CAMECA SCIENCE & METROLOGY SOLUTIONS / AMETEK

APPLICATION NOTE #EIK-01

R&D of Innovative Nuclear Structural Alloys with APT
Analysis of Proton Irradiated 304 Stainless Steel

Atom probe tomography (APT) provides three-dimensional atom locations with subnanometer spatial resolution. The technique is ideally suited for determining the distribution and chemical composition of solute clusters in a wide variety of materials, including clusters induced in steels by radiation.

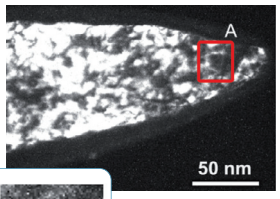
Most structural materials in nuclear reactors are subjected to radiation-induced degradation phenomena, which have become a critical issue in the context of life-time extension campaigns for the existing fleet of electrical power plants. Commonly observed consequences of irradiation are hardening, embrittlement, and dimensional instability including creep and swelling. The microstructural changes that may be responsible for these mechanical property changes include dislocation loops, solute clustering, precipitation, irradiation induced segregation to grain boundaries and other very small features referred to 'black dots' in high resolution electron microscopy. Establishing direct and quantitative relationships between microstructure and properties is not trivial with most techniques because of the complexity of the microstructure involved and the challenges involved in characterizing such microstructures at the nano-scale.

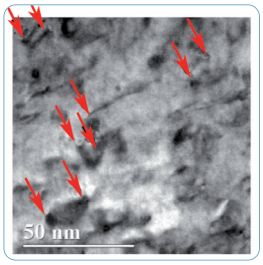
Reliably and reproducibly quantifying these localized chemical changes is essential to the data analysis. The EIKOS™ atom probe microscope is the perfect tool for these studies. Its ease of use, very high spatial resolution and high, single ion sensitivity provide direct documentation of the relationship between irradiation conditions and the micro and nano-structural evolution.

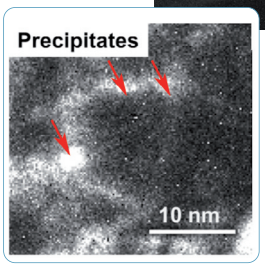
In this study, a 304 stainless steel was proton irradiated at 360 °C to approximately 10 dpa (displacements per atom). The samples were prepared with standard APT techniques (similar to TEM sample preparation) and analyzed with atom probe tomography. The resultant 3D atom map can be automa-tically and objectively analyzed to provide the number density and chemistry of Cu clusters, Ni–Si rich clusters and Si/Ni/P segregation to dislocations from a single volume and analysis. This provides a deep understanding of the consequences of radiation damage to these structural steels.

Chen, Y, Chou, PH, and Marquis, EA (2014), J. Nucl. Mat., 451(1-3).

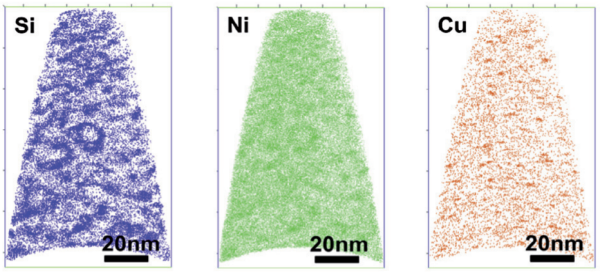
High magnification image revealing a dislocation loop with bright dots



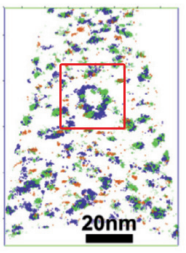




BF-STEM image showing dislocation loops with possible clusters indicated by arrows.

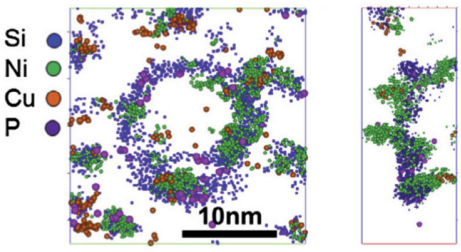


Atom maps with 10 nm thicknesses oobtained from the as-irradiated alloy.



Left: Atom map filtered by local concentration showing the distributions of Si, Ni, Cu and P.

Bottom: front (planer) and side close-up views of the dislocation indicated by the red square.

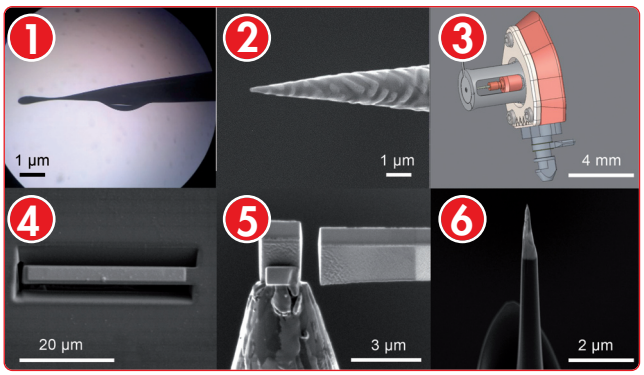


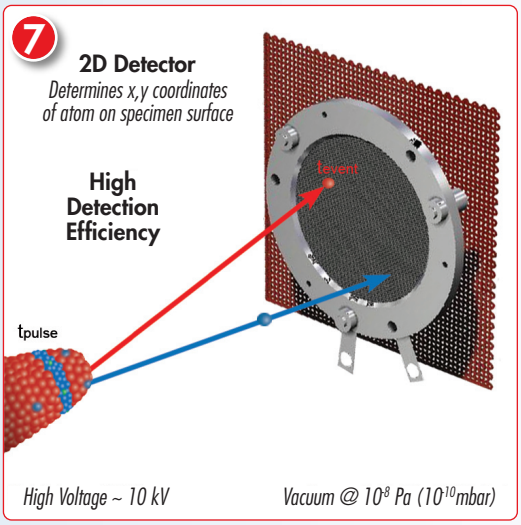
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Three Steps to 3D Nanoscale Analysis with the EIKOS-UV™ Atom Probe

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.

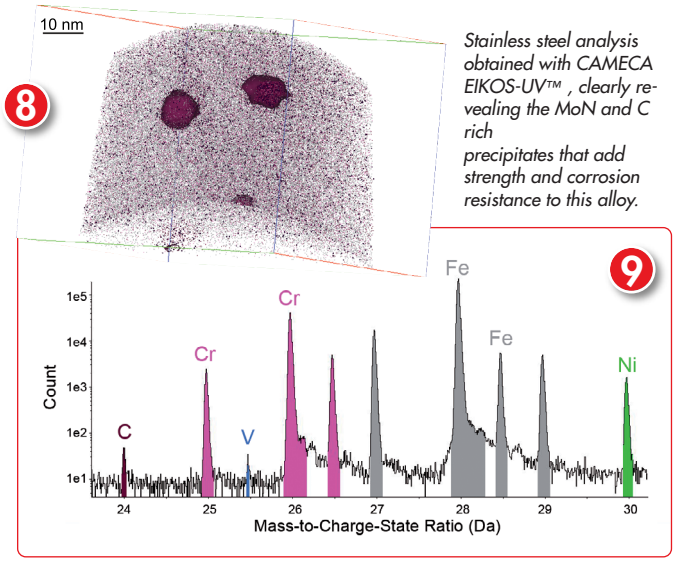
Atoms are identified by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

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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