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Atom Probe Tomography

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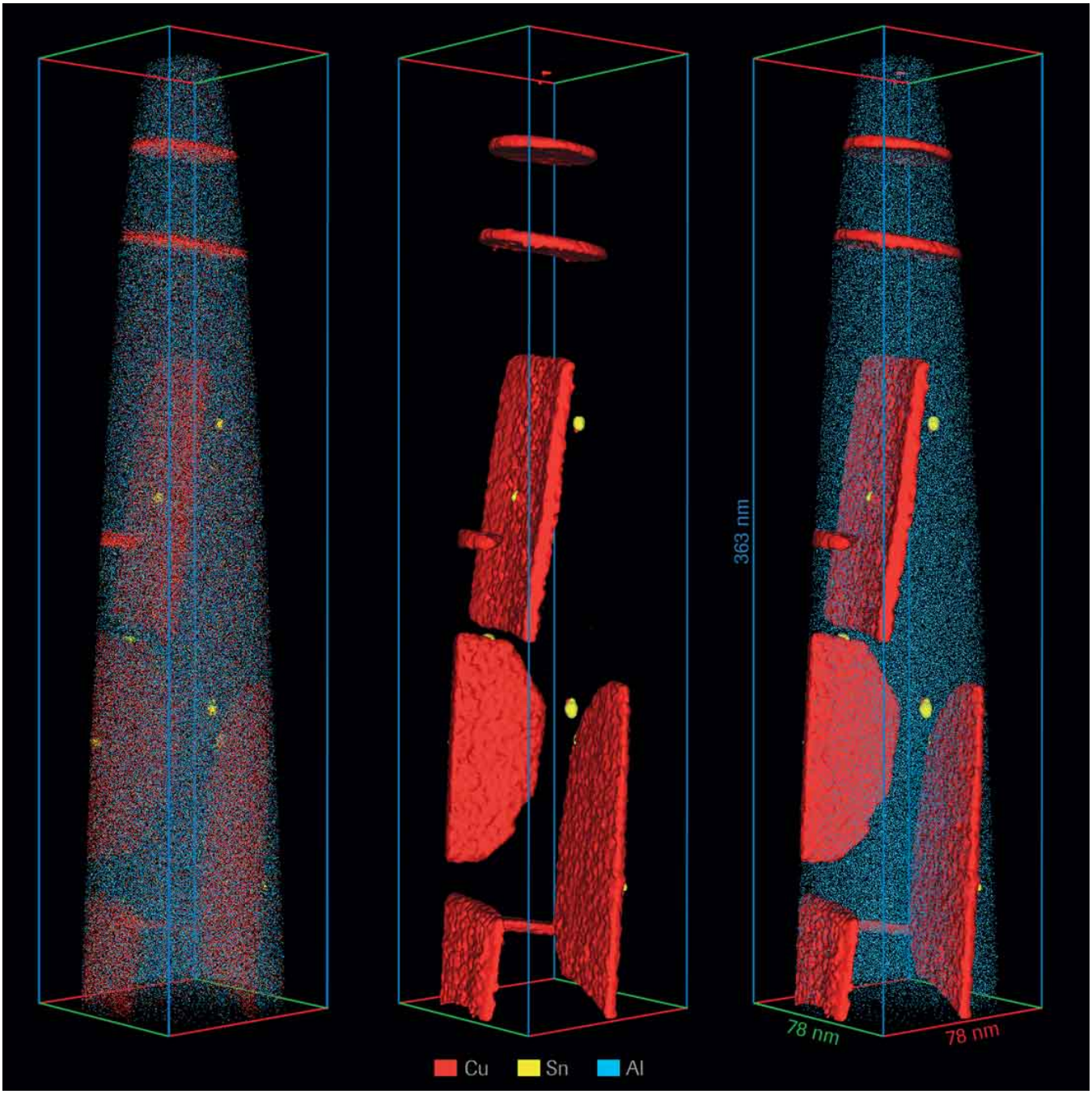
Welcome

MyScope was developed by Microscopy Australia to provide an online learning environment for those who want to learn about microscopy. The platform provides insights into the fundamental science behind different microscopes, explores what can and cannot be measured by different systems and provides a realistic operating experience on high end microscopes.
We sincerely hope you find our website: www.myscope.training an enjoyable environment. In there you can explore the microscopy space and leave ready to undertake your own exciting experiments.

Module Contents

What is APT? - Background Information - Overview

Atom Probe Tomography (APT) is a powerful microscopy technique that generates a three-dimensional (3D) atom-by-atom reconstruction of a small volume within a material. The resulting 3D atom maps show the position and identity of individual atoms in a region containing potentially hundreds of millions of atoms. An example of an atom probe dataset can be seen in the image below, which shows an atom probe reconstruction of an aluminium-copper-tin alloy, with the aluminium atoms shown in blue, the copper atoms in red and the tin atoms in yellow.

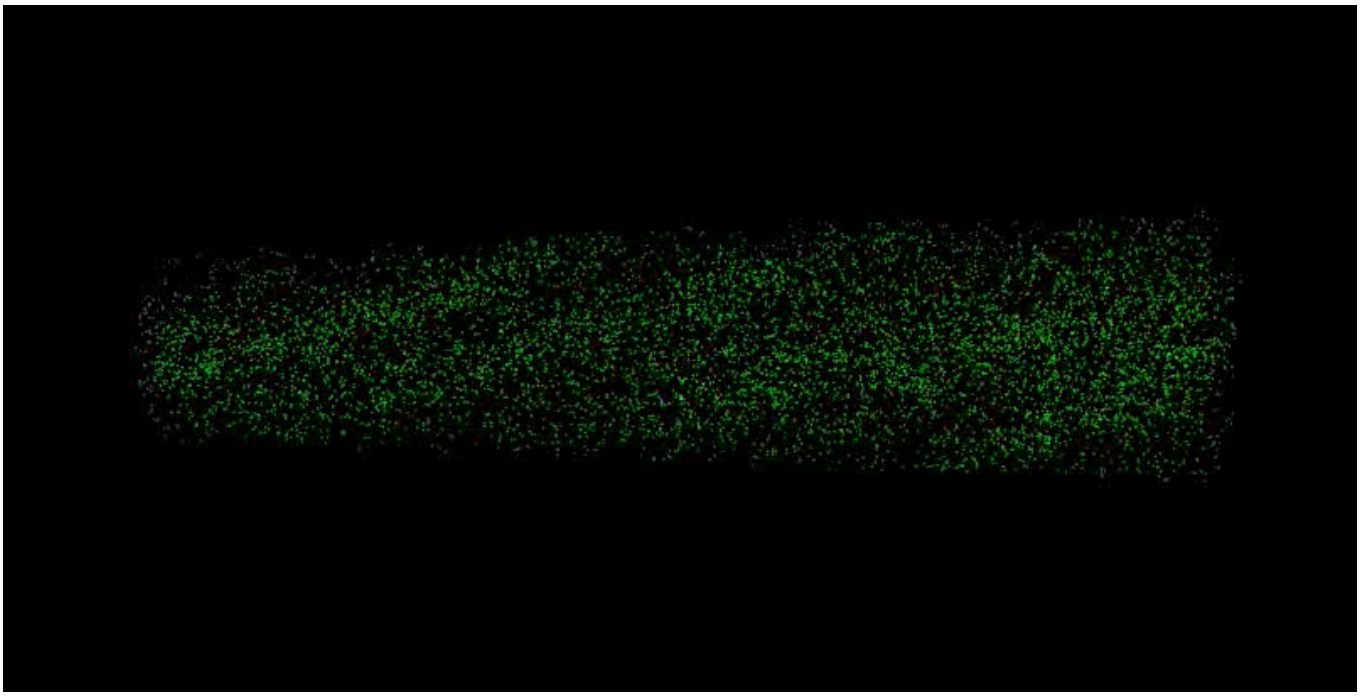


Aluminium-Copper-Tin alloy.

In an Atom Probe, atoms are evaporated, one by one, from the surface of a very small needle-shaped sample (with a tip of typically < 100 nm diameter) and then projected onto a position-sensitive detector. The detector measures the (X, Y) position of each atom's impact, the order of arrival, and the time of the atom's flight from the tip to the detector. This information can be used to reconstruct a 3D image of the material at the atomic scale.

Animation showing APT.

The way the atom probe 'looks' into a material can be compared to a person observing an individual atom, recording its position, removing it and weighing it with a scale to identify it. By doing that for millions of atoms, a very precise 3D atomic map of the material can be developed. The atom probe uses an electrostatic field combined with voltage or laser pulses to remove the atoms, a position-sensitive detector to observe their positions, and a time of flight spectrometer as the scales.



Inside human tooth enamel. (Calcium, Phosphorous and Magnesium atoms).

In contrast with optical and electron microscopy, magnification in an Atom Probe does not come from lenses (optical or magnetic) but instead the sample itself acts as the lens, with the particular shape of the Atom Probe needle inducing a highly curved electric field. The Atom Probe is a tool to observe atoms within a material, with typical resolution below a nanometre. Every element (and their respective isotopes) in the periodic table can be identified with high precision using time of flight mass-spectrometry. Computer models are used to build a 3D view of the sample, allowing scientists to 'dive into' the material and observe each individual atom.

Applications of APT

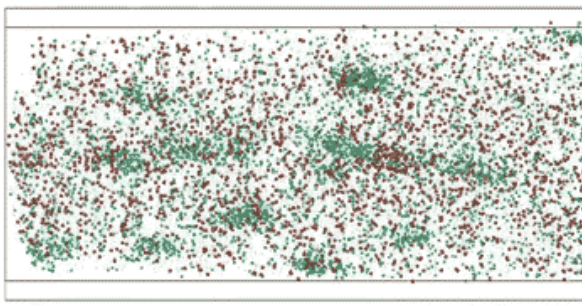
APT's unique ability to image 3D variations in chemistry at the sub-nanometre scale has proven to be of great importance for the development of new materials in industries such as automotive, aerospace, nuclear storage, renewable energy and microelectronic industries, just to name a few. APT enables the investigation of nanoscale features and processes, such as clustering of solute atoms, phase transformations or precipitation. It is these features that often govern the functional and mechanical properties of materials.
APT is most commonly applied in the area of materials science. In particular it is used to study metals, semiconductors, ceramics and composite materials. However, recent developments in instrumentation have expanded the application areas to geological and biological science.

Materials science

In the past, the use of APT was limited to materials that are good conductors of electricity (as the atoms of conducting materials more easily ionise and evaporate under an electric field). Metallic alloys such as steels, aluminium alloys, magnesium alloys or nickel superalloys were among the first materials to benefit from the unique capabilities of APT. The development of laser-assisted APT considerably expanded the range of materials studied by APT (the laser provides additional energy to promote field evaporation in non-conductors). Less conductive materials can now be analysed, such as semiconductors (group IV, silicon and germanium nanowires, optoelectronic and solar cells), ceramics (ferroelectrics, piezoelectrics), oxides (metal-oxide interfaces, MOSFET and FinFET structures), commercial devices (transistors) and even synthetic polymers.

Geological science

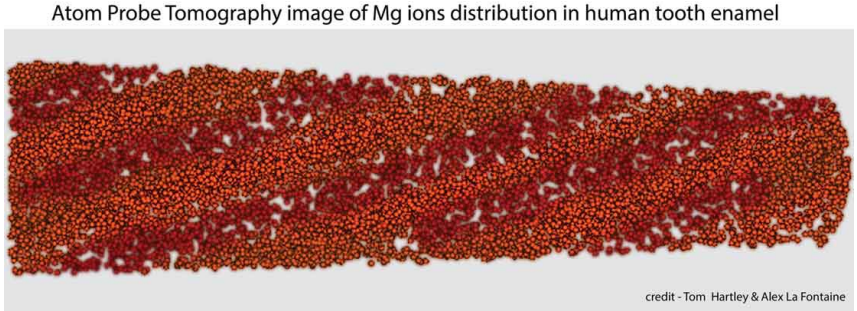
The microstructure of most minerals contain a record of their history. APT has recently been utilised to look into the nanostructure and chemical variations of several important geological systems such as zircons, extrasolar nanodiamonds, olivines and even meteorites. This contributes to our understanding of the earth, the solar system and even extrasolar events. The ability of APT to measure isotopic ratios at the nanometer length-scale is one of the main factors that drives the growth of this field of application and, without a doubt, applications in geology will expand rapidly in the future.



3D-rotating volume of Y and Pb in Zircon.

Biological science

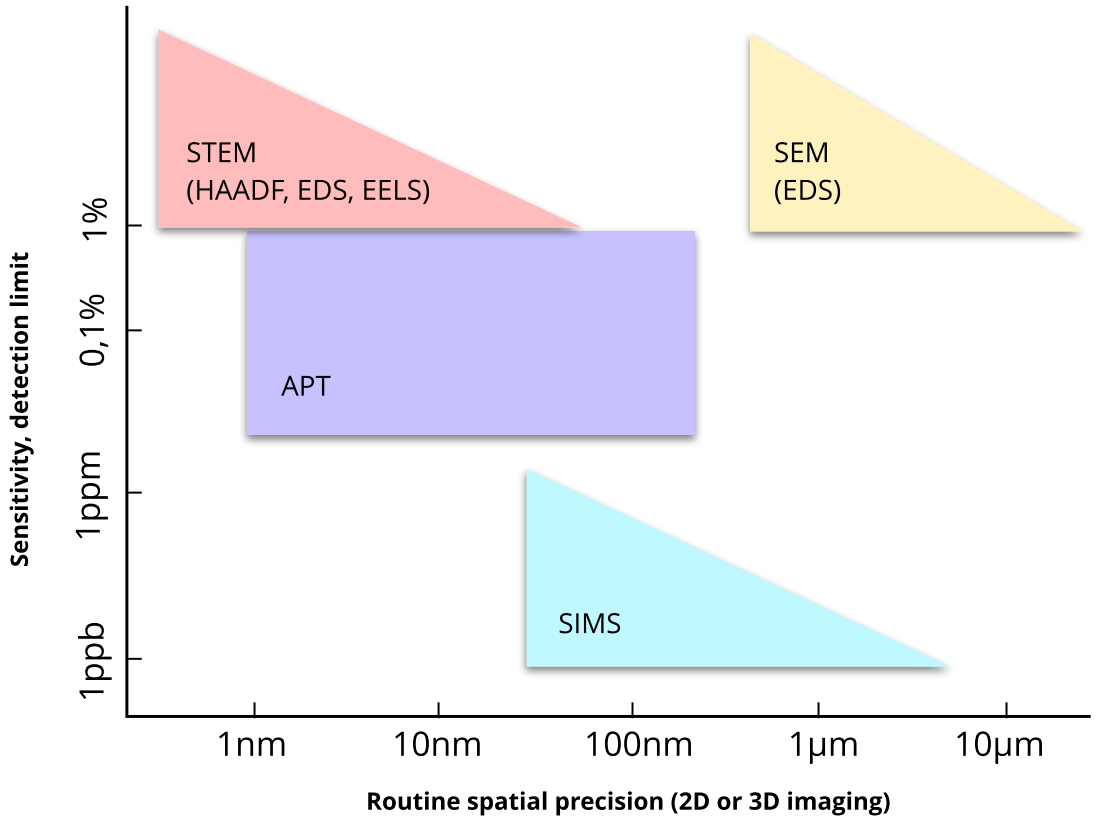
In biological science, most samples are soft, thus the requirement of a dense, solid needle shaped specimen means that they are extremely challenging to investigate by atom probe. Until recently, only a few biological systems have been successfully analysed by APT, including teeth, bone-implant interfaces, ferritin and even part of a fixed cell. However, the recent development of fully integrated cryogenic systems, from sample preparation to atom probe investigation, is expected to spur growth in this area of application.



Atom probe tomography dataset showing the distribution of Mg ions within human tooth enamel.

What can APT do differently?

Scanning electron microscopes can achieve a spatial resolution of around 5 nm when equipped with a field emission electron gun. However, their analytical capability (X-ray emission) is often limited by the scattering of electrons within the large interaction volume (around 1 micrometre).
Transmission electron microscopes (TEM) can now achieve atomic resolution but their chemical sensitivity is usually limited to one atomic percent. Moreover, data and images are obtained in 2D and most of the 3D information may be lost. While TEM tomography provides some of the 3D information, apart from for very specific cases, its resolution is still far from the atomic scale. The detection limit of Secondary Ion Mass Spectroscopy (SIMS) can reach parts per billions but its spatial resolution is limited to around 50 nm.



Spatial resolution and chemical sensitivity of SEM, TEM, SIMS and APT technique.

APT provides information in three dimensions, with the capacity to achieve sub-nanometre spatial resolution (up to 0.1 nm in depth and below 1 nm in lateral resolution). It also has a chemical sensitivity of up to atomic parts per million.

What can't APT do?

Measuring the sample again

APT is a destructive technique. Measurements cannot be repeated on the same sample.

Use various specimen geometries

An atom probe sample must be needle shaped with a maximum tip diameter of around 200 nm in order to enable field evaporation and must not contain pores. However, there are many sample preparation techniques available that enable either transformation of the bulk sample into a needle, directly manufacture samples in the form of a needle, or to transfer of the original material onto a pre-sharpened needle. For nanoscale materials or porous materials it may be necessary to combine the sample with another solid material before shaping into a needle.

Analyse wet materials

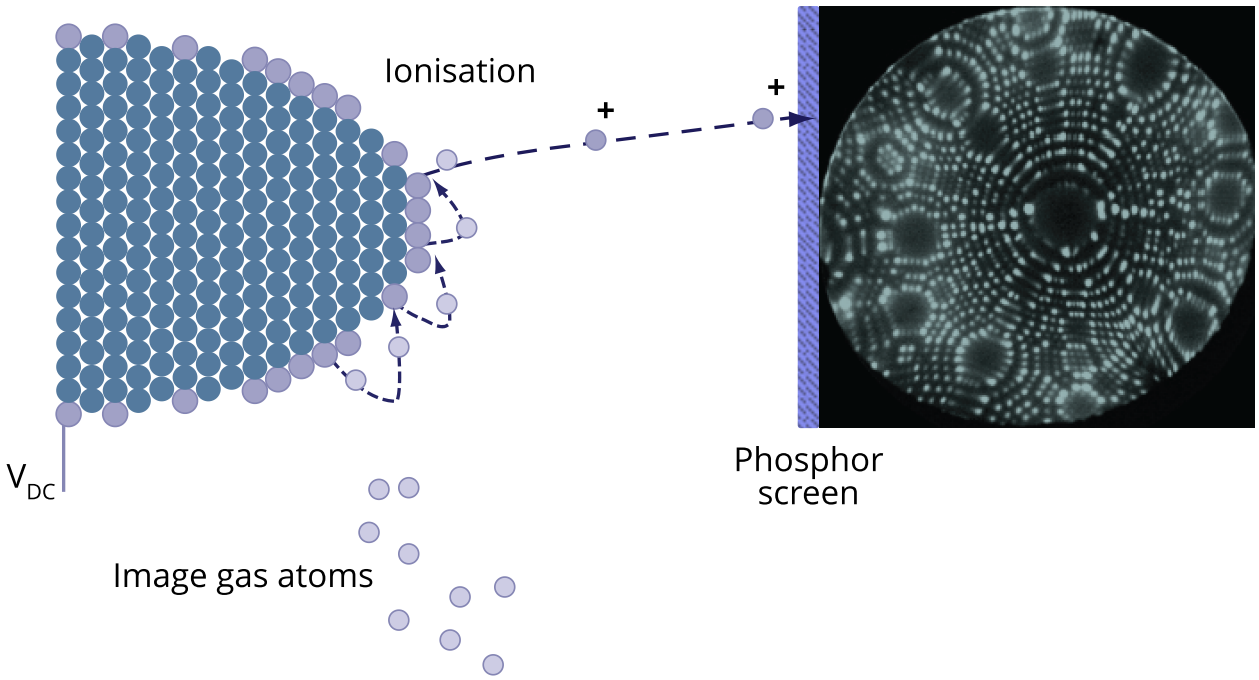
An atom probe is kept under ultra-high vacuum. Water within an atom probe sample will damage the system and the sample itself. A wet sample should be dehydrated before APT sample preparation. However, with recent advances in cryogenic sample preparation, transportation and analysis in the atom probe, the analysis of frozen samples in the atom probe might soon be achievable.

Analyse a large region of interest

The field of view is limited by the sample geometry. Typically, the tip diameter does not exceed 200 nm and it is usually less than 100 nm. In a typical atom probe experiment, the volume analysed would be around 60 x 60 x 300 nm^3. In some rarer cases with 'robust' samples, volumes can exceed 60 x 60 x 3000 nm^3.

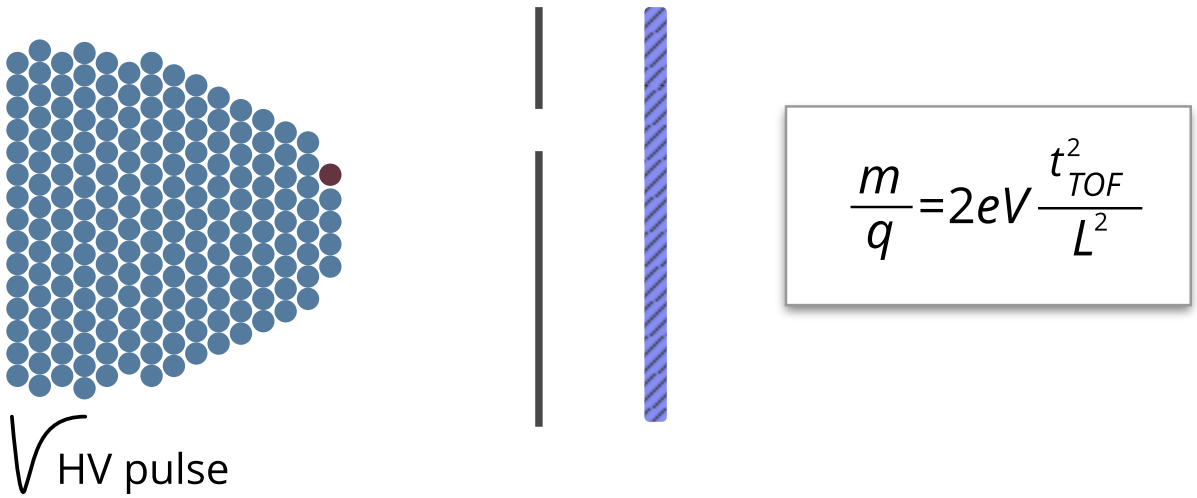
A brief history of APT

APT originates from the work of Erwin Müller, who led the team that developed the field ion microscope (FIM) in the 1930s. To undertake field ion microscopy, a sharp needle-like specimen is placed in an ultra-high vacuum chamber where the tip is cooled to cryogenic temperatures. An imaging gas such as Helium or Neon is pumped into the chamber and a high voltage applied to the sample (up to 10kV). The imaging gas atoms adsorbed on the surface are positively ionised by the strong electric field and repelled from the surface toward a phosphorus screen. The image formed represents individual atoms on the tip surface. This was the first time individual atoms had been observed.



Field Ion Microscopy (FIM) technique pioneered by Erwin Müller.

In 1968, the first time-of-flight atom probe was developed by combining a time-of-flight mass spectrometer and FIM.



Atom Probe Field Ion Microscopy technique.

A position-sensitive detector was first used at Oxford University in the UK in 1988 and gave rise to the position-sensitive atom probe (PoSAP). In 1993, the University of Rouen in France developed the tomographic atom probe (TAP), introducing a multichannel timing system and a multi anode array. Both instruments were commercialised by Oxford Nanoscience and CAMECA respectively.

In 2003, the local electrode atom probe (LEAP®) was introduced by Imago Scientific Instruments, leading to a large increase in the field of view and the size of datasets that could be acquired by atom probe. In 2005, the pulsed laser atom probe (PLAP) was also introduced by Imago Scientific Instruments, allowing the analysis of non-conducting materials. AMETEK acquired CAMECA in 2007 and Imago Scienti c Instruments in 2010. Today, the most common design for APT is the local electrode atom probe (LEAP®), manufactured by CAMECA®.

How does APT work? - Intro to the technique - The principle of APT

APT is a microscopy technique that enables 3-dimensional visualisation and chemical identification of materials at the atomic scale. Ions are successively field-ionised from the tip of a needle shaped sample, collected by a position sensitive detector and their mass-to-charge ratios are recorded by time-of-flight mass-spectrometry. In a typical APT analysis chamber, the sample is cooled to cryogenic temperatures (~ 50 K) and maintained at ultra-high vacuum (~ 10^{-11} Torr). For APT the sample is required to be a sharp hemispherical-shaped tip with a typical diameter of < 100 nm. When a DC voltage (typically between 2 and 10 kV) is applied to the specimen, a high electric field is achieved at the surface of the tip according to this equation:

$$F = \frac{V}{k_f R}$$

Where F is the electric field at the apex of the tip, V is the applied voltage, k_f is the field factor (a correction parameter accounting for experimental aspects such as sample shank angle), and R is the radius of curvature of the tip.

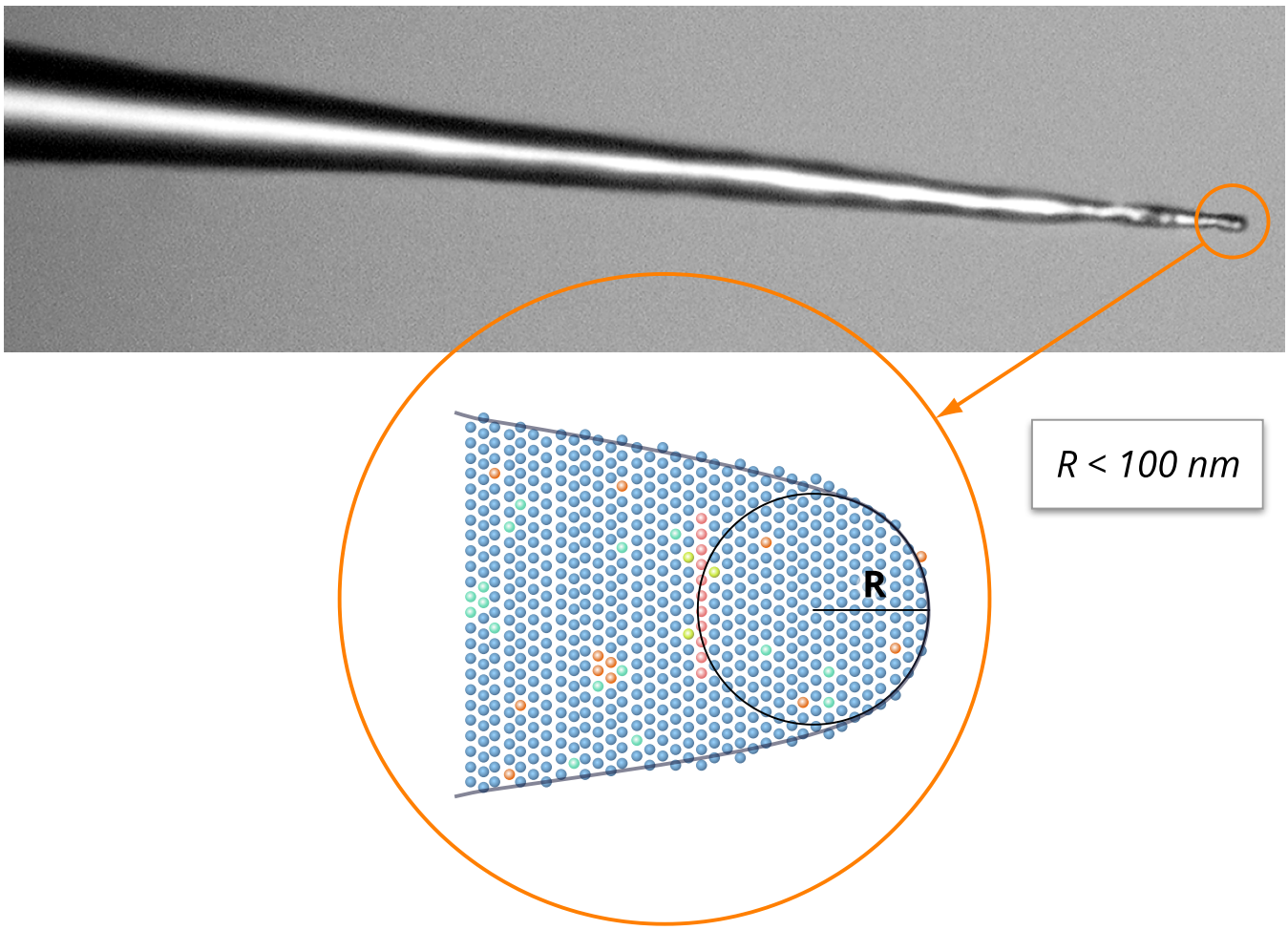
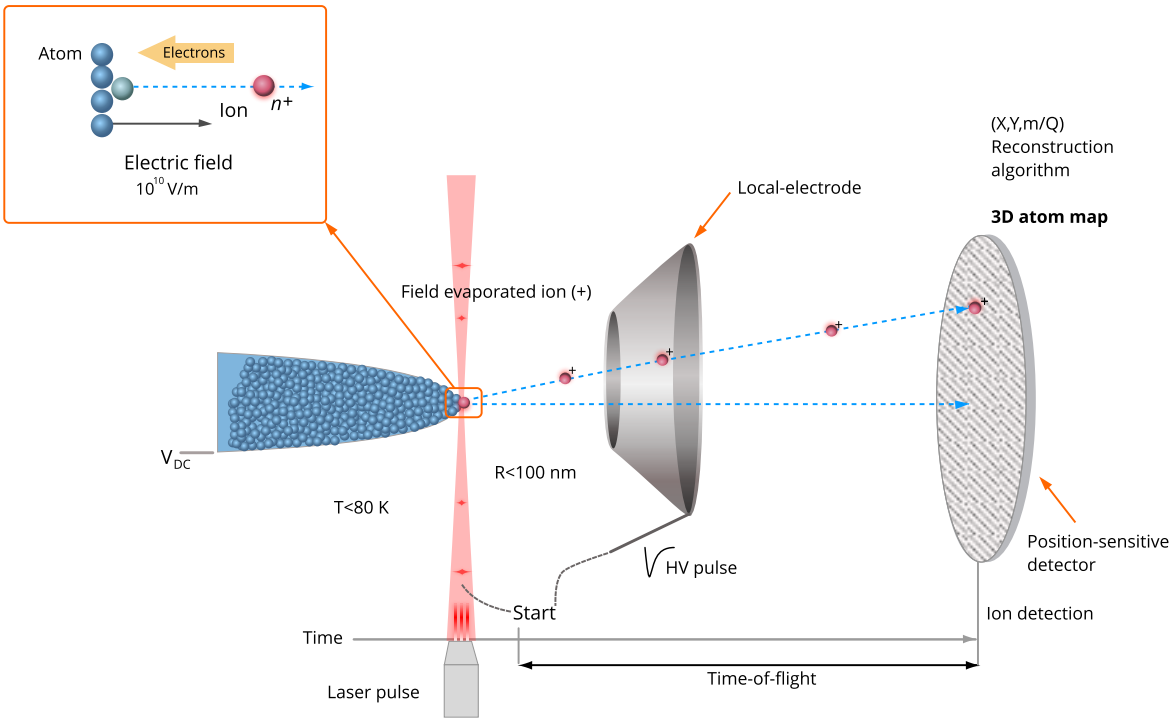


Image and schematic of a typical APT needle.

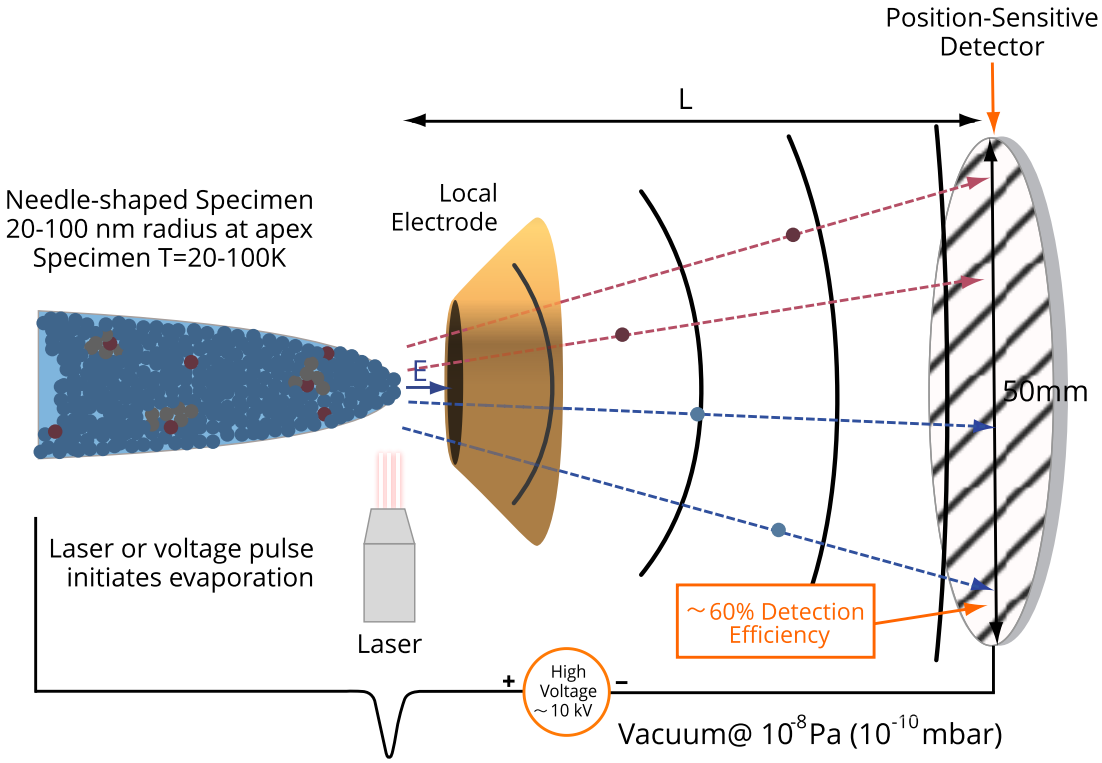
The DC voltage applied to the sample is usually just below the value required to initiate field evaporation. In the case of electrically conductive materials, voltage pulses are applied to the tip, creating an electrical field strong enough to induce field ionisation. For less conductive samples, laser pulses are applied enabling thermal evaporation of ions from the tip. Laser pulsing is generally used for higher resistivity materials such as semi-conductors or minerals where high voltage pulses are insufficient to promote field ionisation.

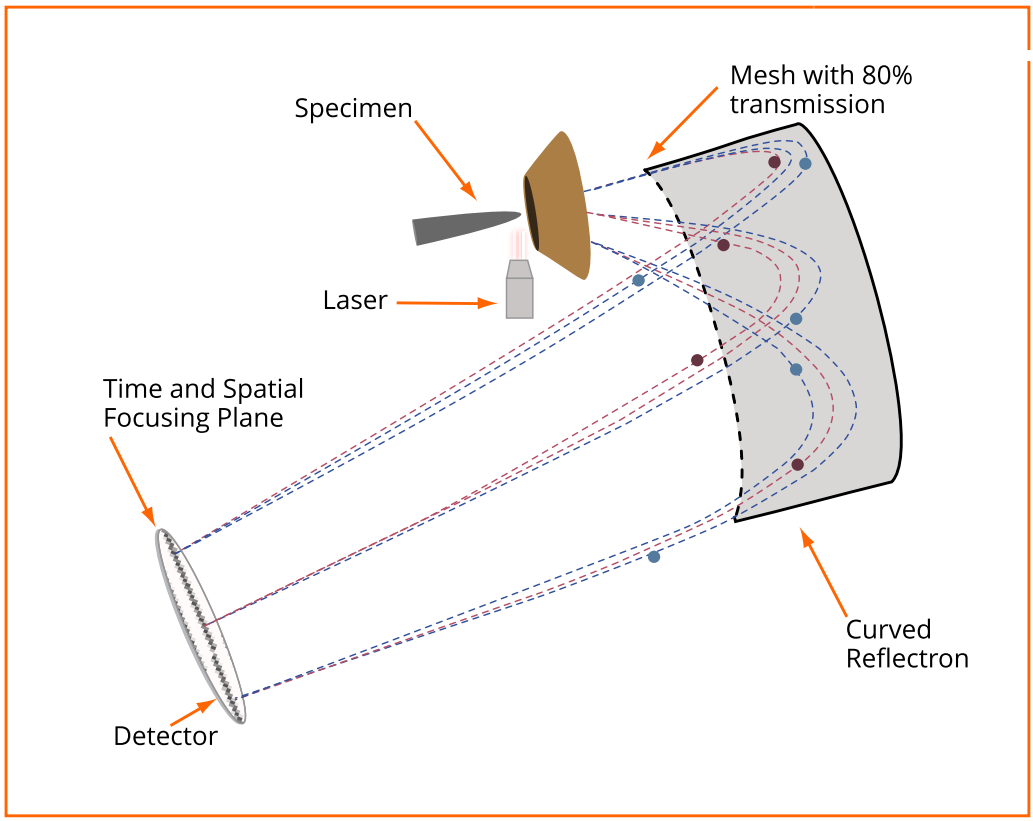


Schematic of APT.

Two flight path options

Modern atom probe microscopes utilise two main types of flight paths, depending on the configuration of the instrument. In one case, a reflectron lens acts as an electrostatic mirror that modifies the flight path of ions with different kinetic energies. This improves the mass resolution, while maintaining the field-of-view, but slightly decreasing the overall detection efficiency. Alternatively, a straight flight path enables a higher detection efficiency but generally has a poorer mass resolving power.

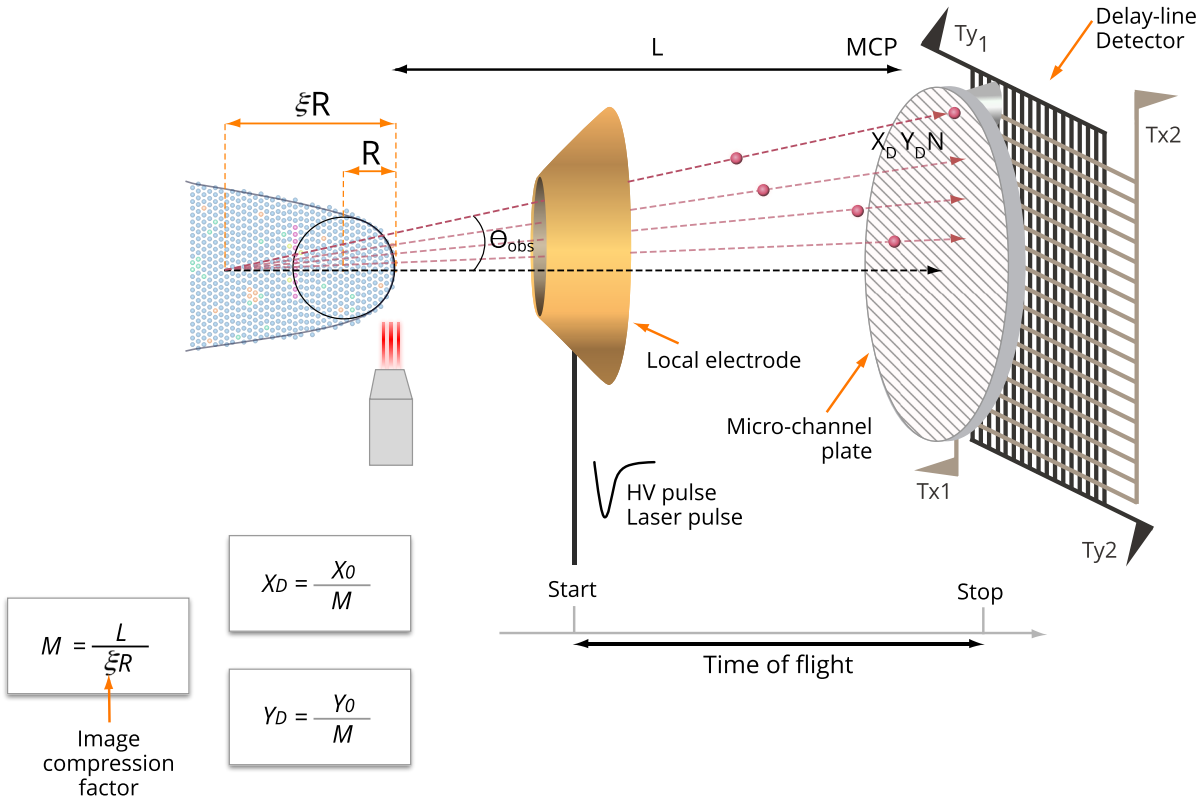




Schematic of straight flight path vs reflectron APT.

Position of atoms within the sample

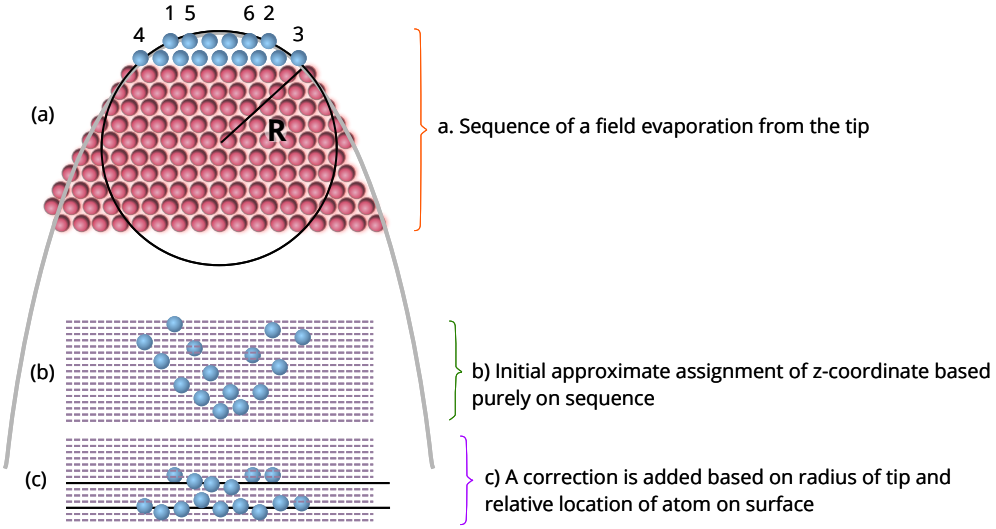
In a LEAP®, an electric potential is applied between the sample (negatively biased) and a counter electrode (local electrode). Once atoms have been field ionised, the now positively charged ions fly through a small aperture in the local electrode towards a micro-channel plate detector (MCP) that converts each ion into a cloud of electrons. The electron cloud then hits a delay-line position-sensitive detector and the detector position Xd and Yd of each 'hit' is recorded (i.e. the exact position at which the atom hit the detector is logged).
Using the simple flight path geometry and the assumption that the sample is a hemispherical cap on a truncated cone, the ion detector coordinates can then be used to determine the original lateral (x and y) positions of the atoms in the sample via a back projection algorithm.



Schematic showing basic reconstruction algorithm.

Atomic position in the sample

The in-depth coordinate (z) are directly determined by the sequence of evaporation. This approach assumes a uniform atom-by-atom, layer-by-layer evaporation of ions across the surface of the tip.
The approaches to reconstruction described here are the most commonly adopted. They are not perfect, and are known to lead to aberrations in certain datasets, especially for samples that are highly heterogeneous or for samples that do not have a hemispherical tip. Ongoing research is underway to improve the reconstruction of atom probe data and for more information, the reader is referred to the scientific literature on the topic.



Sequence of a field evaporation from the tip
Atoms near the tip and atoms with the least number of surrounding neighbours are preferentially evaporated.

Schematic showing basic z-coordinate reconstruction.

Chemical identification

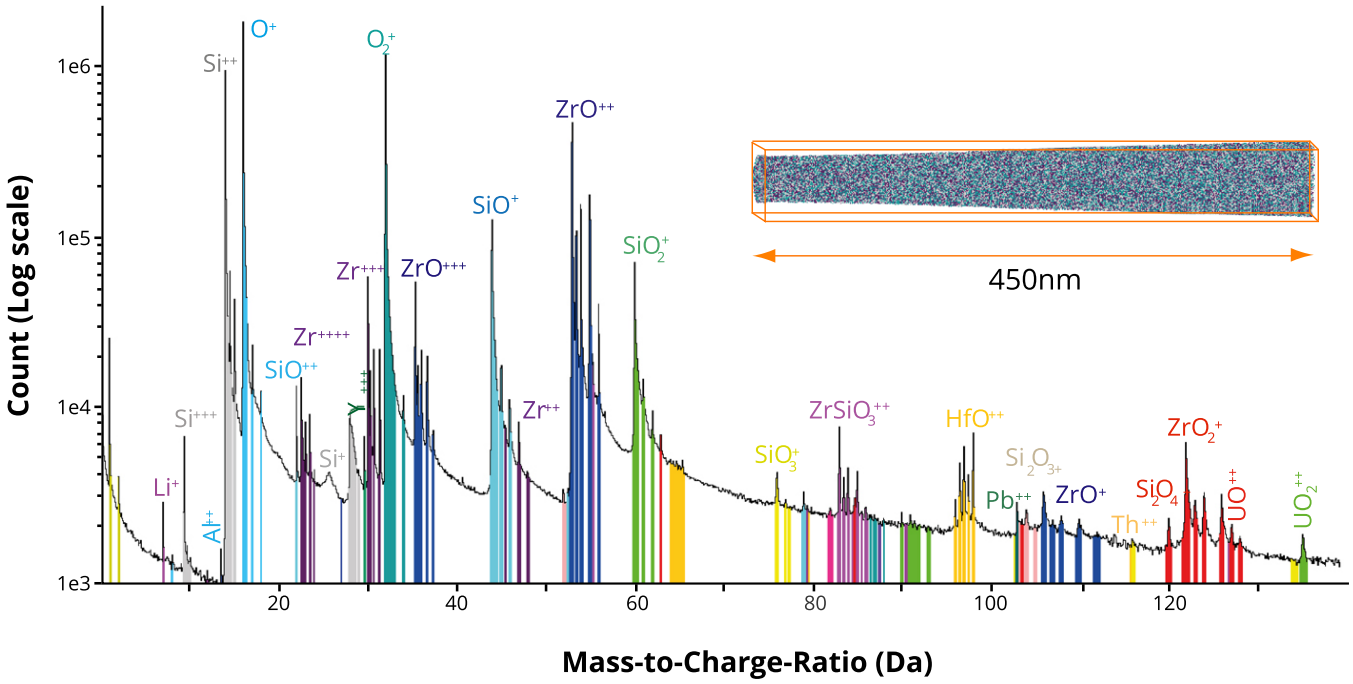
The high-voltage or laser pulse that triggered the evaporation event is correlated to the corresponding ion(s) detection. From information known about the time that the pulse occurred and information known about when the ion was detected, the time-of-flight can be calculated. Time-of-flight is then converted into a mass-to-charge state ratio (m/q) (units of Dalton (Da)), that is used to chemically identify each ion. m/q is derived from the time-of-flight according to:

$$\frac{m}{q} = 2eV\frac{t_{TOF}^2}{L^2}$$

Where q is the charge state of the ion (e.g. +1, +2, +3), m is the mass of the ion, e is the charge of an electron, V is the voltage applied to the specimen, L is the flight path (distance from specimen to detector, generally 90 mm), tTOF is the time-of-flight (tTOF = t1 - t0, where t1 is the time at which the ion reaches the detector and t0 is the time at which pulse was applied). An example of a doubly charged aluminium ion is shown below.

$$^{27}Al^{2+} \quad \frac{m}{q} = \frac{27}{2} = 13.5\ Da$$

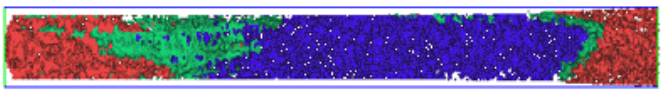
A histogram of the resulting mass-to-charge of ions can be created providing a mass-to-charge spectrum, commonly referred to as an APT mass spectrum. An image of a typical atom probe mass spectrum (from the mineral Zircon) can be seen below.



Typical APT mass spectrum (from the mineral Zircon).

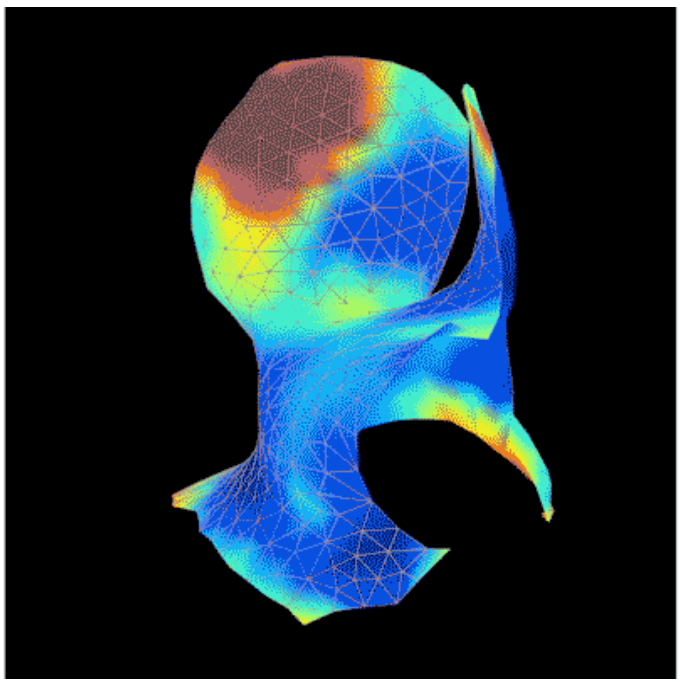
3D data visualisation

A typical APT experiment results in a large number of collected ions (up to several hundred million), which are investigated using various methods.
The most prevalent software package to visualise APT data is the Imago Visualization and Analysis Software (IVAS™), which is the CAMECA® LEAP® commercial software. This software enables the reconstruction of APT data, through semi-automated steps, and 3D visualisation capability. Once the data is reconstructed in IVAS™ a POS file is created containing (x, y, z and m/q) information. The information contained in the POS file can be used to visualise and process the 3D data. Several data analysis techniques are available in IVAS™ such as isosurfaces, proximity histograms, ion range selections, clipping and so on. These analysis techniques will be described in more detail in the section 'How do I analyse APT data'. The information contained in a POS file can also be opened using in-house programs and visualised with other 3D-visualisation software such as Blender or Avizo.



Isosurfaces of different concentration of Fe in an stainless steel oxide scale.

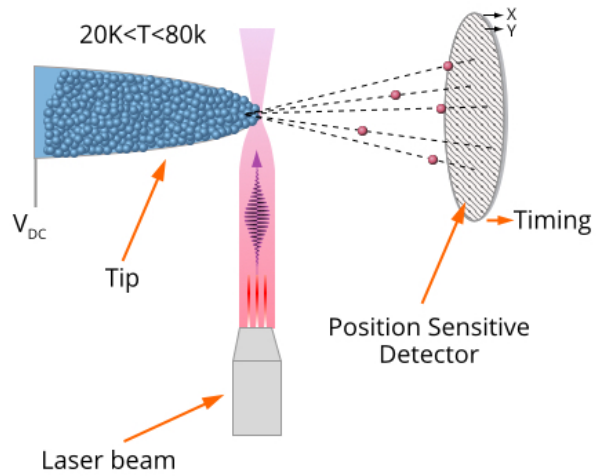
Grid-based data analysis techniques are based on dividing the whole dataset into small 3D volumes. This is called voxelisation, and it facilitates the management of large datasets. Each atom is assigned to a voxel, which is a rectangular prism with size defined by the user. Typically, a voxel size can be based on volume (defined size) or population (defined number of atoms). The voxel size should be optimised with consideration to the analysis time and the statistical significance. Density or concentration analysis is one of the most common techniques that uses voxelisation by volume. The concentration of a particular species can be calculated for each voxel. As a result, a 3D density map can be used to visualise variations in density or composition through the whole dataset. Microstructural features present within the whole volume, such as precipitates or grain boundaries, can be revealed by this technique.

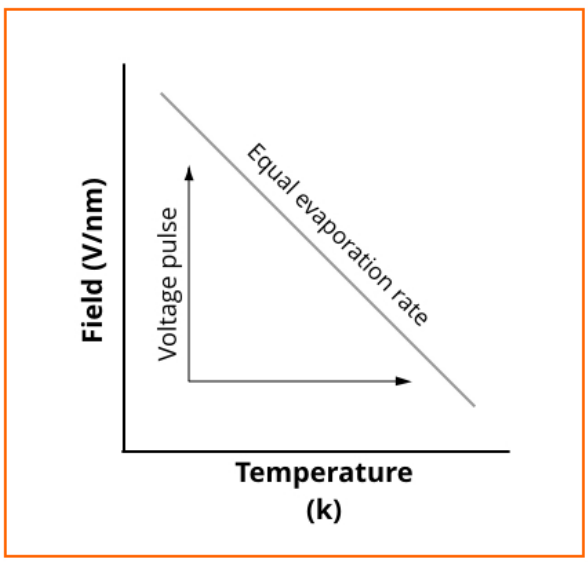


DCOM model of the interface between Ni and YSZ with an interfacial excess map. The mapped ions are NiO.

Laser-assisted APT

The evaporation of the surface atoms from an APT tip relies on field-ionisation triggered by a pulse, either voltage or laser, while a DC voltage is applied to the sample. Modern LEAP® instruments can now generate ultra-fast pulses (picoseconds) and be equipped with UV lasers (355 nm wavelength) that are focused on the tip of the sample. The use of laser pulses to field-ionise surface atoms has increased the range of materials that can be analysed by APT. For example, it is now possible to investigate large band gap materials such as mineral oxides or even bio-minerals. However, there are some challenges in interpreting the data that are related to the intrinsic properties of such materials that result from the complex laser-specimen interaction.
The laser-assisted field ionisation mechanism has been the subject of much debate and is still not fully understood. However, thermally-assisted evaporation is now considered by most to be the main mechanism. Thermal energy in the form of local temperature spikes is applied to the tip by laser pulsing. The increased temperature has the effect of decreasing the required ionisation field. The combination of DC voltage and local temperature rises allows field ionisation of the surface atoms of a tip.



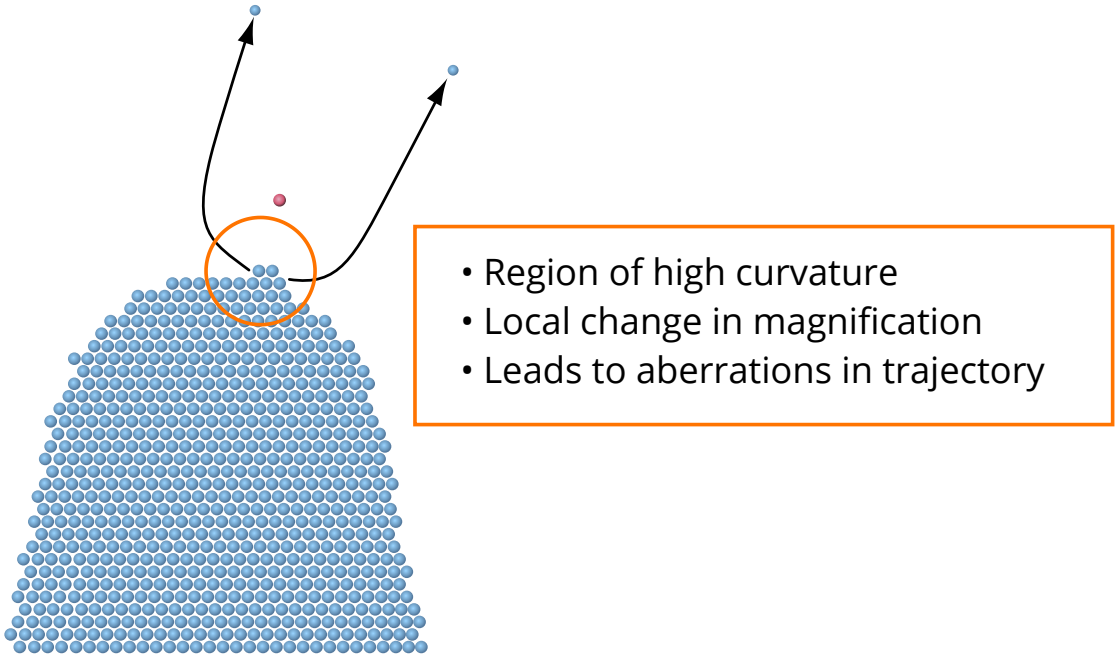


Schematic of laser-assisted evaporation.

In a modern LEAP® equipped with a UV-laser, the spot size of the laser beam is typically < 5 µm. The thermal absorption area is confined to the apex of the tip, and is limited to about the length of the laser wavelength, which is much smaller than the spot size. The small size of the heating zone allows for fast heat diffusion in the apex of the tip, inducing both fast thermally-assisted field ionisation and rapid cooling of the tip.

Spatial resolution of APT

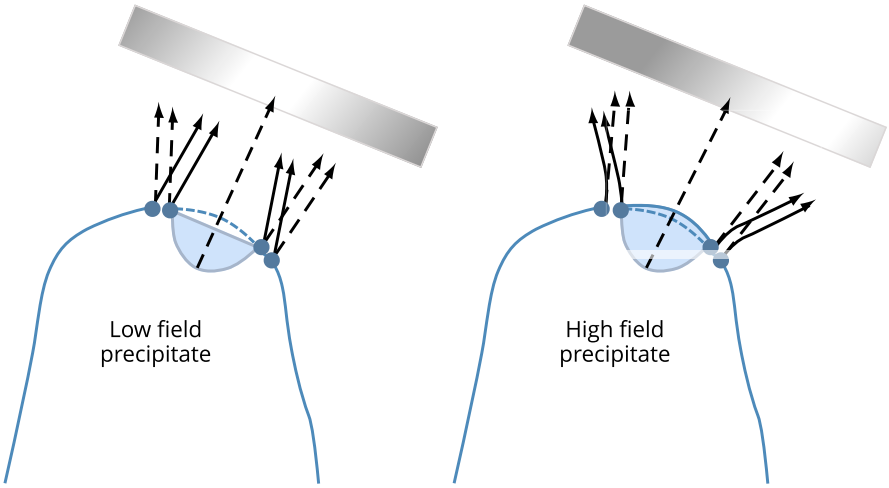
The optimum spatial resolution of APT is estimated to be better than 0.06 nm in depth (z) and the lateral resolution (x, y) below 0.2 nm. However, several factors can affect the spatial resolution.
Variations in the local geometry and composition on the surface of the tip can cause trajectory aberrations in the flight path, degrading the spatial resolution.
The effect of atoms moving at the surface of the tip just before field ionisation is known as surface migration and is a limiting factor for spatial resolution.

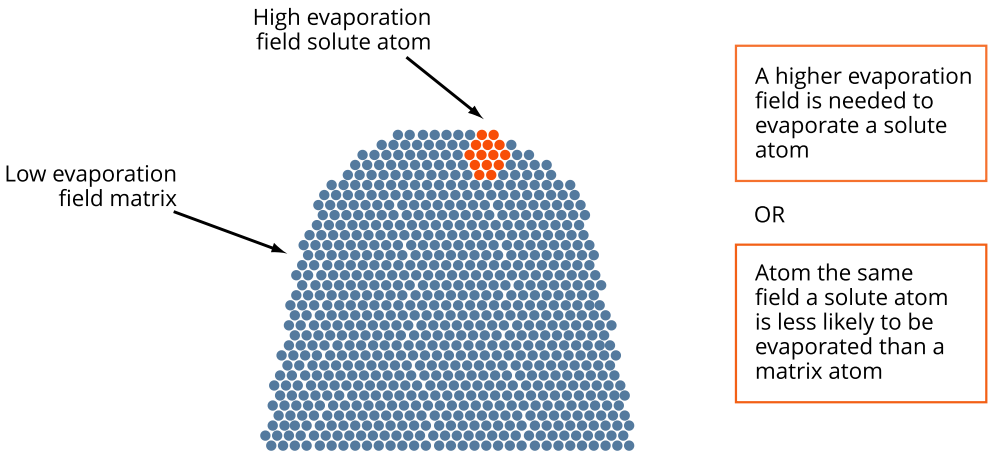


Schematic showing inhomogeneity in the surface topology causing trajectory aberations.

Trajectory aberrations, such as local magnification, can occur as a result of the tip becoming mis-shaped by the retention of, for example, a high field precipitate. Local magnification effects usually involve preferential evaporation or non-evaporation of particular elements that require a higher field of evaporation than the rest of the surface. This can be seen in the image below. This typically occurs when precipitates are present, and It results in a non-uniform sequence of evaporation, which is detrimental for the spatial resolution.

Atomic Density





Schematic showing different evaporation fields causing trajectory aberations.

Finally, the overall accuracy of an APT experiment is limited by the reconstruction algorithm. Since the reconstruction is not perfect (the assumptions it is based on do not always hold true), it is therefore a source of error in atom probe datasets.

Mass resolution of APT

The mass resolving power (MRP) in APT is generally defined as m/Δm, where Δm is measured as the full-width at half-maximum (FWHM) of the peak. MRP is typically up to 500 for voltage-pulsed atom probes and up to 1000 for laser-pulsed atom probes. It can reach more than 2000 with laser-assisted reflectron-equipped instruments.
The mass resolution is dependent on many factors such as voltage pulse fraction or laser energy, pulse frequency, temperature, thermal and electrical conductivity of the specimen and the geometry of the tip. Optimisation of the mass resolution requires testing the experimental conditions for each sample type.

Essential parts of an Atom Probe - The vacuum system

The LEAP® system is designed with three vacuum chambers:

- The load-lock chamber - for specimen transfer in/out of the instrument
- The buffer chamber - used as an intermediate storage chamber
- The ultra-high vacuum (UHV) analysis chamber - where field evaporation takes place

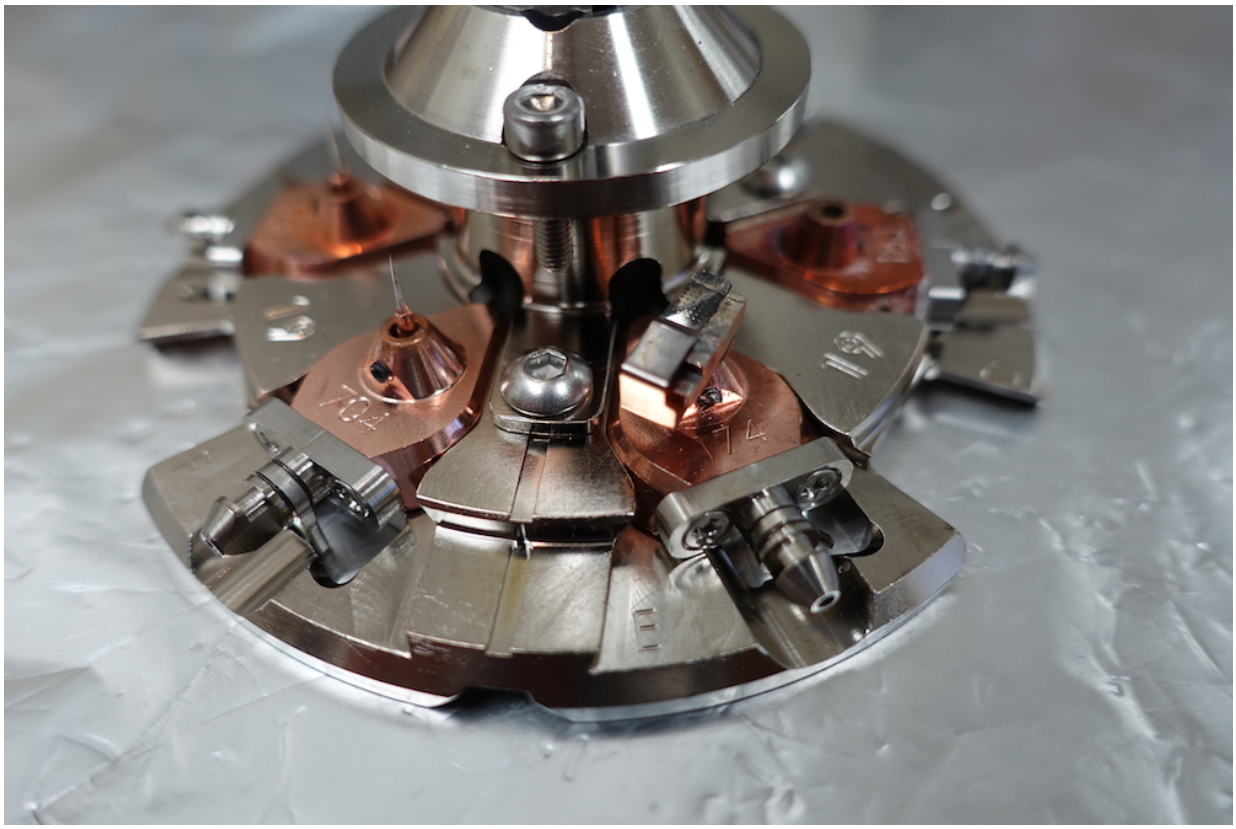
The load-lock chamber allows fast specimen loading from atmospheric pressure to high-vacuum conditions. A high-speed turbomolecular pump is used to reach a typical vacuum of 10^{-3} / 10^{-4} Pa (10^{-5} / 10^{-6} Torr), which can take between 10 minutes and a few hours, depending on the cleanliness of the sample and whether outgassing occurs. A good vacuum is required before opening the gate valve between the load-lock and buffer chambers.

The buffer chamber can be used to store specimens and local electrodes under UHV conditions. It also allows fast specimen transfer in and out of the analysis chamber. In the buffer chamber a typical vacuum of 10^{-5} / 10^{-7} Pa (10^{-7} / 10^{-8} Torr) is obtained using a turbomolecular pump. It can take between 10 minutes and a few hours to reach this vacuum level depending on how long the specimen was kept in the load-lock chamber.

The analysis chamber is home to the specimen stage, the detector, the ion pump, the optics (for laser assisted APT) and the reflectron (for reflectron systems). It is always kept under UHV conditions with a typical vacuum of 10^{-7} / 10^{-9} Pa (10^{-9} /10^{-11} Torr). This is achieved using an ion pump and a titanium sublimation pump. In addition, the cryogenic specimen stage acts as a third vacuum pump.

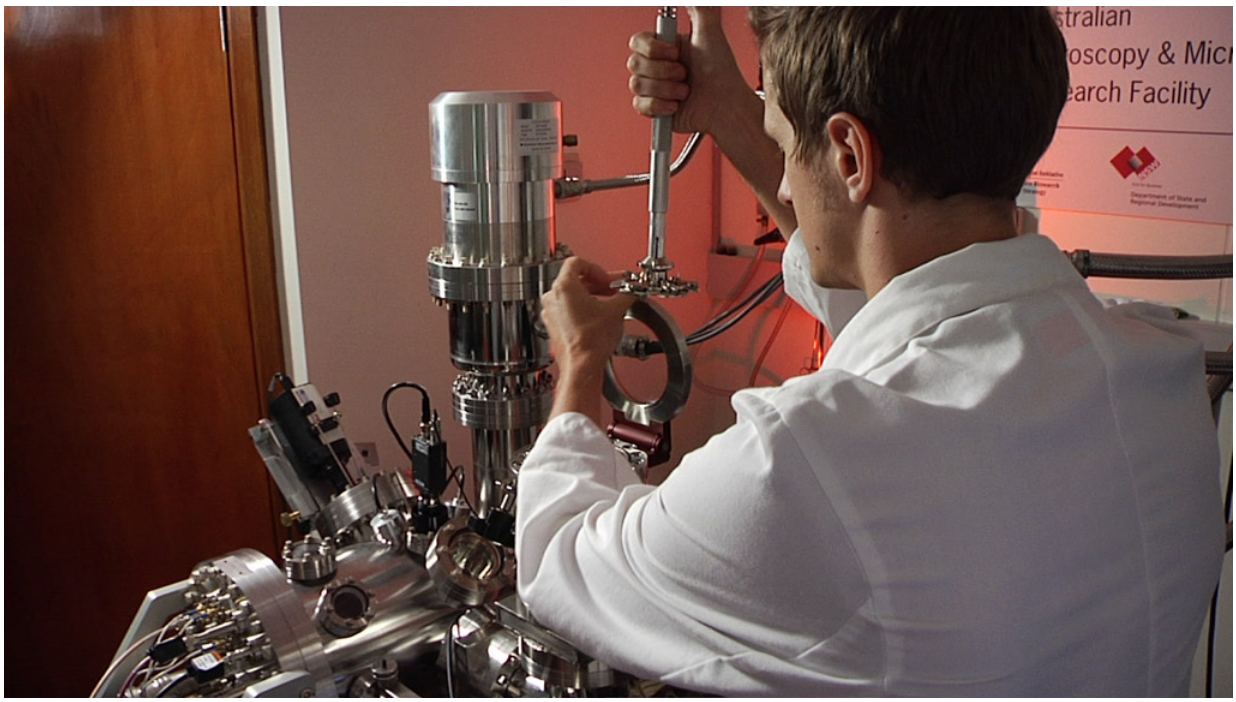
Handling and transferring samples

In APT, the most common samples are individual needles, microtip coupons or half-cut TEM grids. These samples are usually mounted onto a stub which in turn can be inserted into a puck (see image below). The puck can then be inserted into a carousel which can hold a number of pucks. There are three carousels in a LEAP® atom probe (some models can accommodate four).



Picture of a carousel with needle shape sample and a typical microtip array.

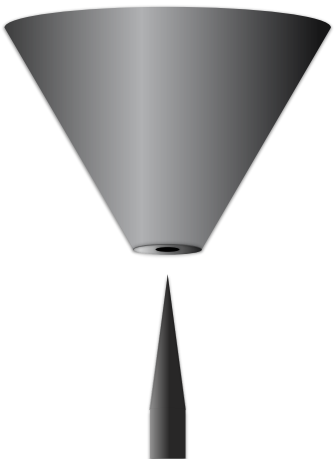
A motor-driven vertical transfer rod allows movement of the carousels between the load-lock and the buffer chamber. The pucks are transferred between carousels and in/out of the analysis chamber by a hand-driven horizontal transfer rod. Gate valves isolate each chamber and an interlock system controls the maximum pressure at which a valve can be opened.



Video or animation showing carousels transfer between chambers and sample transfer between buffer and analsysi chamber.

The local electrode

The local electrode is an essential part of the LEAP® as it creates and confines the electric field to the very small specimen tip. In a LEAP®, the local electrode is funnel-shaped with a 40 µm diameter aperture at the apex of the electrode. The specimen is aligned to the centre of the local electrode aperture and positioned at a distance of around the diameter of the aperture, i.e. 40 µm .

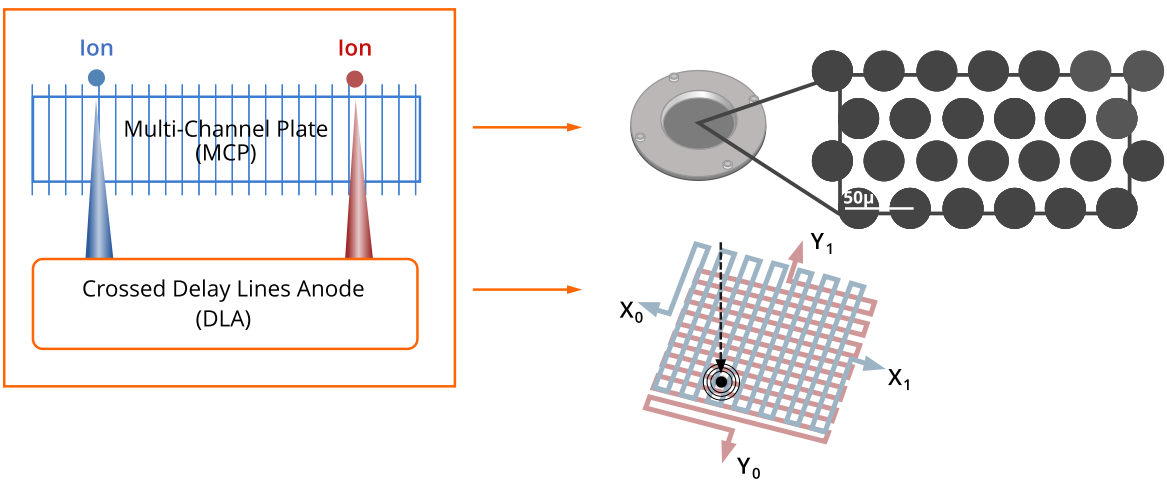


Schematic of local electrode with sample.

Ion detection

The detector in a LEAP® consists of a Multi Channel Plate (MCP) assembly and a crossed Delay Line Anode (DLA).

The MCP assembly is an array of tiny capillaries or microchannels, each of which is an electron multiplier tube. When ions enter the microchannels, they hit the walls, resulting in a cascade of electrons. The cloud of electrons propagates through the microchannels and hits a grid of wires, the crossed DLA. When the electron cloud hits the grid, an electrical signal is created and travels through to the end of the delay lines. The position of the original hits from the electron cloud are then calculated by the time differences between the signal arriving at each end of the delay lines, which are recorded by an amplifier time-to-digital conversion system (ATDC).



Schematic of APT detector assembly.

Not all ions striking the detector are detected, with a significant percentage hitting the plate between the microchannels on the MCP surface. The fraction of ions detected is called the detection efficiency and varies from 37 % for reflectron based systems to up to 80 % for new straight flight path LEAP® models.

The voltage control system

During an atom probe experiment the DC voltage that enables field evaporation can go up to more than 10 kV in both laser and voltage mode. Very stable high DC voltage supplies provide voltage not only to the specimen stage but also to the MCP assembly (gain and bias voltage) and the reflectron system. In voltage mode, the voltage pulse is generated by a high-amplitude solid-state pulse generator with a very narrow width and a rise time of less than 1 ns.

The Laser system

In a laser-equipped LEAP®, UV-laser (355 nm) is the most commonly used. The whole laser assembly is enclosed and sealed, from the laser power generation to the final laser pulse, which is focused and aligned on the specimen tip by using piezoelectric mirrors. Laser pulses with a repetition rate of 1 to 1000 kHz are generated. The available energies for the laser pulses vary from a few pJ to nJ. The pulse duration is below 1 ns.

The cryogenic system

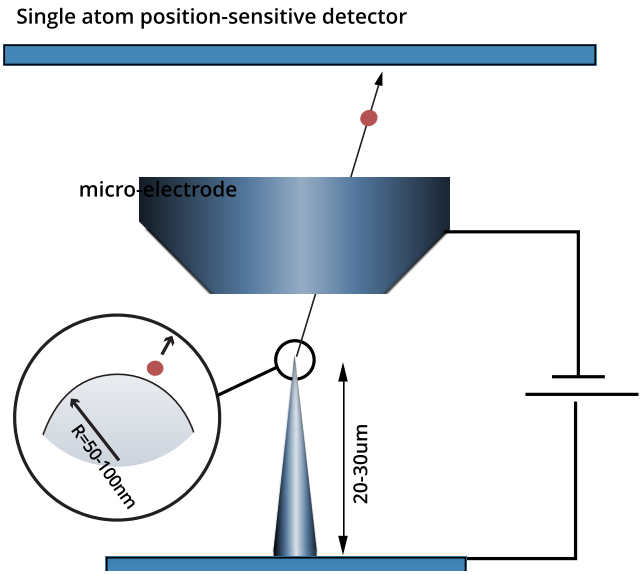
In APT, the sample needs to be held at a cryogenic temperature to limit the thermal motion of atoms and preferential evaporation. Cryogenic conditions also improve the UHV quality in the analysis chamber. The minimum sample stage temperature is typically as low as 20 K in conventional systems. This temperature can be increased, but above about 60 K, the UHV degrades, which can lead to contamination of the ion pump.

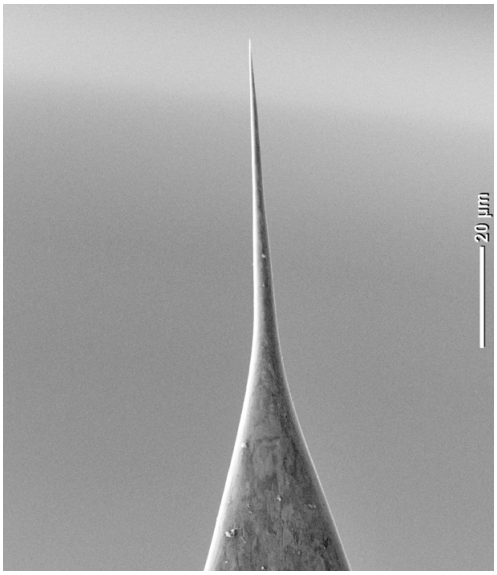
The control system

For local electrode atom probes, the LEAP Control Centre (LCC™) is the manufacturer software that controls almost every function of the instrument, such as the gate valves, specimen stage motions and data acquisition. Visualisation software (DAVis™) enables the live acquisition display. The usage of both softwares is essential to aquire atom probe data.

How do I get good APT data? - Specimen preparation - Specimen requirements

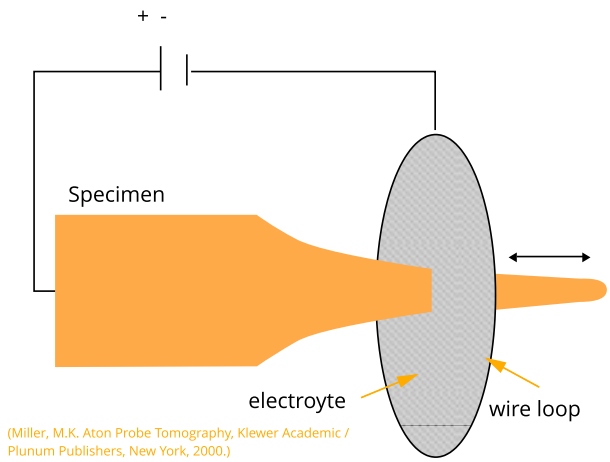
The specimen preparation is an important and challenging part of a successful APT experiment. Requirements for APT specimens include:
- The specimen should be needle-shaped with a radius of curvature at the apex of ~ 50-100 nm
- The specimen length must be ~ 20 - 30 µm and the tip clearance ~ 100 µm
- The specimen must have a smooth surface, be free from protrusions or cracks and be clean enough for ultra-high vacuum conditions
- The feature of interest (i.e. grain boundary, precipitate) must be within hundreds of nm of the specimen apex



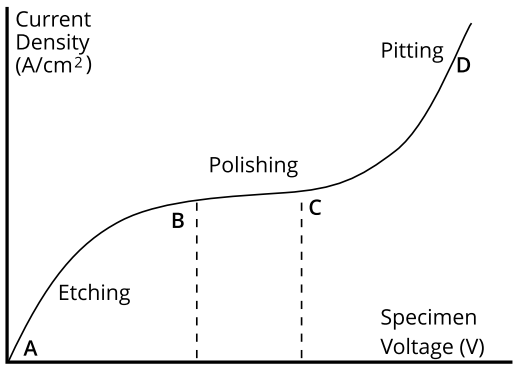


Two main techniques

There are two main preparation techniques: electropolishing and focused ion beam methods. Electropolishing suits conductive materials, such as metallic alloys, as long as site-specific preparation is not required. Focused ion beam (FIB) techniques suit all kind of materials, but are usually applied to high resistivity materials, thin films and site-specific features.

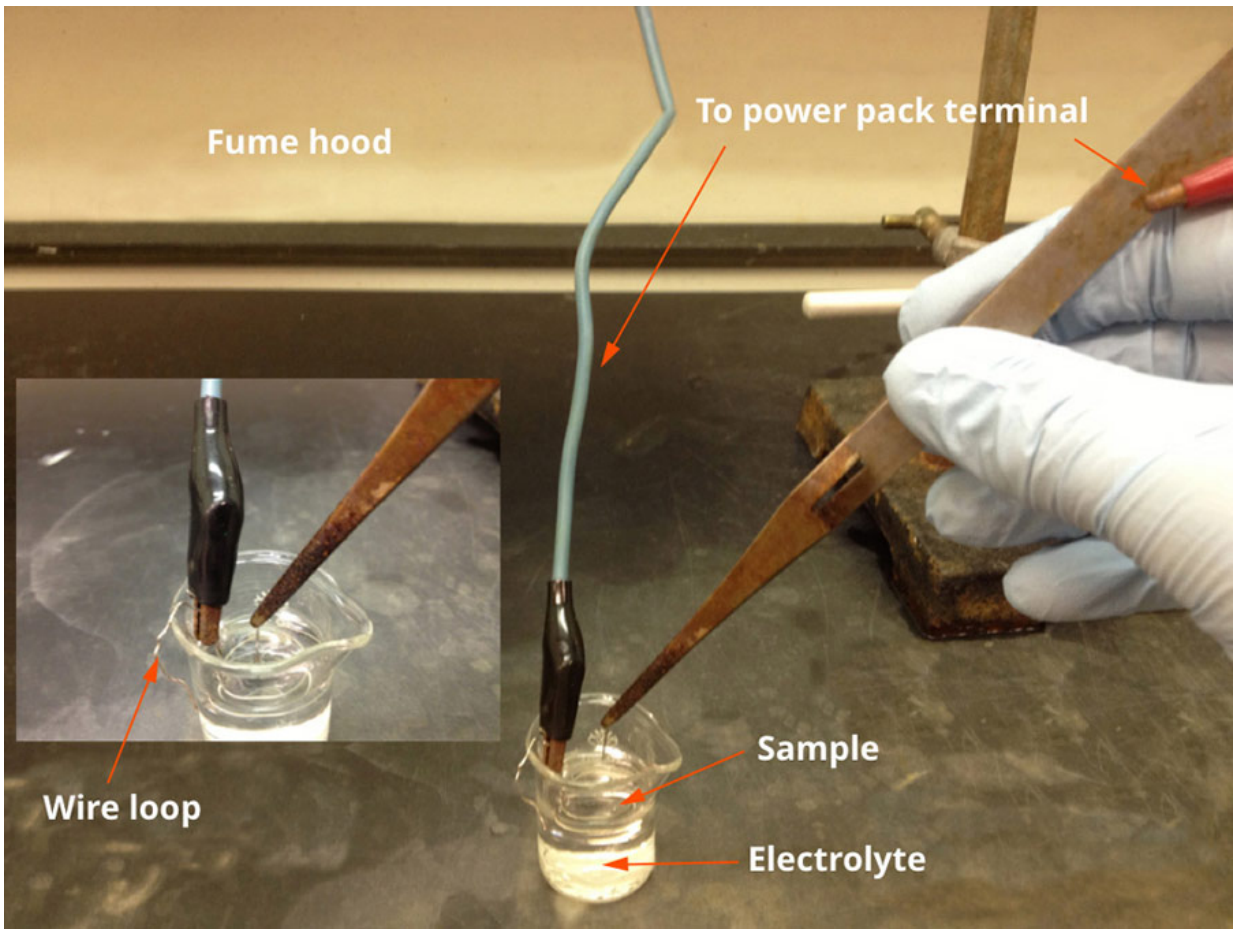


(Miller, M.K. Aton Probe Tomography, Klewer Academic / Plunum Publishers, New York, 2000.)

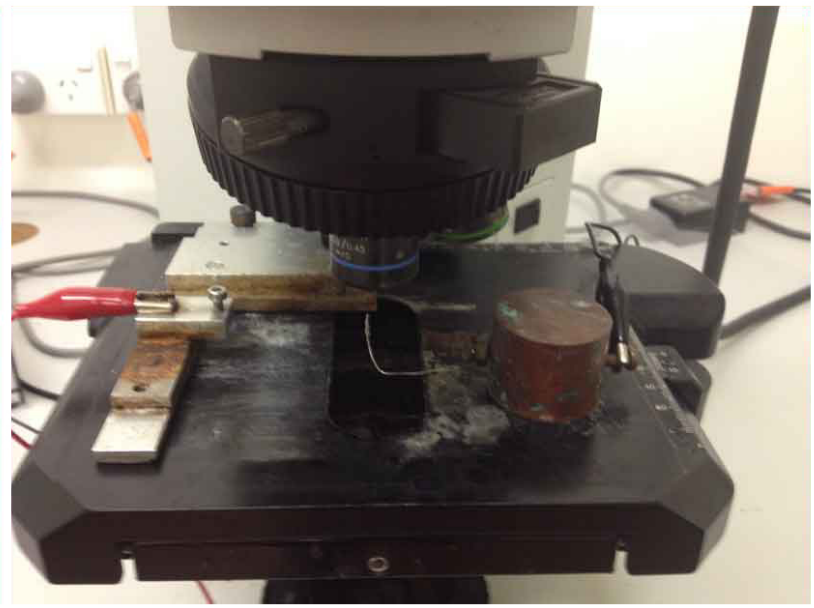


Electropolishing

The electrochemical process removes material from the specimen surface within an electrolytic cell. The electrolyte is suspended within a wire loop (cathode), made from a metal such as platinum or stainless steel. The specimen (anode) is pushed back and forth through the electrolyte until sufficiently sharp.
There are two steps: rough and fine polishing. The choice of electrolyte and specimen voltage depends on the material being polished and are different for rough polishing (stronger electrolyte, higher voltage) and fine polishing (more diluted electrolyte and lower voltage).
Prior to electropolishing, blanks of ~10 mm long with a cross section of 0.3 by 0.3 mm must be prepared from the sample, usually by cutting with a saw. The blanks are then rough polished into an initial needle shape.

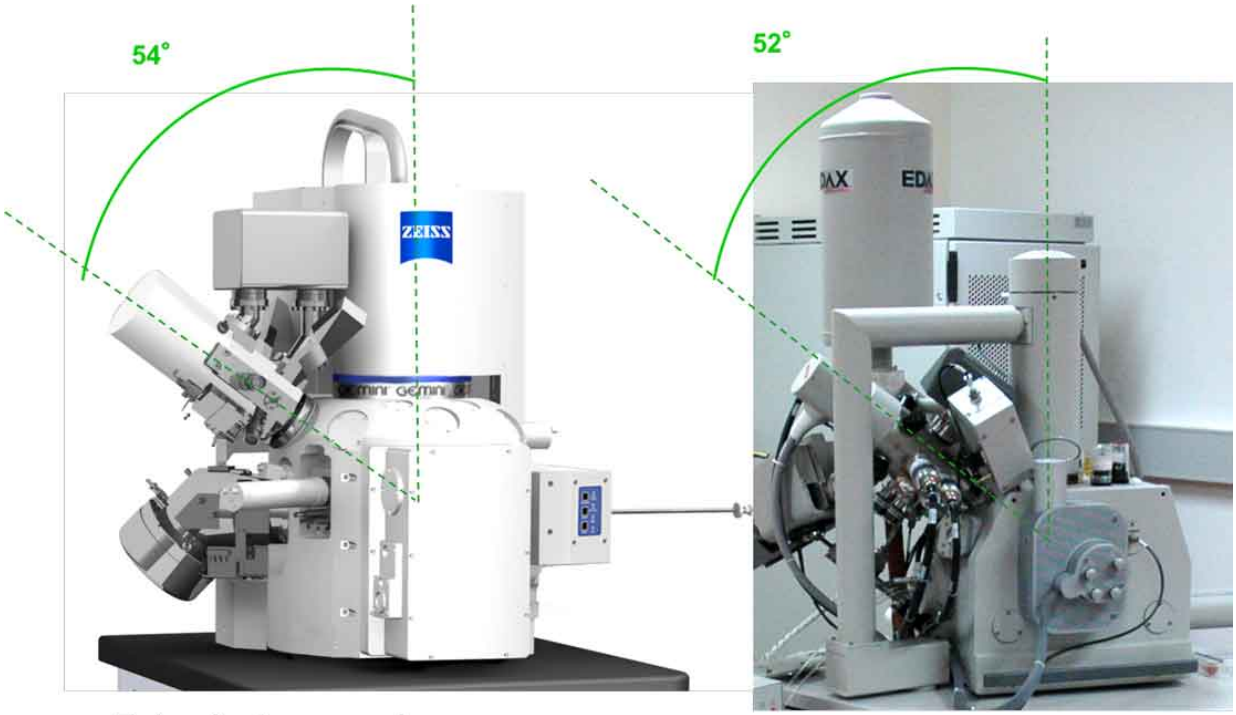


During polishing, with the aid of an optical microscope, the rough polished sample is repeatedly moved in and out of the wire loop, while a DC or AC voltage is applied (typically between 5 V to 20 V). This can be seen in the image below. The sample is ready when a sharp and smooth tip is obtained (< 200 nm in diameter)

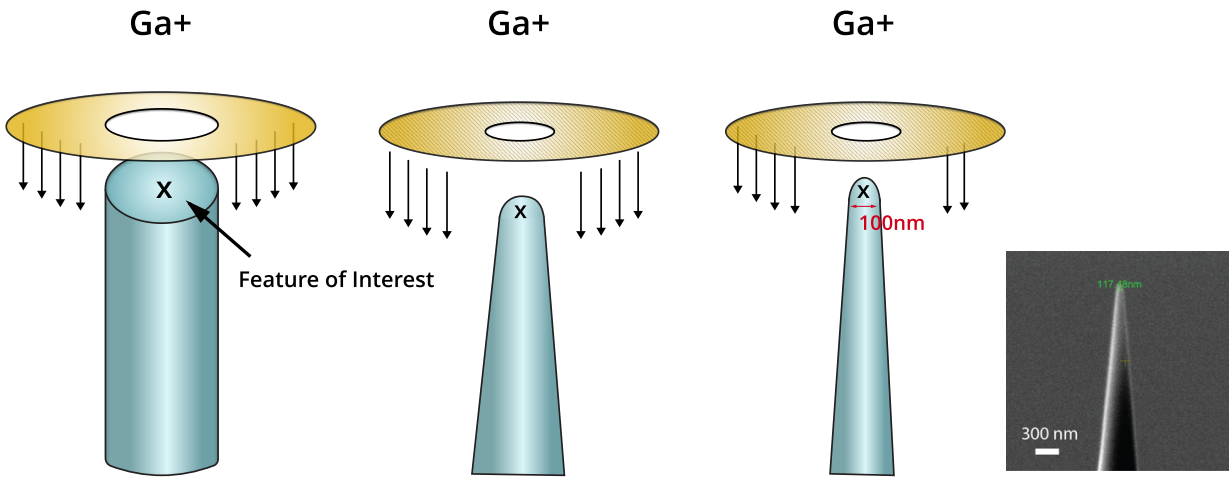


Focused Ion Beam (FIB)

FIB methods rely on the use of a gallium ion (Ga^+) beam to mill away material. This is done in a dual beam (ion/electron) microscope, such as the ones shown below.

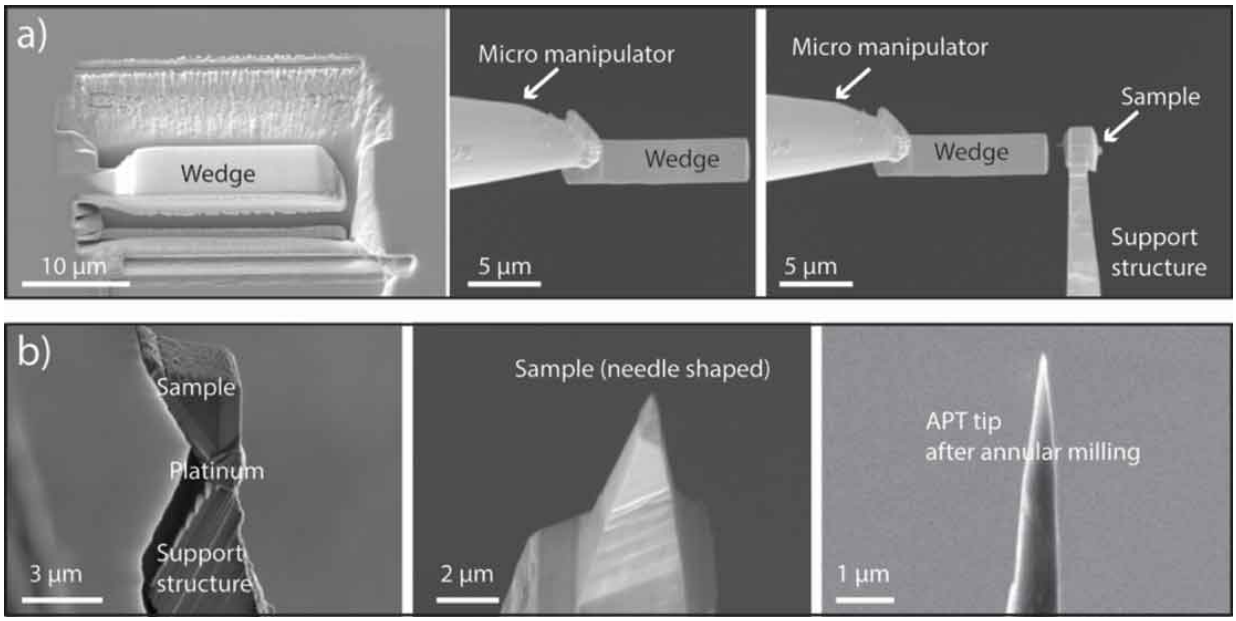


A Ga^+ beam is scanned across the surface of a sample, sputtering away material exposed to the beam. Conical shapes are easily obtained using ion milling, enabling the preparation of needle-shaped samples suitable for APT.
The optimum resolution for imaging during milling is obtained by using a high acceleration voltage (e.g. 30kV) and low ion current (30 pA). However, it is recommended to work at lower ion energies during the final stages of APT sample preparation, to limit ion beam damage. The final stages of APT sample preparation with FIB involve annular milling, enabling the formation of a tip with an even radius.



FIB can be used on an electropolished sample in order to position a feature of interest within the tip. This method is quick and easy, but can only be used for conductive materials (suitable to be electropolished) that have a sufficiently high density of the feature of interest (usually grain boundaries).
Another method uses a thin wedge, typically obtained by tripod polishing. The wedge is attached to a support grid and material is removed using FIB, to produce one or more ~ 4 µm wide posts. APT tips are then sharpened using annular milling.
Most FIB systems can also be used to deposit material onto the surface of the sample. A precursor gas is delivered to the surface region of the sample. When explosed to the ion beam (or electron beam), the gas decomposes into a solid material, allowing deposition onto specific areas of the surface of the sample. A typical gas source used for deposition contains platinum (Pt). Deposition of Pt onto the sample surface can be used to protect a beam-sensitive region of interest or to attach small samples to a micro-manipulator within the instrument chamber.
To perform the FIB lift-out technique, a wedge is first cut from the sample and attached to a micromanipulator needle using Pt-deposition. Pieces of this wedge are then milled and welded to a support structure (typically electropolished metal grids

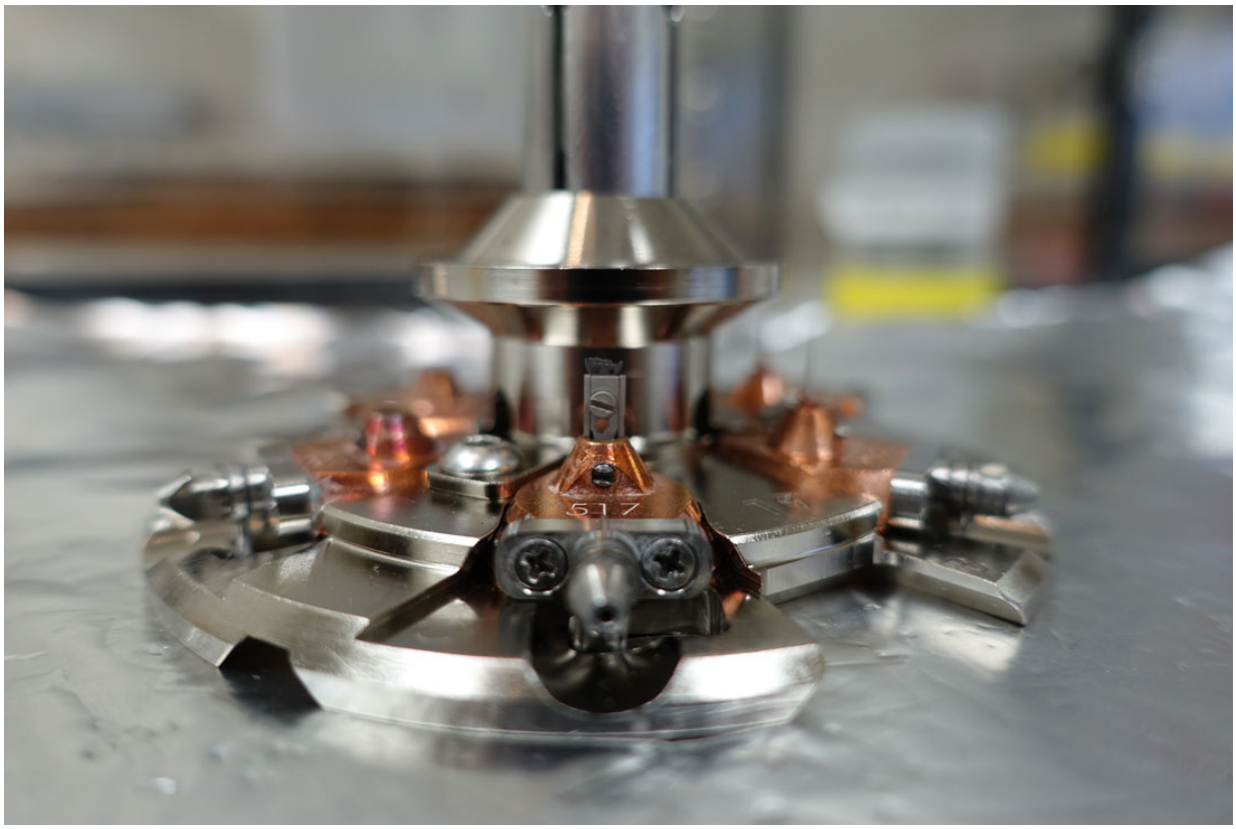
or a commercial micro-tip array) by using ion beam assisted Pt-deposition. The sample is then milled to form a needle shape. The final milling uses annular patterns to form an APT tip with a typical diameter of ~100 nm. All of the steps involved in this lifout technique can be seen in the sequence of images below.



FIB techniques are now routinely used for APT sample preparation and thanks to improved instrumentation and methods, the quality and consistency of specimens prepared by FIB is very high.

Sample insertion in the atom probe - Mounting the sample

The processes of mounting, inserting and coarsely aligning the specimen in the atom probe is common to most atom probe instruments. The sample needs to be placed in a puck, in order to be inserted into the atom probe. Samples prepared by FIB are usually held in a specially-designed sample holder that is compatible with SEM, TEM and APT. Needles prepared by electropolishing are clamped within a stub that can also be inserted into the APT puck. The puck can then be inserted into a slot on a carousel (which can hold multiple pucks).



Inserting the sample in the atom probe

The carousel must first be manually transferred into the load lock chamber. The chamber is vented (by introducing air) in order to bring it to atmospheric pressure so that the gate may be opened.
Once the carousel has been inserted, the load-lock chamber is re-evacuated. Only after the vacuum reaches a suitable level can the gate valve between the load-lock and buffer chambers be opened and the carousel lowered down into the buffer chamber using the vertical transfer rod.
Once the vacuum in the buffer chamber has reached a suitable level, the puck containing the sample can be taken out of the carousel by using the horizontal transfer rod. The gate valve between the buffer and the analysis chamber can then be opened and the puck that holds the specimen of interest is transferred to the specimen stage using a horizontal transfer rod. The puck is then locked in place and the transfer rod removed. The gate valve can then be closed.

Specimen coarse alignment

After the specimen is inserted, the stage temperature must be stabilised, which typically takes 10-30 min. During that time, the specimen can be coarsely aligned to the electrode using optical microscope cameras within the machine.

Collecting data

Once the sample is placed on the stage, the temperature stabilised, and the tip visually aligned to the local electrode, field evaporation of the sample can be initiated. This is achieved by increasing the voltage (and thus the electric field at the sample tip). If the atom probe is equipped with a laser, a choice needs to be made between voltage and laser pulse mode. This choice is usually guided by considerations such as the material's intrinsic properties (mainly electrical and thermal conductivity), the specimen's resistance to field-induced stress (multi-layer samples, nanostructural features) or the level of mass resolving power that is needed (limiting peak overlaps). The choice of experimental parameters is guided by important metrics reflecting the data quality.

Data quality

The mass resolving power (MRP)

The mass resolving power (MRP) is defined as the peak width of the time-of-flight (or mass-to-charge ratio) for each ion type. A narrow peak corresponds to a high MRP and a better mass resolution.

The background level

The background level, also referred to as noise, corresponds to detected events that do not arrive at a specific mass-to-charge ratio and ions that field evaporate out of sync with the pulse. This noise is usually related to the material itself and the residual gas present in the analysis chamber. As much as possible, the background level should be minimised.

Multi-hits events

Multi-hit events are defined by the simultaneous evaporation of two or more ions from the sample surface. If several ions hit the detector in close proximity to each other and within a short time span, ions may not be recorded, resulting in a drop of the compositional accuracy. Species that tend to field evaporate in bursts, such as carbon or boron, may be under represented.

Experimental parameters

Base temperature

In APT, the base temperature is usually set between 25 K and 80 K. Surface diffusion is reduced at lower temperature, which increases the data quality. In laser-mode, the mass resolving power improves with lower base temperature. However, low temperature has been shown to increase the chance of tip fracture for some materials.

Pulse rate

The pulse rate in either voltage or laser mode is selected by the user. The laser pulse rate varies from 100 kHz to 1000 kHz (depending on the instrument model). The voltage pulse rate can go up to a maximum of 250 kHz. Usually a high pulse rate is better for data quality as it lowers the background. However, a high pulse rate can limit the detection of high-mass ions. In the case of a material with poor thermal diffusivity, too short a time between high energy laser pulses won't allow the tip to cool down fast enough, reducing the mass spectrum quality.

Voltage pulse fraction

In voltage mode, the combination of DC voltage and pulse voltage should enable field evaporation of all surface elements with the same probability. The DC voltage by itself should be set to a value where it does not cause any field evaporation. The voltage pulse is a fraction of the DC voltage (maximum of 1.4 kV for straight-flight path and 1.7 kV for reflectron) and is responsible for controlling the field evaporation. A small pulse fraction will result in a higher DC standing voltage and evaporation of low evaporation field elements between pulses. A large pulse fraction on the other hand will increase the chance of specimen fracture due to high cycling stresses. The ideal pulse fraction is different for every sample.

Laser pulse energy

In laser mode, ion evaporation is triggered by thermal energy transferred from the laser. The momentary increase in the sample temperature lowers the field required to evaporate ions, which decreases the stress on the specimen, improves the MRP and lowers the background noise. The thermal energy of the laser is absorbed and diffused by the specimen in a non-homogeneous way and depends on the specimen geometry and the properties of the material from which it is made.
A high laser pulse energy usually leads to better data quality. However, if the laser pulse energy is too high it can result in surface migration, complex ion generation, non-uniform surface evaporation and inaccuracies in the measured stoichiometry.

Voltage mode acquisition

Load specimen

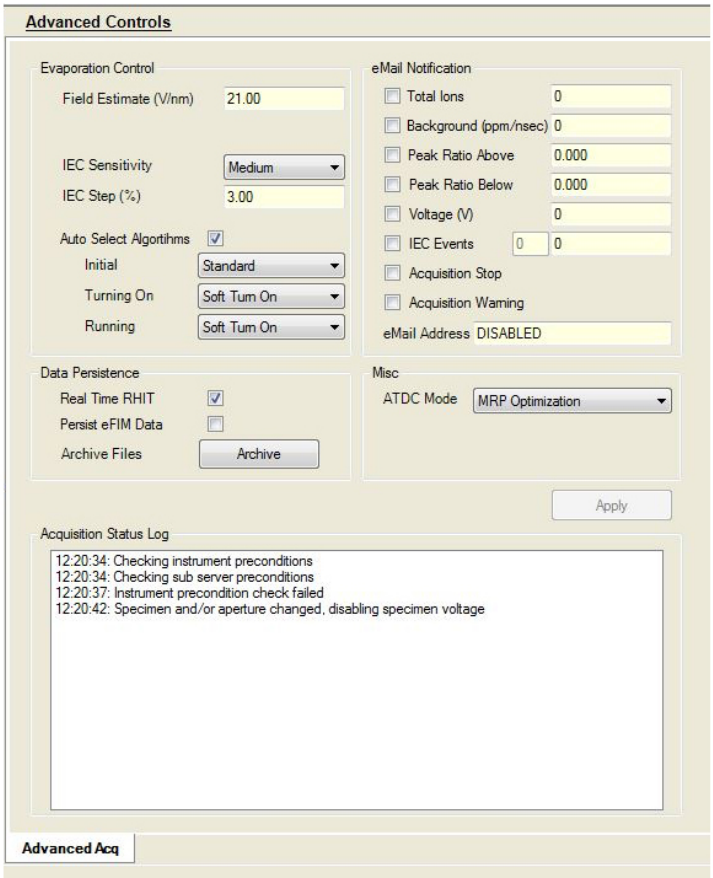
Review the section 'Sample insertion in the atom probe'

Align specimen to local electrode

Review the section 'Specimen coarse alignement'

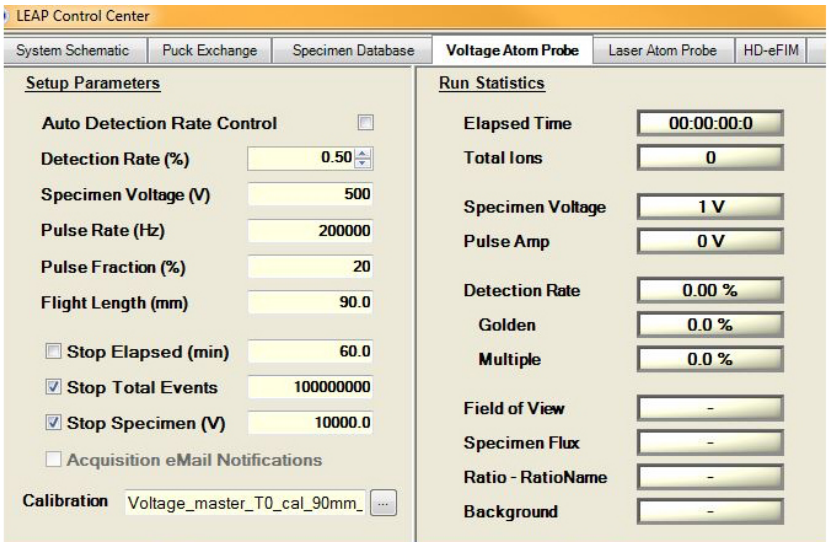
Adjust advanced control settings

Choose an acquisition algorithm that automatically controls the data collection such as the voltage ramp control and interface evaporation control (IEC). On CAMECA® LEAP® instruments, default settings are available in the LEAP Control Centre (LCC™) software (standard, soft turn-on, free running...).



Set initial acquisition parameters

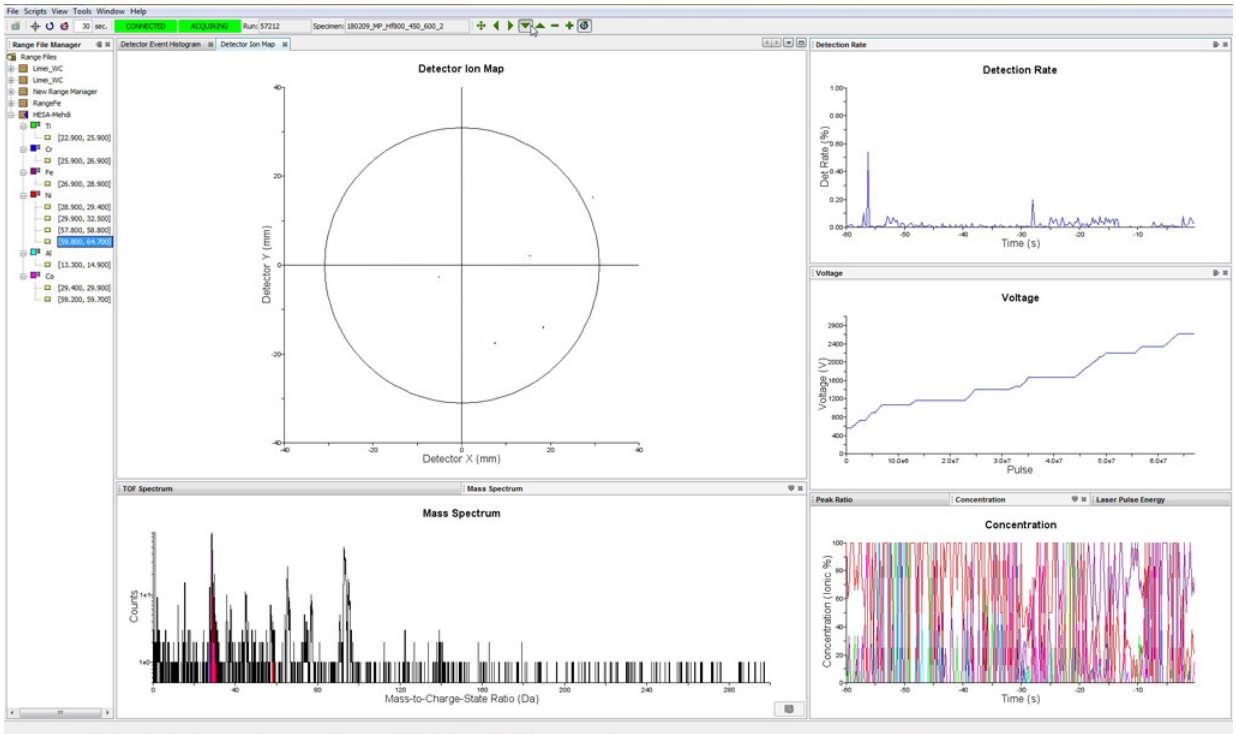
Detection rate (%): start at the lowest available. Adjust when data acquisition has stabilised
Specimen voltage (V): start at the lowest available specimen voltage (500 V)
Pulse rate (Hz): choose the highest pulse rate resulting in heaviest element arriving within a single pulse window
Pulse fraction (%): choose typically between 10% and 30% depending on the material
Auto detection rate control check box: Check the box for voltage to ramp automatically according to the algorithm selected. If uncheck, ramp the voltage manually.



Start data acquisition

Align specimen to detector

When the first ions start to appear (in live) on the detector event histogram move the sample slowly using the motion controls to center the circular shadow of the electrode on the detector.
In a second stage, adjust the distance between the tip and the local electrode so the circular shadow fills about two third of the detector.



Adjust parameters during live acquisition

Once the acquisition is stable (regular and homogeneous evaporation of ions) check the auto-evaporation control box and set stopping conditions to the desired parameters (time, number of ions collected, voltage maximum)

Laser mode acquisition

Laser pre-alignement

Before starting a laser mode experiment, the laser needs to be pre-aligned with a standard specimen such as a Silicon (Si) pre-sharpened microtip. As a result, the laser position and focus will be within a good range in relation to the local electrode. The pre-alignment process is as follows:

- Insert Si pre-sharpened microtip
- Run in voltage mode and align tip to local electrode
- Manually align the laser to the specimen using the manual alignment tab until a bright spot is visible on the apex of the tip
- Fine alignment of the laser (see steps below - maybe create a link to fine alignment of the laser)
- Focusing the laser on the tip using the focus scan function (start with scout focus scan and finalise focus with standard focus scan).

Load specimen

Review the section 'Sample insertion in the atom probe'

Align specimen to local electrode

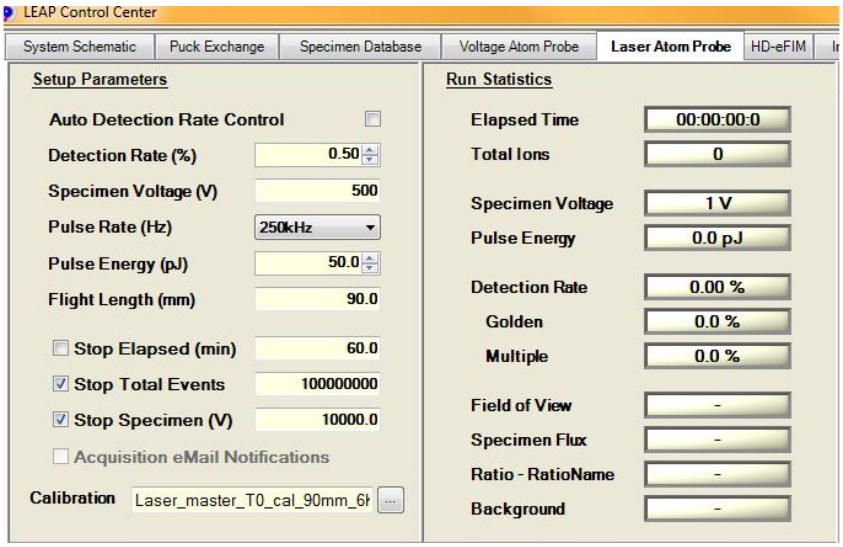
Review the section 'Specimen coarse alignement'

Adjust advanced control settings

Choose an acquisition algorithm that automatically controls the data collection such as the voltage ramp control and interface evaporation control (IEC). Default settings are available in the Leap Control Centre (LCC™) software (standard, soft turn-on, free running...).

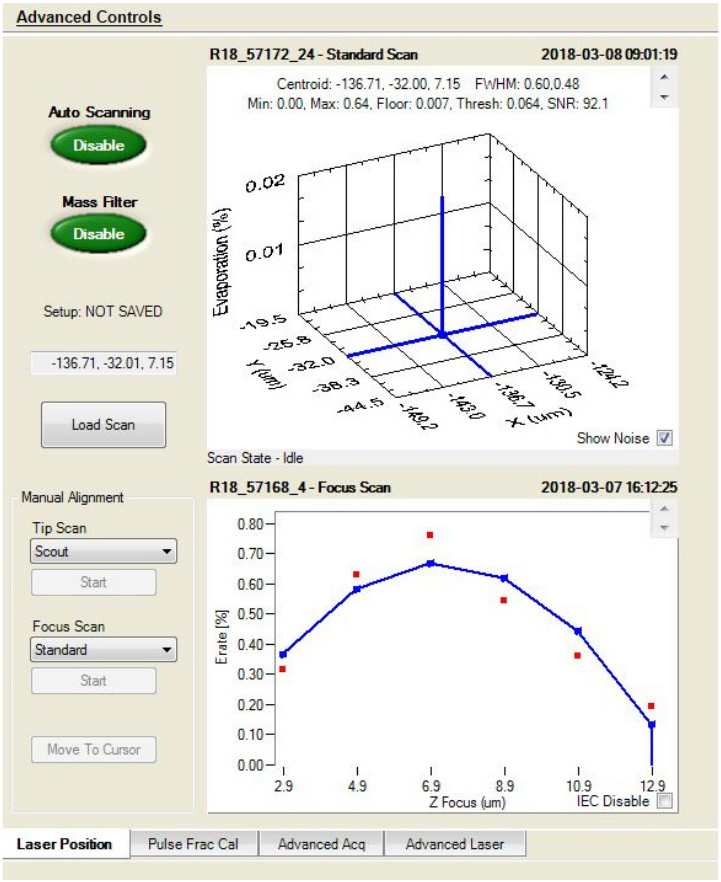
Set initial acquisition parameters

Detection rate (%): Start at the lowest available. Adjust when data acquisition has stabilised
Specimen voltage (V): Start at the lowest available specimen voltage (500 V)
Pulse rate (Hz): Choose the highest pulse rate resulting in heaviest element arriving within a single pulse window
Pulse energy (pJ or nJ): Choose typically between 10 pJ and 100 pJ, depending on the material
Auto detection rate control check box: Check the box for voltage to ramp automatically according to the algorithm selected. If uncheck, ramp the voltage manually.



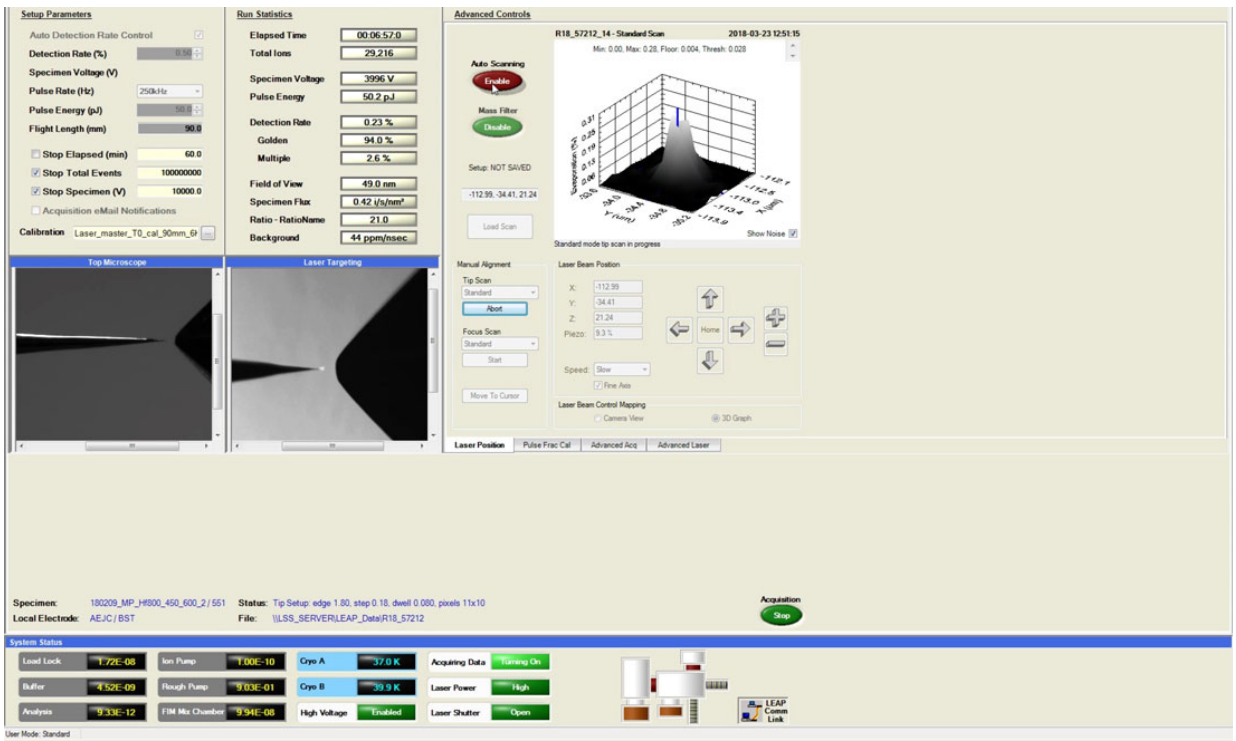
Adjust laser scan parameters

In laser advanced tab, set the settings for each tip scan mode and focus scan mode as required (scout, standard and drift comp).



Align the specimen apex to the laser

Move the specimen stage to align the tip to the laser so a bright spot at the apex of the tip is visible.

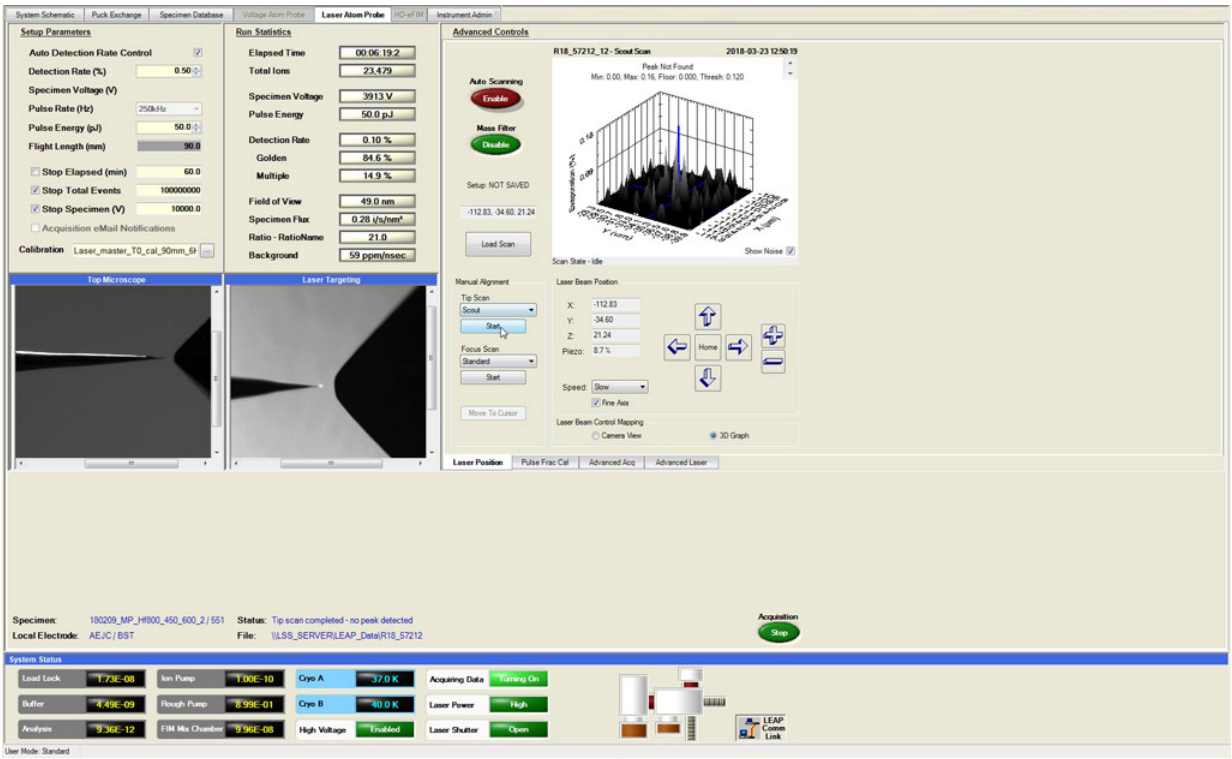


Start data acquisition

Align specimen to detector (fine laser alignment)

Perform scout laser scan regularly until the first ions start to appear (live) on the detector event histogram.
Move the sample slowly using the motion controls to center the circular shadow of the electrode on the detector and immediately perform laser scan (scout or standard) to finely align the laser to the tip apex.
Repeat the process (move sample followed by laser scan) until the circular shadow of the electrode is centered.
Adjust the distance between the tip and the local electrode so the circular shadow fills around two thirds of the detector

and immediately perform laser scan (scout or standard) to finely align the laser to the tip apex.
Once the specimen is aligned, perform a last fine laser scan (standard scan) to complete the laser alignment.



Adjust parameters during live acquisition

Once the acquisition is stable (regular and homogeneous evaporation of ions) set an automated regular drift compensation laser scan, check the auto-evaporation control box and set stopping conditions to the desired parameters (time, number of ions collected, voltage maximum).

End of data acquisition

In voltage and laser mode, the atom probe experiment will stop automatically when the:

- Number of ions collected reaches the maximum value, set by the user
- Specimen voltage reaches the maximum value, set by the user
- User manually stops the acquisition
- Specimen fractures, resulting in the voltage ramping up to the maximum value, set by the user

In laser mode, the voltage ramping will be automatically disabled after the laser loses track of the sample as a result of tip failure or laser drift. The user will have to manually action a laser scan to re-initiate ions evaporation (in the case of laser drift) or stop the acquisition (in the case of tip failure).
In all cases, after the acquisition is stopped the data collected is saved in two different formats: RRAW (or STR for new LEAP® model), which contains unfiltered instrument data for every pulse and RHIT, which contains the processed RRAW (STR) files with identified ion hits.

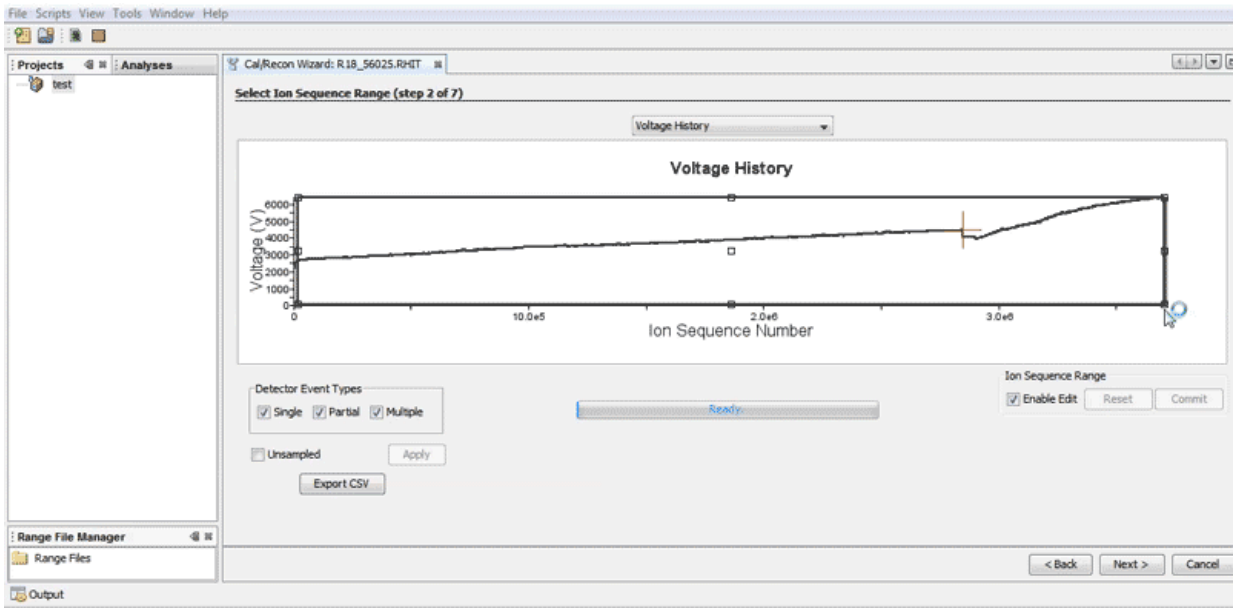
Data processing and reconstruction - Introduction

The conversion from the RHIT raw data into reconstructed positions and chemical identification is currently done via a multiple step process using the software IVAS™ from CAMECA®. The semi-automated steps are:

- Selection of ion sequence range
- Selection of detector region of interest (ROI)
- Time of flight corrections
- Mass calibration
- Ranged-Ion assignment
- Reconstruction

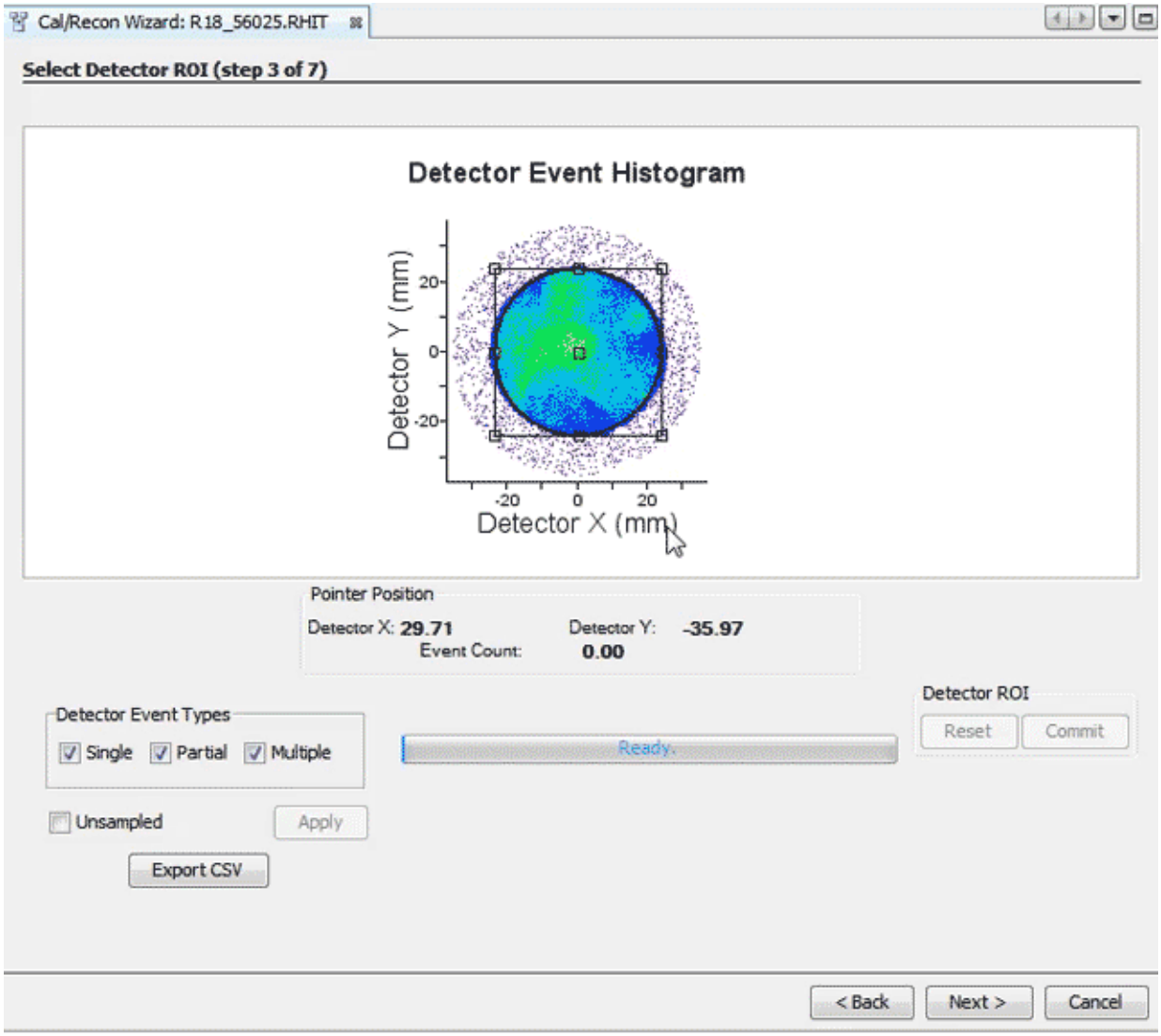
Selection of ion sequence range

In this first step, the user is asked to select a range of ions to include in the reconstruction. Using the voltage vs ion sequence number plot, the user adjusts a rectangle to select a range of ion numbers and voltage to be included in the reconstruction. It is usually recommended to exclude data containing artifacts such as at the very begining of the experiment when the data acquisition is unstable, and regions containing sample micro-fractures or local electrode noise.



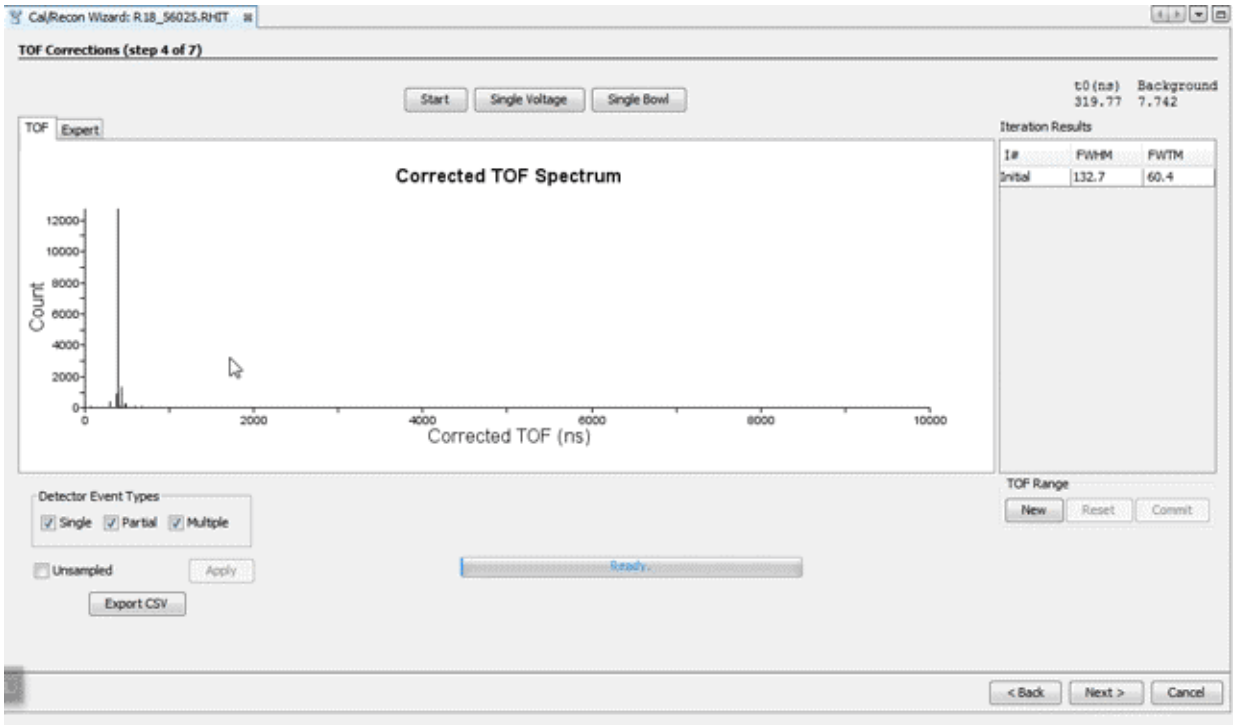
Selection of region of interest (ROI)

In this step, the user is prompted to choose the spatial region of the detector from which to include the ions in the reconstruction. From the detector ion hit-map, the user can select the circular area. It is usually beneficial for the data quality to select most of the ions. However, it is sometimes possible to improve data quality by removing rough edge regions due to the non-uniform local electrode edges.



Time of flight corrections

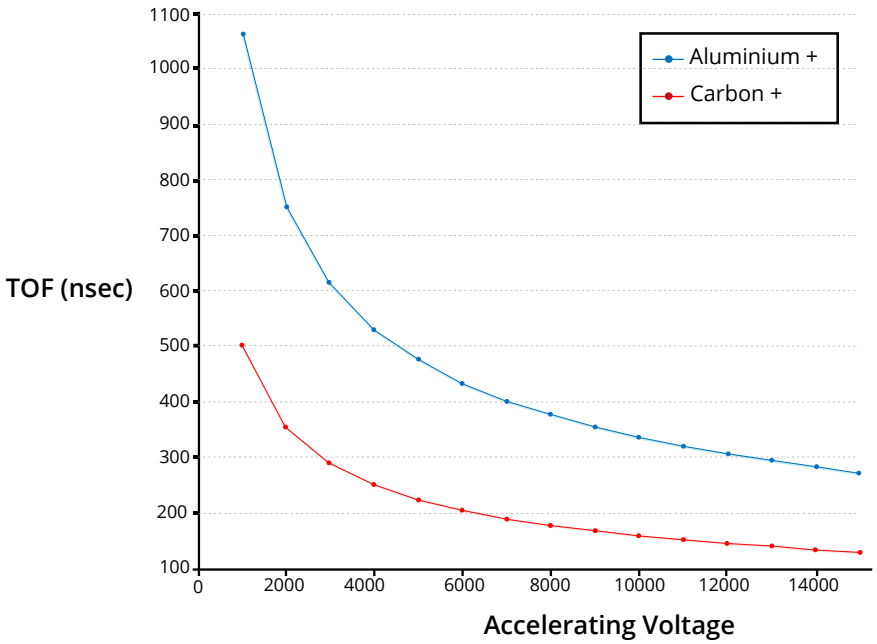
Individual ions are identified by measuring their time of flight (the time it takes then to reach the detector). The data collected is a collection of flight times, i.e. the raw time of flight (TOF) data.



Before proceeding to convert time-of-flight distribution to a mass-to-charge spectrum, two important corrections must be applied to the raw TOF data:

Voltage correction

As the radius of the tip increases, the voltage must also increase to generate the field necessary to maintain the evaporation rate. As the total applied voltage changes, so does the resulting kinetic energy of the ions. Hence an ion's time-of-flight to the detector changes over the length of the experiment.
A correction is required that removes the voltage dependence from the TOF.



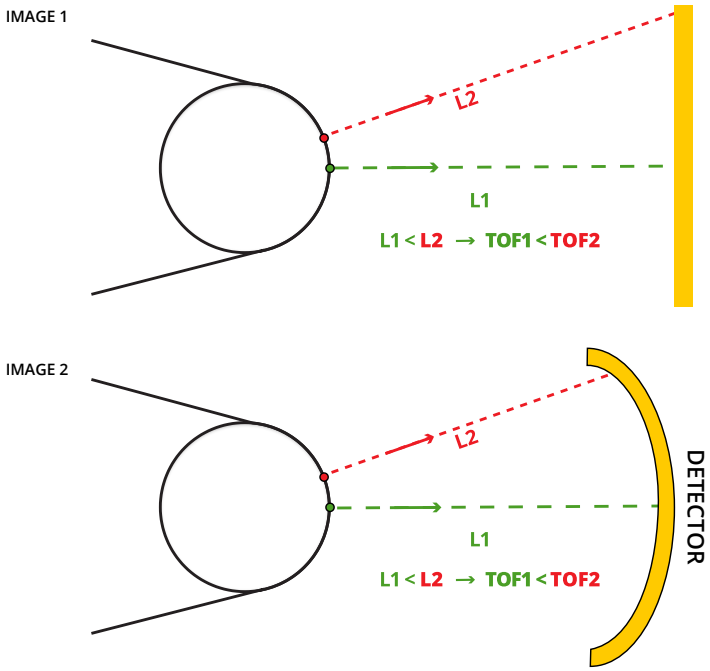
$$m/n = 2eVt^2/d^2$$

$$t = d\sqrt{m/n}\,/\,\sqrt{2eV}$$

$$t_{TOF} = \frac{a_0}{\sqrt{a_1 + V + a_2 V^2}}$$

Bowl correction

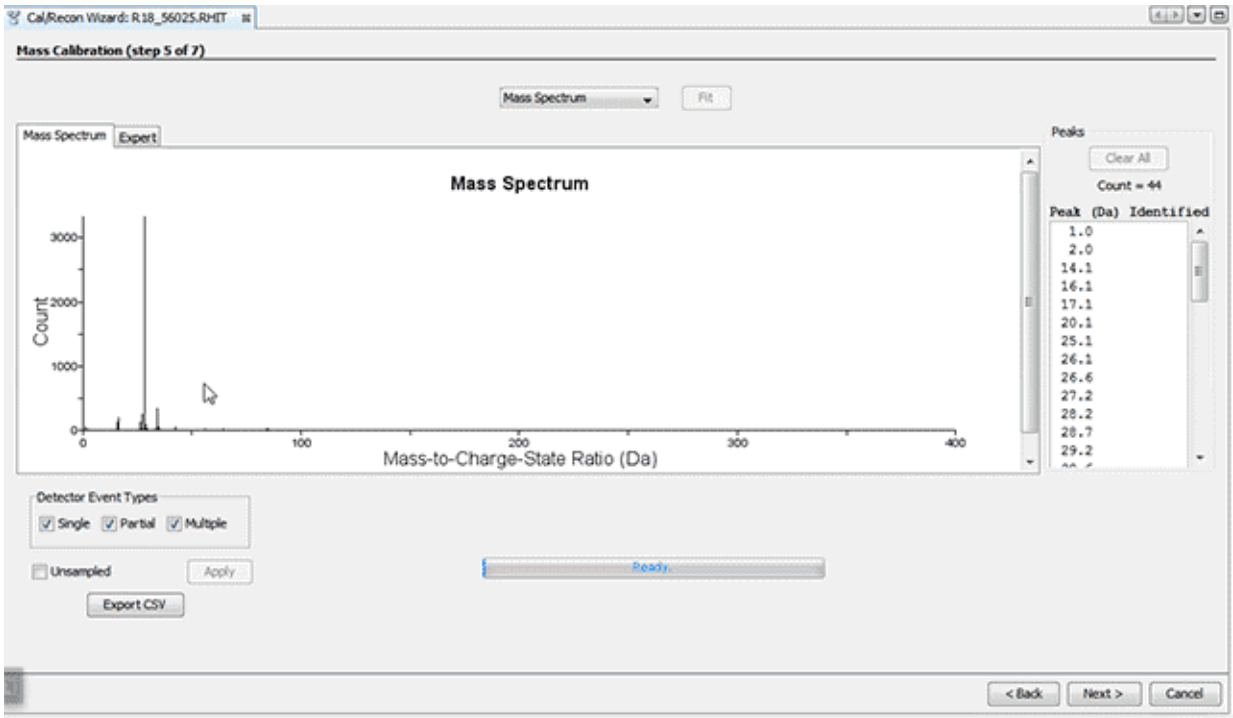
The path length from the tip to different parts of the detector varies significantly enough to affect the time of flights. A so-called 'bowl' correction takes into account flight path length differences as a function of detector hit location. 'Bowl' refers to the shape of the detector that would be required to normalise the flight path.



"Bowl" refers to the shape of the detector that would normalise the flight path.

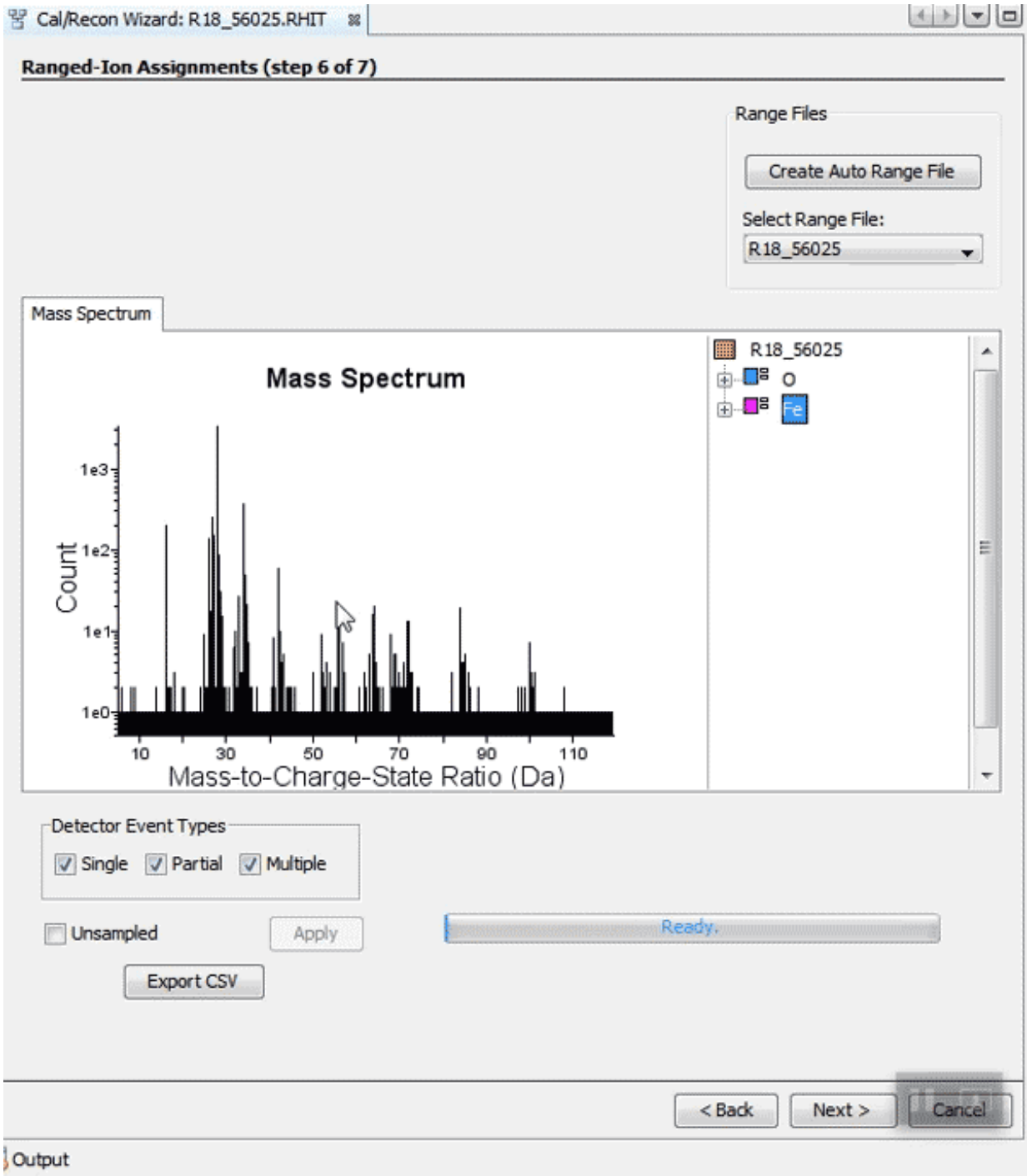
Mass calibration

Once the voltage and bowl corrections have been satisfactorily completed, conversion from TOF to mass-to-charge is performed (linearisation method is the default strategy).
Known peaks are used for this final calibration of the mass-to-charge spectrum. Selecting several known peaks at different mass-to-charge values leads to a more accurate mass calibration.



Ranged-ion assignment

Once the mass-to-charge spectrum is calibrated, each peak can be assigned an ion or molecular ion. This step can be arduous if unknown ions or direct peak overlaps are present.

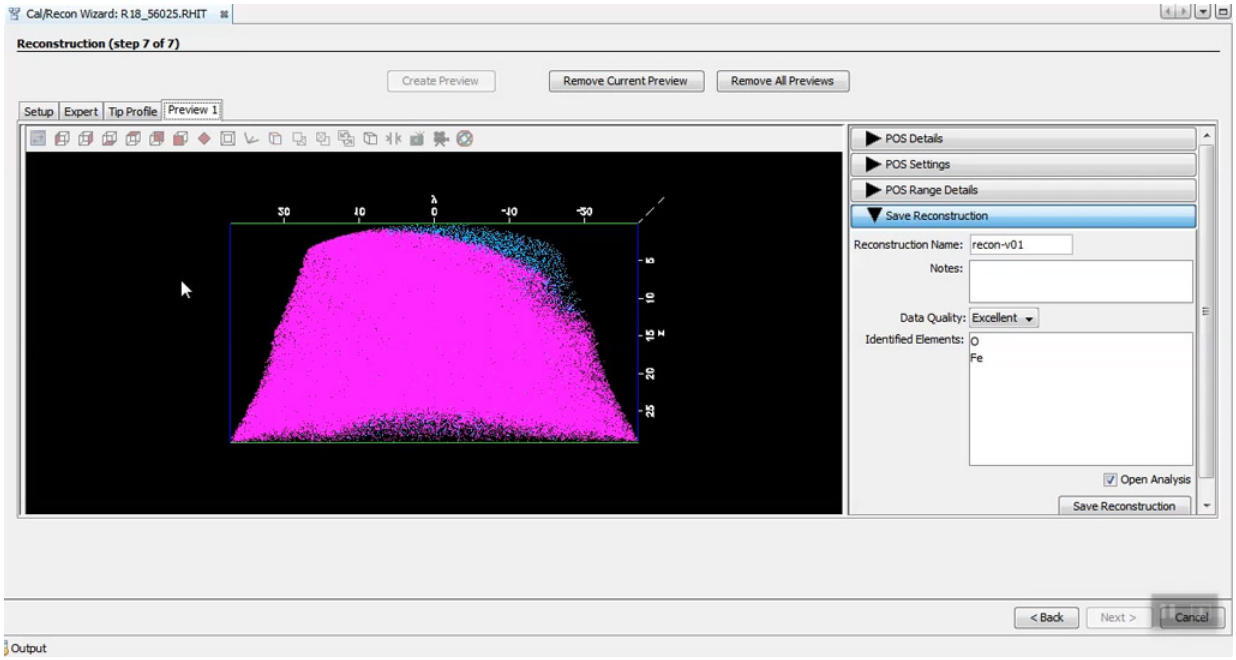


Reconstruction

In the course of an atom probe analysis, hundreds of millions of ions are potentially collected. At the end of the experiment, this data is in the form of:

- Sequence of evaporation
- Voltage required to evaporate each atom
- 2D detector hits (x and y positions of each ion on the detector)
- Raw time of flights

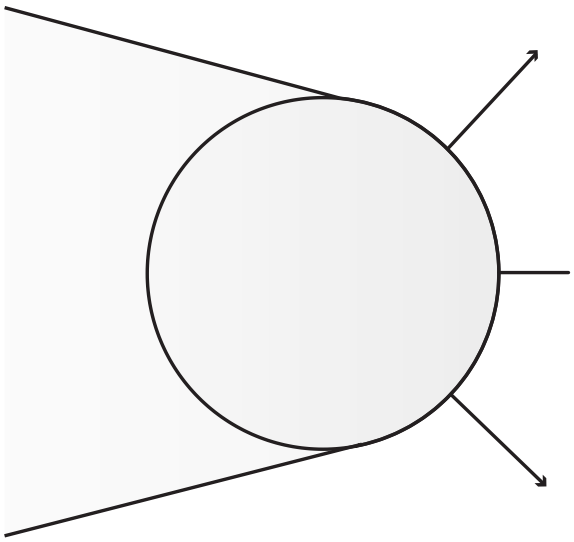
Reconstruction is the process of turning this information into a chemically and spatially resolved 3D rendering of the atoms in the original specimen.
In this last step, the user is asked to input several important parameters (or use the default values) that will be used to perform the final 3D reconstruction.



Key reconstruction parameters

Field factor k_f

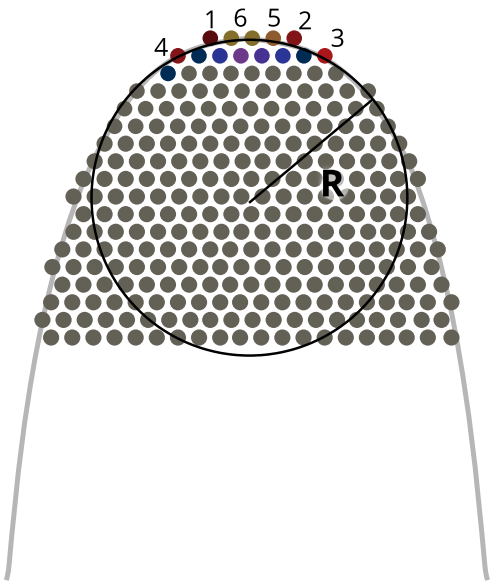
In atom probe, it is assumed that the tip takes the form of a hemisphere of radius R upon a cone. In this case:
$F = V/k_f \times R$
where F is the field, V the voltage, R the radius and k_f the field factor.



The field factor accounts for the combined electrostatic effects due to both the instrument and the shape of the specimen. The value of k_f is not known and is unique to each experiment, however, it can be inferred via experimental information and is typically between 2 and 5. Knowledge of k_f enables an accurate estimation of the tip radius, which is critical for the reconstruction procedure.

Tip radius evolution

The voltage is increased until it reaches the desired rate of evaporation (ions/pulse). As the radius of the tip increases, the voltage must be gradually increased to generate the field necessary to maintain the evaporation rate.
The evolution of the tip radius is constrained by the shank angle α.

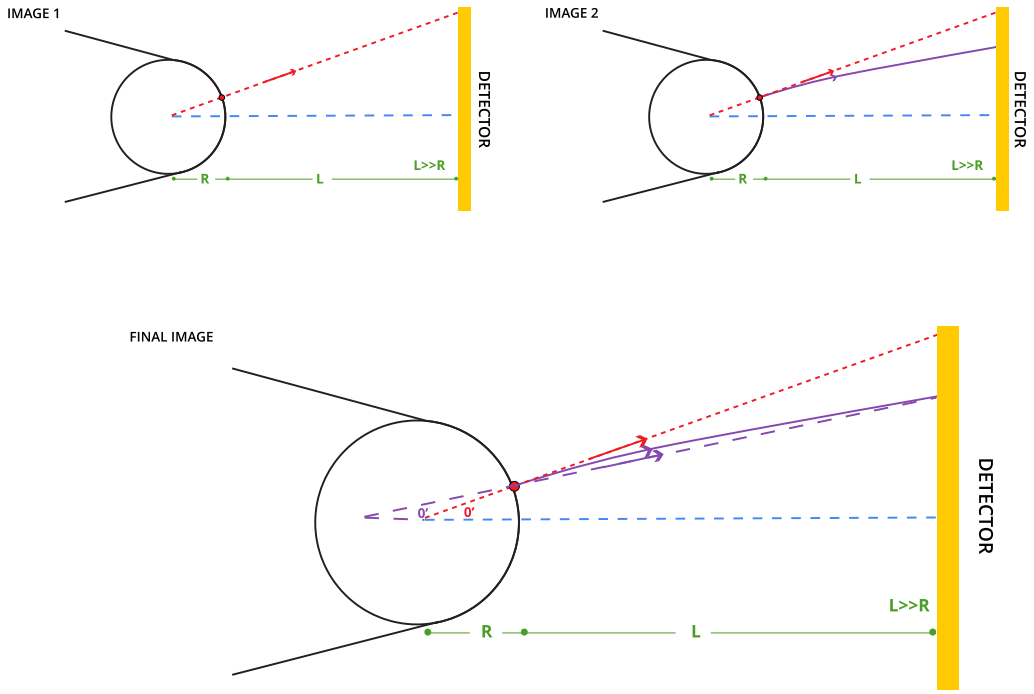


$$z_j = \sum_{i=1}^{j} dz_i$$

Atoms are sequentially evaporated from the specimen

Image compression factor

It is a simplification to assume that the field only leads to evaporation of atoms from the spherical part of the specimen in a direction tangential to the surface. In fact, the field is affected by the local environment (instrumentation and the specimen itself), which effectively focuses the ion's trajectory towards the centre of the detector.
This effect is described by ξ , the Image Compression Factor (ICF):
$\xi = \theta / \theta'$
where θ is the observed angle between 2 crystallographic planes and θ' the theoretical angle between these 2 crystallographic planes. ξ is experiment-dependent and typically $1 < \xi < 2$



Magnification

The magnification M is described by:
$M = L / \xi R$
where L is the distance between the specimen and the detector, ξ the ICF and R the tip radius.

Lateral coordinates

The lateral position of each atom (x_d,y_d) in the original specimen is described by the following equations:
$x_d = X_d \xi R / L$ and $y_d = Y_d \xi R / L$
where (X_d and Y_d) are the lateral position of each ion hit on the detector, ξ the ICF, R the tip radius and L the distance between the tip and the detector.

In-depth (z) coordinates

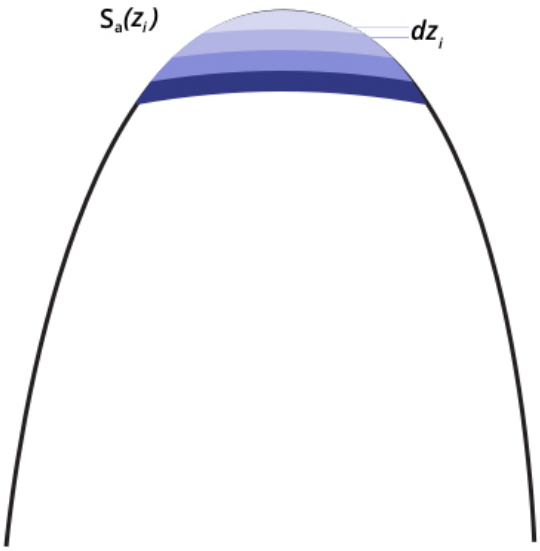
Reconstruction of each atom's z-coordinate is based on the sequence of ion detection in the course of the experiment. Each time a new atom is added to the reconstruction, the position of the model surface is adjusted downward incrementally, assuming the volume of the atom was equal across the tip surface.
In the first approximation, the z-coordinate of the j^{th} atom detected is simply assigned to be:

$$z_j = \sum_{i=1}^{j} dz_i$$

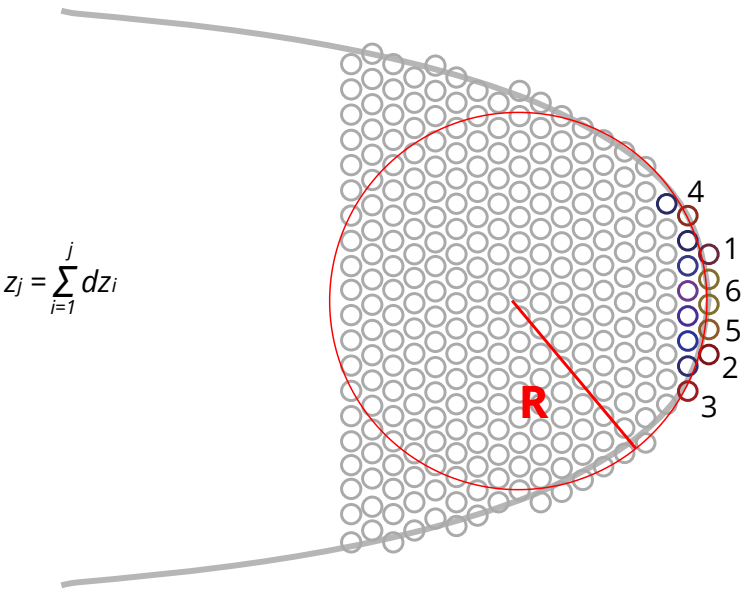
dz_i is based on the volume of the atom (Ω) and its contribution to the total volume of the entire reconstruction (V):

$$V = \sum_{i=1}^{n} \frac{\Omega_i}{\eta} = \int_0^{z_{max}} S_a(z)\, dz$$

where each ion represents a thin shell of volume at the surface of the specimen instead of a discrete local chunk. η is the detection efficiency.

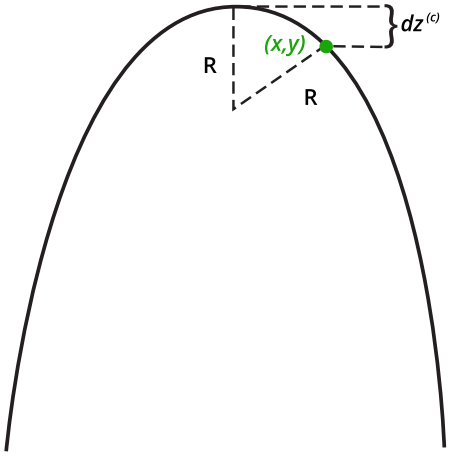


Atoms are then sequentially evaporated from the specimen



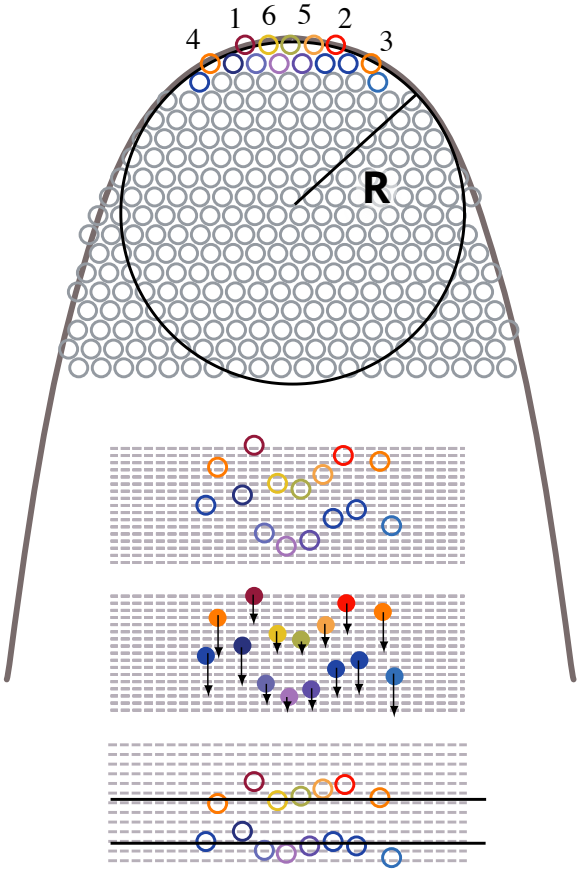
In a second step a correction is made to the z-coordinate to account for the fact that the positions of two atoms on the surface of the specimen may differ significantly in depth due to the high radius of curvature of the tip:

$$dz^c = R\left(1 - \sqrt{1 - \frac{x^2 + y^2}{R^2}}\right)$$



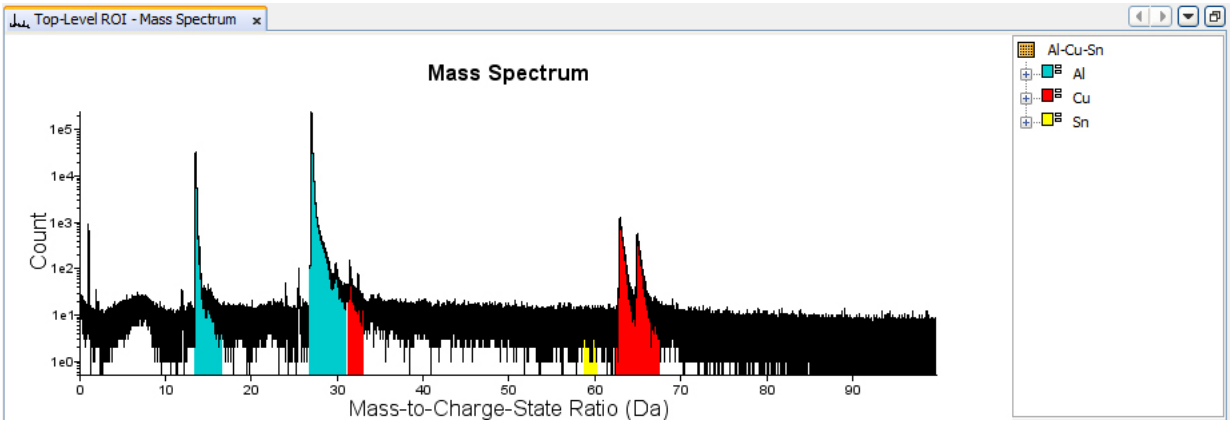
As a result, the in-depth coordinate of each atom is derived from:

$$z_n = \sum_{j=1}^{n} dz_j + dz^c{}_n$$



How do I analyse APT data - Mass spectral analysis

Mass spectral analysis is required to interpret the mass-to-charge ratio values for each atom. For this, every peak in the mass spectrum has to be assigned an element or a molecular ion, and this is referred to as ranging. This process is usually done manually during the reconstruction steps, but can also be done once a 3D reconstruction has been obtained. It is important to range every peak, as the ranges are used to determine the elemental identities of each ion within the reconstruction. The colours assigned when ranging peaks in the mass spectrum are also used to depict the ions in the reconstruction, so that segregation, precipitation and multiple phases are more easily identified.
To help identify unknown peaks, the peak position of single ions and complex ions can be displayed within the mass spectrum.
In some instances it can be advantageous to extract the mass spectrum of a feature of interest. Once extracted, the local and global mass spectrum can be compared to determine if there is any chemical variation between the two. To be able to do this, the area of interest has to be defined or extracted.



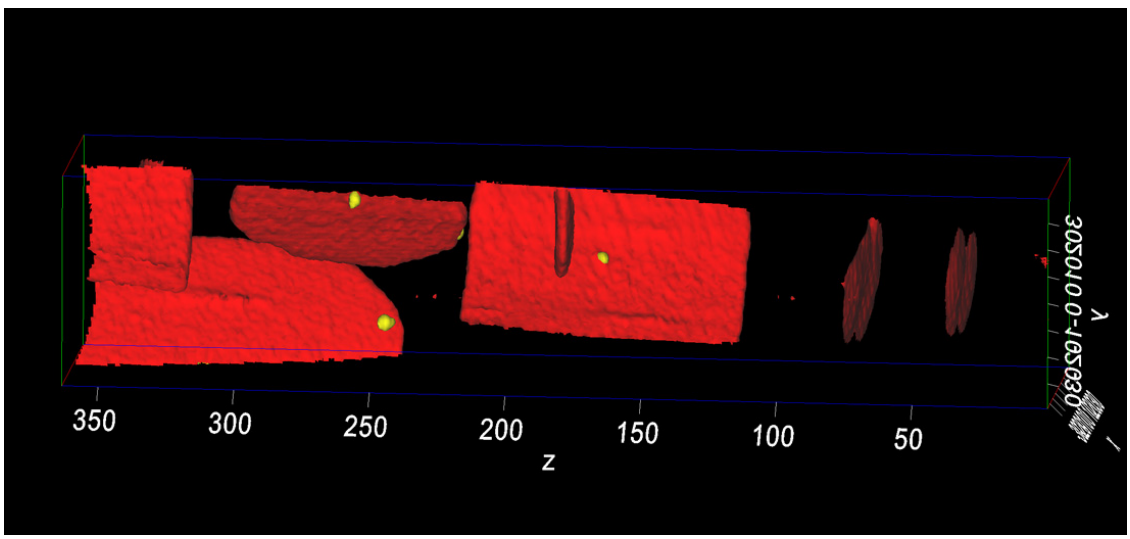
Mass spectrum with ranges.

Concentration space analysis - Grid voxelisation

A simple and quick method for analysing a 3D spatial volume filled with atoms, is by analysing the concentration of each ion species in a given volume. Instead of analysing each atom individually, the volume is seperated into 3D voxels (rectangular boxes), and the concentration of all atoms falling within each voxel is calculated.

Interfaces

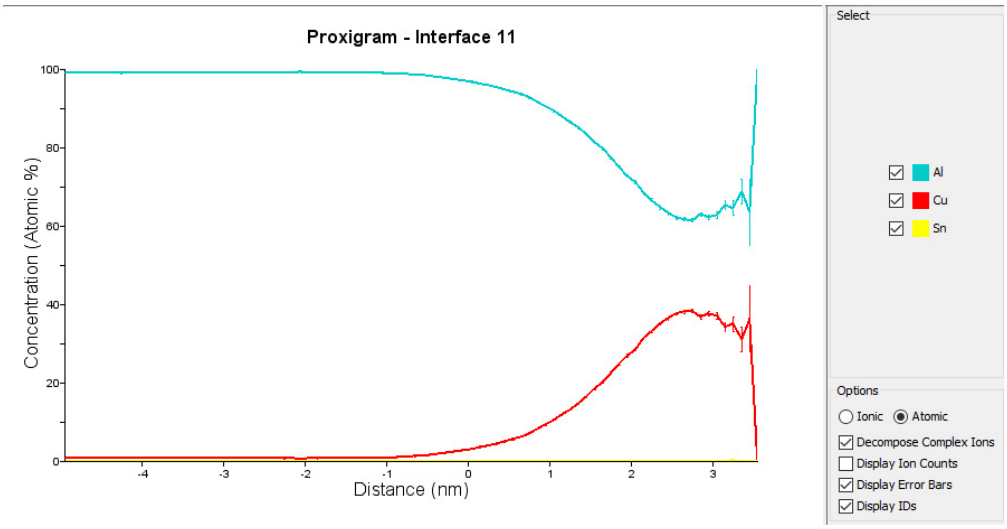
Once the concentration is determined, iso-concentration surfaces can be produced. Interfaces can also be visualised using density variations, instead of concentration differences. The two types of iso surfaces are referred to as iso-concentation surfaces and iso-density surfaces.



Isoconcentration surface.

Proxigram analysis

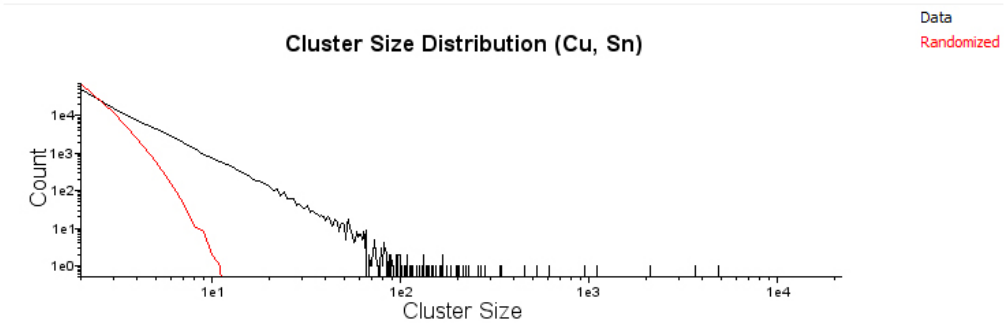
Creating isosurfaces enables the production of proximity histograms, or proxigrams. Proxigrams are a graphical representation of the number (or the concentration) of atoms that occur at a certain distance from a defined interface.



Proxigram.

Solute analysis / Clustering

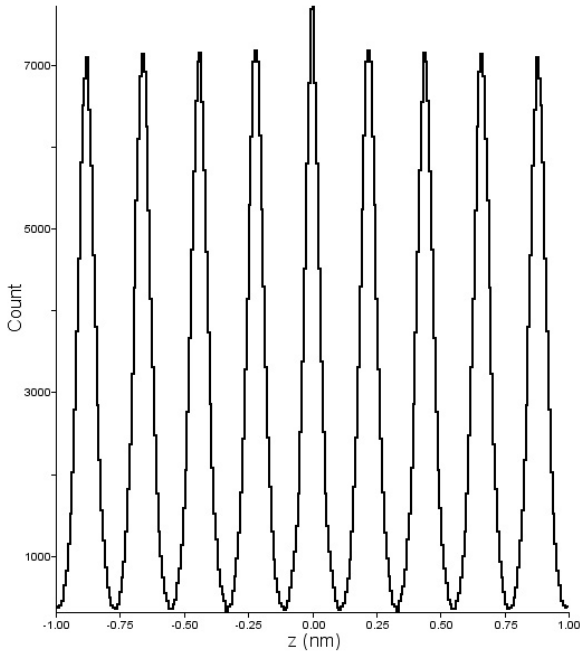
Solute analysis can be used to determine if clusters are present within the dataset, calculating their distribution and size. Cluster analysis tools provide atomic-scale analysis, treating the distance between atoms as a factor for choosing whether those atoms are in the same cluster. The first step is solute clustering, where the chosen d_{max} is used for classifying solute atoms. Once the solute atoms have been classified into individual clusters, the rest of the atoms are considered in enveloping and erosion algorithms.
Other, more complex solute analysis functions are the radial distribution function, the frequency distribution anlaysis and nearest-neighbour distribution. Many different approaches to solute analysis are available, and the reader is referred to the scientific literature for more information.



Cluster size histogram.

Spatial distribution maps

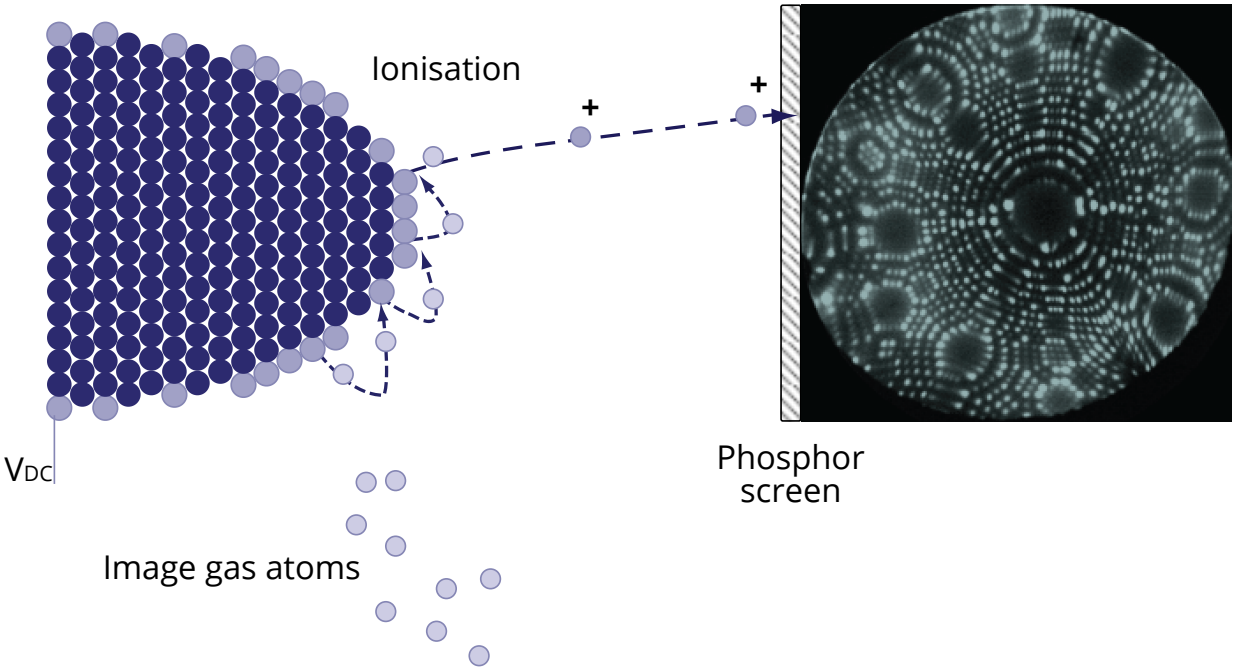
Spatial distribution mapping (SDM) is another atomic-scale analysis technique. SDMs are calculated by creating a histogram, either in 1D for a particular direction vector, or in 2D for a particular plane. SDMs are the clearest evidence we have that APT can resolve atomic planes. They can also be used to calibrate a reconstruction, by measuring the interplanar spacings and angles, and calibrating the ICF and k_f parameters to create a more accurate reconstruction.



SDM.

Specialised APT techniques - Field Ion Microscopy

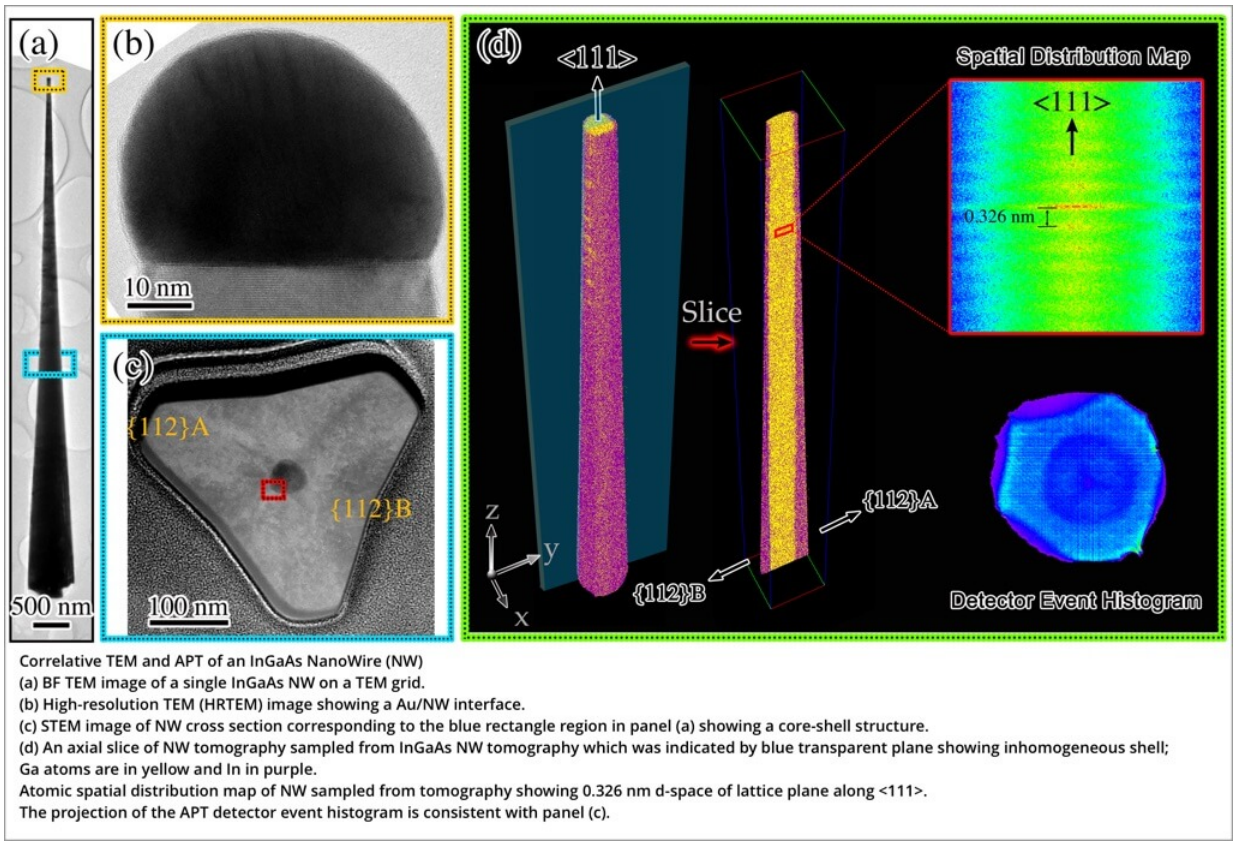
Field ion microscopy (FIM) is the precursor to APT. In FIM, an intense electric field induces ionisation of rare gas atoms (imaging gas) in the vicinity of a needle shaped specimen. Beams of ions are generated near the sample surface and projected towards a screen (phosphorous or digital) forming an image of the topography of the sample surface at the atomic scale.
In the LEAP, FIM experiments can be digitally captured.
FIM can be useful as it provides complementary structural information about the specimen. FIM has higher spatial resolution than APT due to the fact that gas ion trajectories are less influenced by local bonding and local changes to the radius of curvature, so the relationship and size of small phases or precipitates may be more precisely imaged.



FIM schematics.

Correlative SEM / APT

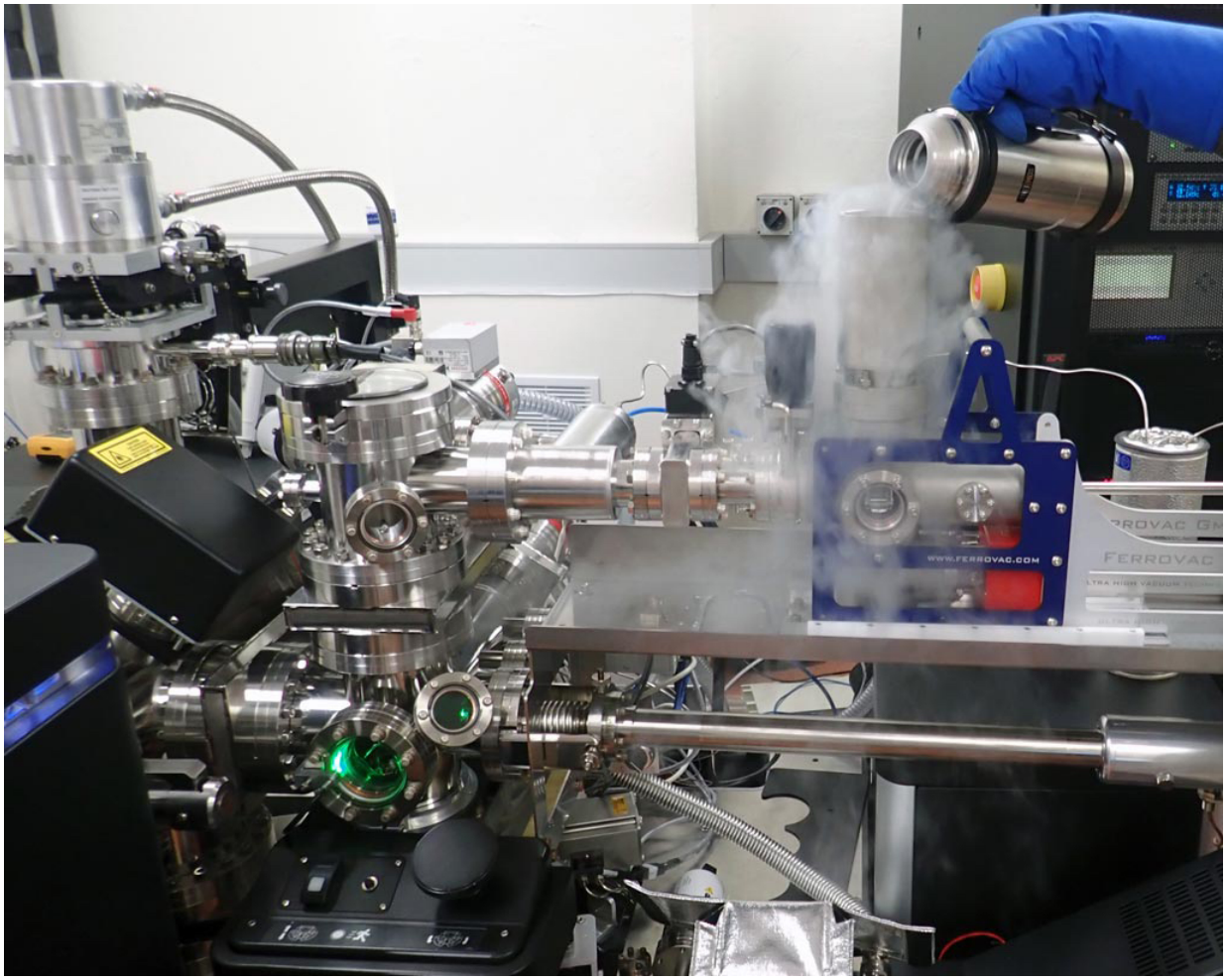
APT sample preparation in a FIB can be a difficult process when small features of interest such as grain boundaries or dislocations need to be within ~ 100 nm of the tip apex. Transmission Kikuchi Diffraction (TKD) is a technique that uses diffraction information from an electron beam to map the orientation of crystals within a thin sample, and can be performed simultaneously with APT sample preparation to locate and position such small features within the APT tip. The use of TKD during APT sample preparation not only facilitates site-specific preparation but also allows the crystallographic characterisation of features such as grain boundaries, dislocations or phases in an APT tip before field evaporation.
Transmission electron microscopy (TEM) and APT provide complementary information enabling more complete characterisation of the microstructure and chemistry of a material.
TEM imaging can provide specimen radius and shank angle with high accuracy, which improves the 3D-volume reconstruction accuracy.
It can also provide information on the internal structure of interfaces and precipitates.
Analytical techniques such as energy-dispersive X-ray spectroscopy and electron energy loss spectroscopy, as well as different imaging modes in scanning TEM, can provide information about the composition of precipitates and interfaces.



Correlative TEM and APT of an InGaAs NanoWire (NW)
(a) BF TEM image of a single InGaAs NW on a TEM grid.
(b) High-resolution TEM (HRTEM) image showing a Au/NW interface.
(c) STEM image of NW cross section corresponding to the blue rectangle region in panel (a) showing a core-shell structure.
(d) An axial slice of NW tomography sampled from InGaAs NW tomography which was indicated by blue transparent plane showing inhomogeneous shell; Ga atoms are in yellow and In in purple.
Atomic spatial distribution map of NW sampled from tomography showing 0.326 nm d-space of lattice plane along <111>.
The projection of the APT detector event histogram is consistent with panel (c).

Cryogenic transfer capabilities

Recent developments in APT technology have enabled the transfer of cryogenically frozen samples into the APT analysis chamber. Sample preparation can be done at cryogenic temperatures under controlled atmospheres in a 'glove box'. Then, the sample can be transferred while at cryogenic temperature and under vacuum (or controlled atmosphere) to the FIB where a cryogenic stage can be attached allowing FIB specimen preparation of the cryogenically frozen material. The APT sample can then be transferred to the APT analysis chamber while maintaining cryogenic conditions. This new technology potentially enables the analysis of soft, liquid or dynamic materials. A combination of deuterium charging and cryogenic transfer also provides the potential for the examination of hydrogen.



An APT Cryo transfer system.

Credits

Microscopy Australia acknowledges the huge input of time and expertise by the many staff members and associates who have contributed to the development of MyScope over the years.

For the APT module we thank: Alex La Fontaine, Ingrid McCarroll, Katja Eder, Anna Ceguerra and Julie Cairney.