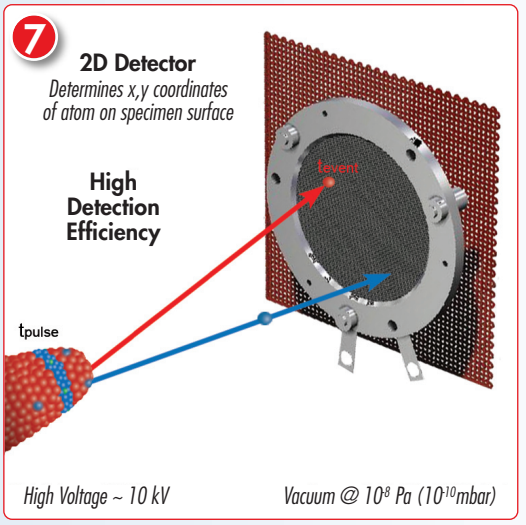
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Three Steps to 3D Nanoscale Analysis with the EIKOS-UV™ Atom Probe

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. Very similar to standard TEM preparation, the needle shape can be created using commercially available electropolishing or Focused Ion Beam systems. At the right, standard electropolishing (1, 2) and sample mounting in an integrated electrode puck (3) and standard FIB liftout (4) mounting, and sharpening (5) of a specimen to a carrier needle (6).





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).

With the EIKOS-UV™ Atom Probe microscope, a high magnification is created from the ~ 80nm tip being projected onto an 40mm detector, resulting in a magnification of approximately 5E10^5.

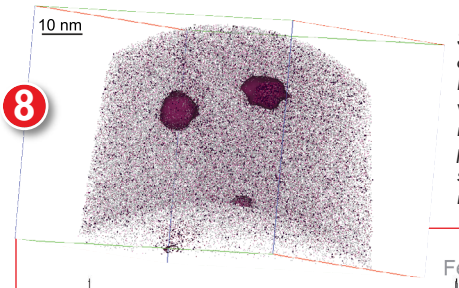
Atoms are identified by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

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 40% of the collected ions, independent of ion mass.

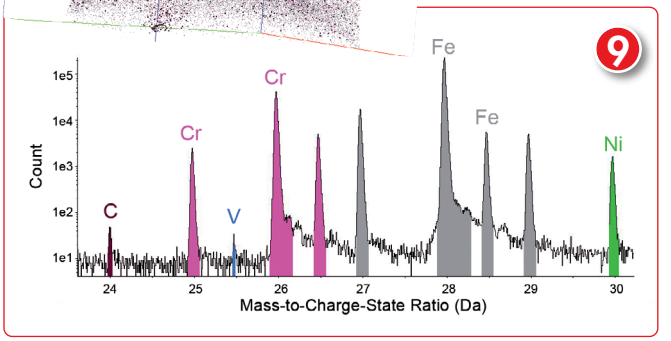
Step 3: Data Visualization & Analysis

Examples of data output are illustrated by a 3D atom map of a stainless steel (8), and a mass spectrum of the entire data set (9).

The image shows the positions of the individual atoms in the alloy with subnanometer resolution). From the reconstructed data set many types of useful analysis are possible. These include 3D visualization, 2D atom mapping, 1D depth profiling and line scanning as well as mass spectra and compositional analysis from user-selected or chemically distinct volumes.



Stainless steel analysis obtained with CAMECA EIKOS-UV™ , clearly revealing the MoN and C rich precipitates that add strength and corrosion resistance to this alloy.



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