Atom Probe Tomography

Multi-Axis Sample Positioning in Ultra-High Vacuum with ECSx5050/NUM Stages

Markus Maier
attocube systems AG, Eglfinger Weg 2, 85540 Haar, Germany

Introduction

Atom Probe Tomography (APT) is a 3D nanoscale technique for the analysis of different materials based on controlled field evaporation of individual atoms. In practice, the sample under examination is required to have a needle-like shape to generate a highly curved electrical potential at the tip of the specimen, which in turn results in the evaporation of atoms from the surface of the material. While historically most atom probes used voltage pulsed fields, since the early 2000s a resurgence of activity in laser pulse applications can be observed in the community [1]. The electrical field pulse method introduces a high stress-level on the specimen and is only suitable for metals with high electrical conductivity, whereas the laser pulse method made APT applicable to a wider range of materials such as minerals and nanomaterials [2].

Due to the more general applicability, reduced cost and increased efficiency of the technique, and other theoretical and practical advances in the field, the user base of APT has expanded significantly since the turn of the century. The latest APT systems can generate three-dimensional information at near atomic resolution. As such, they are not only used to analyze a sample's chemical composition, but also its morphology and the spatial distributions of its elements [2]. An example of the astonishing ability to observe micro- and nanoscale features by using APT can be seen in Figure 1.

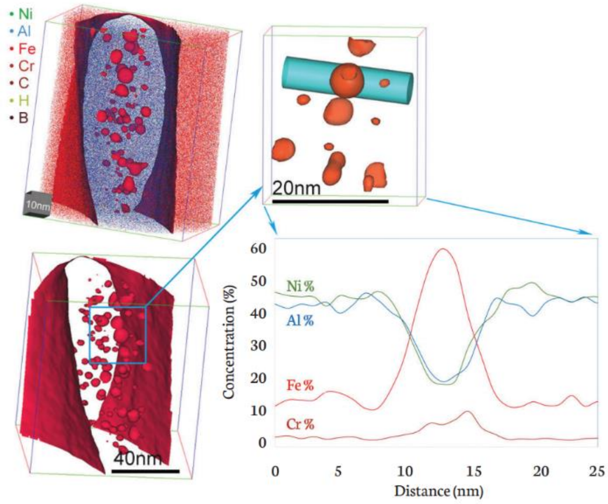

Figure 1: Exemplary APT analysis data, taken from [2].

Today, CAMECA is the exclusive manufacturer of atom probe tomography and uses attocube's ECSx5050/NUM/UHV for the precise positioning of the specimen with respect to the counter electrode. This application note introduces the reader to the technique of APT and explains how and why attocube's positioning solution is perfectly suited for the application in the challenging environment of an APT system.

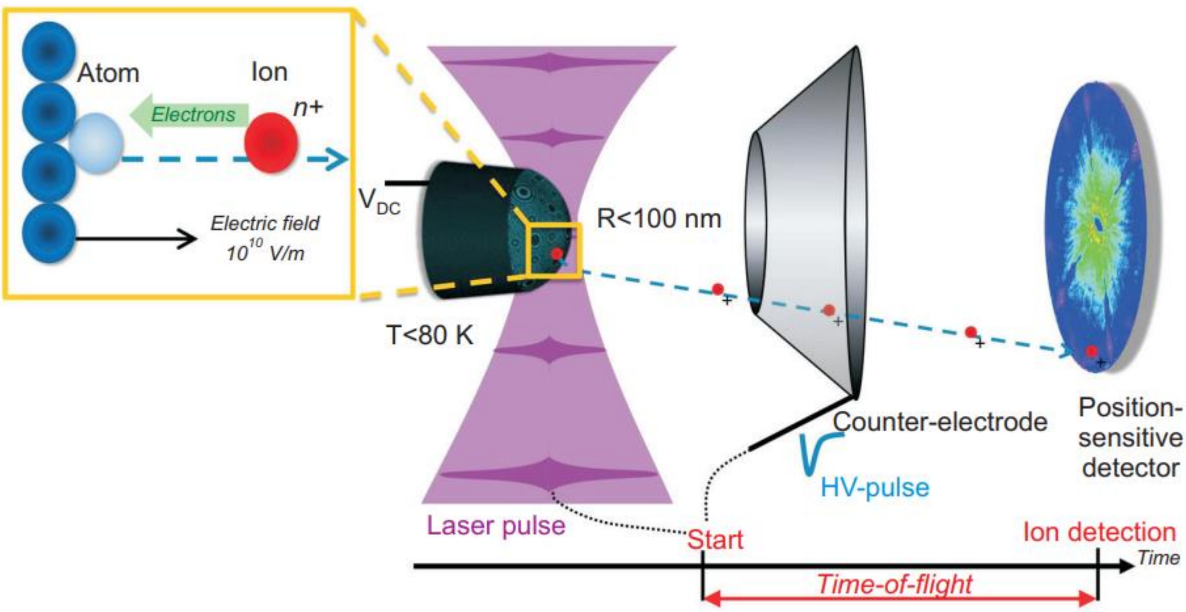

Figure 2: Schematic view of an atom probe microscope. The specimen (black) with a tip-radius of less than 100 nm is subjected to a high voltage and illuminated by either laser or high voltage pulses, which trigger the field evaporation at the sharp specimen tip. The positive ions are accelerated by the electric field, their impact position and time of flight is recorded with a position-sensitive detector [3].

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Application

The application of an APT system can be roughly divided into three steps:

1. Specimen preparation,
2. Data generation and collection,
3. Data analysis.

Each of these steps is subject to continuous research and improvements. For the scope of this Application Note we will focus on the second step – data generation and collection – exclusively.

In this step, the atom probe technique is basically a combination of field evaporation of the atoms from the tip of the needle-shaped specimen and the identification of the evaporated ions by time-of-flight mass spectrometry [4]. Figure 2 shows a schematic setup of an APT system. The specimen of the material under investigation is subjected to a high voltage electric field and a high voltage pulse or a laser pulse then triggers the field evaporation.

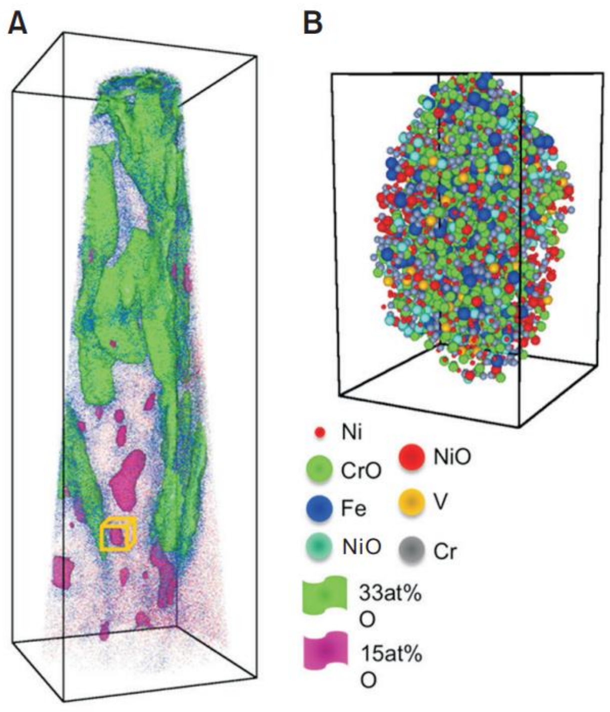

Figure 3: Dataset of an oxidised Nickel-based alloy. B shows a closeup, indicated by the orange region inside A. Graphic taken from [3].

To prevent surface migration and to improve control over the field evaporation process, the specimen is cooled down to cryogenic temperatures below 80 K. Furthermore, the analysis takes place under ultra-high vacuum (UHV) conditions [3].

During the time of flight, spatial differences of the ions (*i.e.*, position of the atoms) are magnified, resulting in an enlarged mapping of the tip surface to the detector [4]. Determining the time of flight (which also yields a mass-to-charge ratio) and impact position of each ion, together with the voltage applied at the moment of evaporation, allows for a tomographic, atomically resolved image of the evaporated volume. This data is often represented as a point-cloud where every point is an elementally identified atom [3]. Figure 3 shows an example of APT data where every single atom is visualized as a member of such a point cloud.

In order to enable resolution down to 0.1 nm, the specimen tip with an end radius of below 100 nm needs to be accurately positioned along 3 axes (X, Y, and Z) within an UHV environment.

attocube's Solution

The demand for a high-precision positioning system inside an ultra-high vacuum environment is a prime application for attocube's positioners. The compact and modular nano positioners can be combined into multi-axis motion systems, offering the highest precision and stability even under challenging environmental conditions. This allows reliable sample or probe positioning in advanced material analysis systems such as the APT.

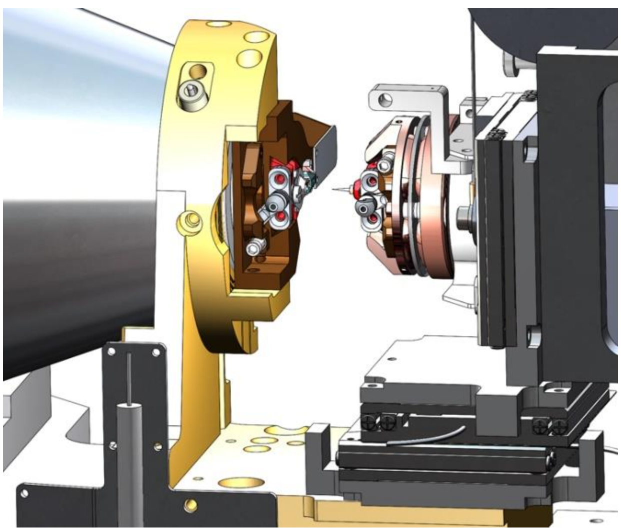

Figure 4: Setup of the APT system with the sharp specimen on the right. The positioning system allowing for multi axis positioning can be seen behind and below the specimen.

In the case of the APT application, attocube's ECSx5050 with integrated encoder for closed-loop control is used to accurately position the specimen inside the system. The ECSx5050 is specifically designed for ultra-high vacuum conditions and provides high-resolution positioning over a travel range of 30 mm. For xyz-positioning, two ECSx5050 positioners can be mounted on top of each other and combined with a third one attached to an L-bracket. Its high accuracy is achieved with crossed roller bearings. Figure 4 shows a representation of the setup. Not only does the large range of motion and precise control allow the user to align single specimens, it also allows the use of arrays of specimens. A CAMECA developed microtip array can carry as many as 25 specimens onto the stage at one time, and the stage with its closed loop control, combined with machine vision allows unattended analysis of each specimen freeing up the user to concentrate on data analysis tasks.

Conclusion

Atom probe tomography developed into a versatile analytical technique, and today can be applied to a variety of materials in different research fields [4]. In a single material analysis technique, CAMECA's solution enables both 3D imaging and chemical composition measurements at the atomic scale and is relevant to researchers as well as industrial applications.

Since a system is only as good as its components, the APT crucially relies on a precise, accurate and reliable positioning of its specimen. With nanometer resolution, attocube's ECSx5050/NUM/UHV is the perfect solution for the demanding application of three-dimensional positioning under UHV conditions inside the APT system with extremely high throughput through automated serial analysis.

References

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[3] Gault, B., Appl. Microsc. 46(3), 117-126 (2016).

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