Atom Probes LEAP Ahead

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The role of microscopy in human development

Since early times, the collective understanding of our microscopic universe has been directly tied to the quality of our microscopes. This has been true from the advent of light microscopes through to modern electron microscopes. Indeed, if one is to work on a given scale, one must be able to "see" at that scale. At the beginning of the 21st century, human inquiry is focused on the atomic scale. Though electron and scanning probe microscopes provide us with image information at the atomic scale, they do not deliver analytical information at nearly the same scale. Furthermore, as the length scale of features in materials reaches the atomic dimensions, the three-dimensional character of those features becomes very important. The atom probe is the only technique that provides compositional information at the atomic scale in a three dimensional image. As described below, recent technological development of the atom probe has positioned it to become a widely adopted technique that will allow it to make significant contributions to human endeavors.

What is an atom probe?

The term "atom probe" is often misunderstood to be associated with atomic force microscopy. The atom probe is not a type of atomic force or scanned probe microscope; its principle of operation is quite distinct.

Atom probes have been around since the late 1960s and many people are familiar with the basic concepts of these instruments. Today's atom probes far exceed the performance and applicability of the early instruments. In this article, a brief background on atom probes will be given, followed by an overview of some recent applications and a look to where atom probes are headed.

If we consider what properties of analytical instruments are important for the nanoscale, we might arrive at Table 1. The only techniques that can image individual atoms in technologically significant materials are the scanning tunneling microscope (STM) and the atom probe. Unfortunately, the STM is not capable of providing compositional information. The three-dimensional atom probe (3DAP) is the only technique that combines individual atom sensitivity and atom identification in an inherently three-dimensional image. Analytical information is most useful when it is coupled with the microstructure of a material, and several of the techniques excel in this regard. With today's atom probe technology, 100 nm length scales are accessible so that microstructural information from that size to the atomic scale is obtained. Finally, the atom probe has historically been a relatively slow technique: specimen preparation and data collection could take days. With recent developments in both of these areas, the time-to-knowledge for an atom probe has been reduced to about 1 hour. These advances have been packaged in a new commercial instrument called a Local Electrode Atom Probe (LEAP™) by Imago Scientific Instruments.

Table 1: Key performance parameters of microscopies for the nanoscale.

	Individual Atoms	Composition Analysis	3D	Determine Microstructure	Time to Knowledge
SIMS	No	Yes	~	No	1/2 day
AES	No	Yes	No	No	1/2 day
SEM	No	Yes	No	Yes	1 hour
TEM	~	Yes	~	Yes	1/2 day
AFM	No	No	No	Yes	1 hour
STM	Yes	No	No	Yes	1/2 day
LEAP	Yes	Yes	Yes	Yes	1 hour

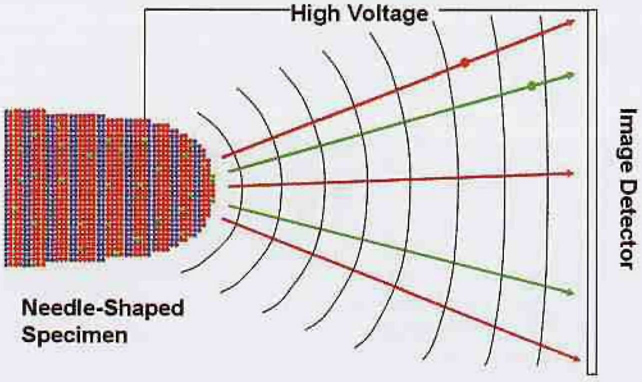


Figure 1, Schematic illustration of basic atom probe operation.

History of the Atom Probe

Atom probes have their foundation in the work of Erwin Müller on the field emission microscope (FEM) in the 1930s [1] and the field ion microscope (FIM) in the 1940s [1]. His work was conducted first in Berlin at the Fritz Haber Institute and later Müller moved his lab to the Pennsylvania State University. FIM was the first technique with which humans obtained images of individual atoms on October 11, 1955. After years of imaging atoms on a surface in a FIM image, the Müller group turned their attention to identifying the atoms that were being evaporated from the surface of FIM specimens. In 1968,

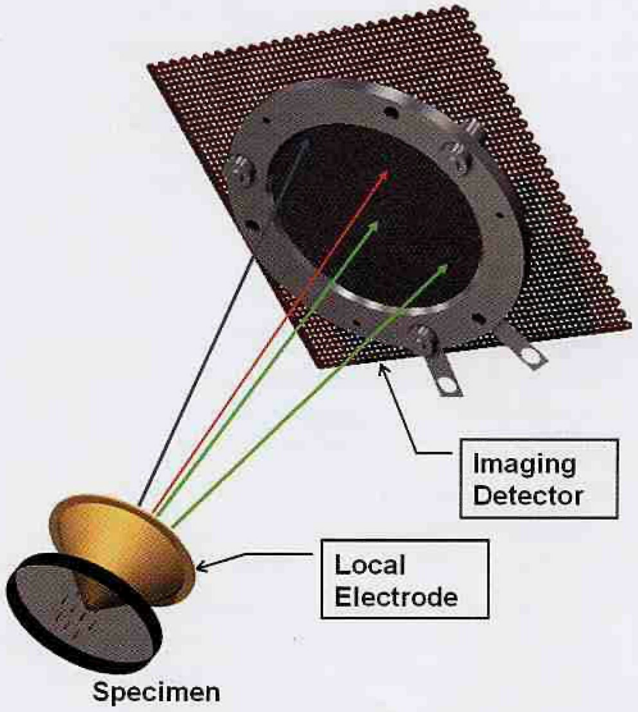
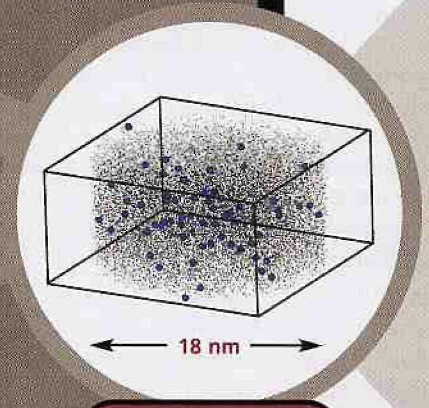
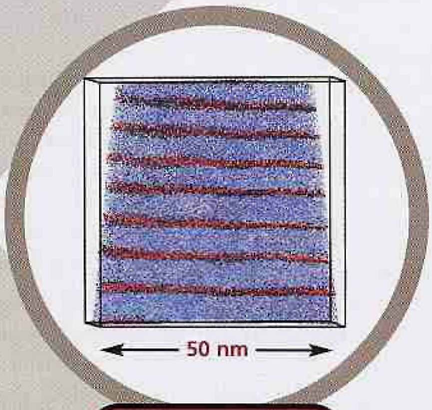


Figure 2, Schematic illustration of a scanning atom probe/local electrode atom probe geometry.



Boron in silicon



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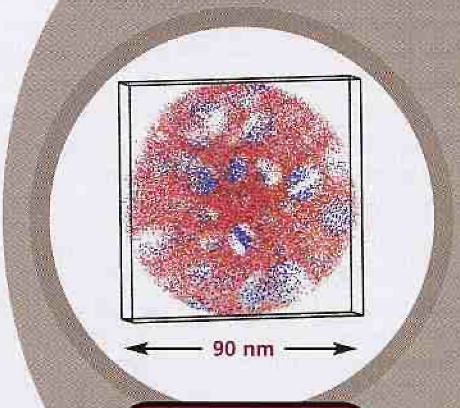

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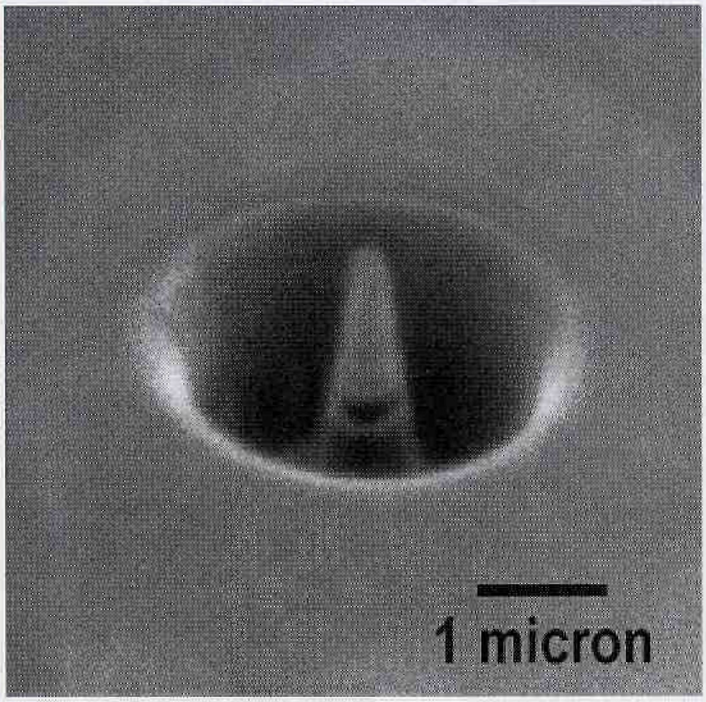

Figure 3, SEM image of a microtip fabricated in about 10 minutes from a multilayer structure on silicon using a focused ion beam instrument.

the first work on atom probe was published by Müller, Panitz and McLane [2]. The term "atom probe" was coined by Müller's group in this first paper. The technique was initially a one dimensional mass spectrometry method capable of analyzing several atoms per minute.

How an Atom Probe Works

The basic operation is shown schematically in Figure 1. A high electric field is created on a surface of a material by applying a high voltage between a sharp needle and an opposing electrode (imaging detector). If the needle is about 100 nm radius at the apex, about 10 kilovolts is needed to create a sufficient field to pull atoms off. This process is known as field evaporation. The basic geometry is that used in cold field emission electron sources, but the field is reversed to pull positive charge off the surface in the form of ionized atoms.

The atoms (ions) that are field evaporated follow the electric field lines out to the image detector. Originally, this image detector recorded only the arrival time of the ions. By pulsing the evaporation event, the time of departure is well known and the total time of flight of the ion from the specimen to the image detector is measured. The pulse magnitude is carefully controlled such that atoms are removed one at a time. In a constant electric field, this time of flight is proportional to the mass of the ion and is used to identify it. Sequential evaporation of atoms makes it possible to analyze the composition of the needle-shaped specimen as a function of the evaporation sequence. The first atom probes were thus one-dimensional (sequence) time-of-flight mass spectrometers.

The atom probe is a projection microscope in which the atoms on the surface of the specimen are projected onto the image detector. Several early efforts were made to record the position information for the evaporated atoms [3,4] and thus obtain three-dimensional information. By the late 1980s, the image detector had successfully been configured to record both the arrival time and the arrival position on the two-dimensional detector [5]. The two-dimensional hit position coupled with the one-dimensional sequence of evaporation events made the atom probe a three-dimensional imaging technique. As the analysis progresses, the entire surface layer of atoms on the specimen is removed, exposing the underlying layer. The process is continued over thousands of atomic layers. This approach achieves very high spatial resolution (better than 0.5 nm) and high magnification (10^6x) by projection. It is important to note that today's best imaging detectors use microchannel plate amplifiers which detect about 60% of all atoms with equal probability. It would be very desirable to realize 100% detection efficency, so that we can see every atom in a material, but no technology has yet appeared to do so. Miller has written a reference book on the atom probe technique for non-experts [6].

The atom probe was constrained to work with long needles by the requirements of creating high electric fields on the end of the needle. Nishikawa [7] provided a key insight into solving this limitation in his concept of a scanning atom probe in 1993. In this configuration, Figure 2, the counter electrode is an aperture placed very close (within several microns) to the specimen. The aperture is mounted at the end of a funnel so that it may be used to approach flat specimens. This nearby electrode, or local electrode, makes it possible to apply the high electric fields to needles that are only about one aperture diameter tall. For 20 micron diameter apertures, these "microtips" can be 20 micron tall on a planar surface. Thus, the need for millimeter long needles is removed, and one can think about making microtips on planar substrates from a variety of materials and methods. An example of a microtip fabricated with a focused ion beam instrument from a specific location in a multilayer structure on a wafer is shown in Figure 3. In practice, the moat around the tip would be larger but the quality of the tip is clearly visible. Nishikawa's insight has proven to be a boon to the potential of the atom probe to realize its great promise.

The local electrode also makes it possible to improve several performance characteristics of the 3DAP that have constrained its applicability. Kelly, Larson, *et al.* explored and identified these advantages in a series of papers [8, 9]. Imago Scientific Instruments Corporation was founded to develop this type of instrument to realize its true commercial potential. Table 2 shows a comparison of the key performance parameters of conventional atom probes with those of the LEAP.

The ability to achieve high mass resolution and a large field of view simultaneously has not been available in 3DAP prior to LEAP. Mass resolution is important for separating closely spaced mass peaks in common materials analysis and also for achieving high sensitivity. A value of 1 part in 300 is considered the minimum acceptable while 1 part in 500 is typical of quality instruments today.

The advantage of the LEAP technology is that the field of view is very large (50x greater) compared to conventional 3DAPs. This makes it possible to analyze much larger features in the atom probe than has ever been possible. Of course, if the number of atoms per unit area is 50x larger, the data sets and data collection time will be that much larger. Fortunately, this is another of the LEAP's attributes; it collects data some 500 times faster than conventional atom probes. Data collection for typical images of 5 million atoms takes minutes rather than days.

Speed is one of the most important characteristics for practical use of the technique in industrial applications. However, the advantage of rapid analysis can be wasted if specimen preparation remains time consuming. Fortunately, microtip preparation for the LEAP greatly speeds up specimen preparation. Literally hundreds of specimens can be analyzed on a single wafer, allowing multiple samples to be tested without the downtime associated with specimen exchange. So we are back to Nishikawa's important contribution that makes this all possible.

Table 2 Comparison of Typical Performance of Conventional 3DAPs with LEAP

	Mass Resolution (FWHM)	Data Collection Rate (atoms/min.)	Field of View (steradian)	Field of View, typical (nm)	Work with microtips
3DAP	500	2 x 10^3	0.03	15	No
LEAP	600	10^6	1.5	100	Yes

What Are Atom Probes Used For?

Atom probe analysis can legitimately claim to offer information that is a step beyond what is possible in an analytical electron microscope. For image resolution, the TEM is clearly superior. However, with regard to analytical image resolution, the atom probe has no peer. This has been true since the advent of the atom probe. The world of analytical techniques can be viewed on a map that plots analytical sensitivity versus lateral spatial resolution, Figure 4. The atom probe has always occupied the position furthest to the left, that of the highest resolution technique. The one-dimensional atom probe, designated APFIM in Figure 4, accessed very small volumes of material and did not cover much of the map. The LEAP is now able to greatly expand that range of applicability to much greater length scales and higher sensitivity as shown in Figure 4.

Buried or internal interfaces are a common feature of materials and devices, yet they are particularly difficult for anything but a three-dimensional technique to characterize well. Interface segregation, interface roughness, interface diffuseness, and layer thickness are all crucial characteristics of buried interfaces that are often difficult or impossible to analyze well in sections. Each of these characteristics can be observed in the following example. Several other examples have recently been described in an article to be published in an upcoming issue of Microscopy and Microanalysis [10].

Information stored on hard drives is read from a rotating disc by a sensor that relies on gigantic *magnetoresistance* (GMR) to detect the small binary-encoded permanent magnetic fields. The GMR sensor is made up of many very thin metal films that each performs a function critical to the operation of the sensor. The film thicknesses, interface roughness and diffuseness, and film composition all influence their performance, especially since each film may be only 2 nm thick or less. Impurities can play an important role, favorable or unfavorable, in the device. It is critical that manufacturers can see what is happening at the atomic scale as they develop the devices, develop the processing steps and tools, monitor the quality of the manufacturing process, and estimate the reliability of the structures.

Figure 5 shows a static cross-section of a LEAP image of a test structure that is used to evaluate the film thickness and compatibility of Pt-50at%Mn (10 nm thick) antiferromagnetic layers with Co-10 at% Fe (2 nm thick) ferromagnetic layers. There are many repeats of these two layers in the image. Figure 5a shows a section of a 3D LEAP image of this layered test structure. Each atom in the image is depicted as a dot of the chosen color for that specie. The PtMn layers are clearly visible and distinct from the Co layers. These films were deposited onto a prepared silicon wafer with 3 micron diameter by 100 micron tall posts etched into the top surface. This specimen was prepared using a focused ion beam instrument to sharpen the apex of a silicon post to create a tip ready for atom probe.

Figure 5a shows the Mn and Co in their respective layers. The thickness, interface roughness, and interface diffuseness can all be seen and quantitatively evaluated in this image. In Figure 5b, the Ga distribution in the image is shown with the Mn layers. The Ga concentration exceeds 10 atomic percent at the apex but drops to less than one part per thousand after about 20 nm into the top surface. Figure 5b is a nice illustration of the use of the atom probe for studying ion implantation profiles in materials. Note also that the layers are mixed in the heavily damaged region near the apex. One surprise of this particular study is the presence of carbon in the structure. This impurity was not supposed to be present but was *found to constitute a level of approximately* 0.45!! atomic percent in the entire structure. Furthermore, as shown in Figure 5c, the carbon is segregated to the CoFe layers. This amount of carbon in the structure could play a significant role in the performance of the device. It is unlikely that this amount of carbon would ever be noticed in a TEM let alone analyzed successfully with this level of detail.

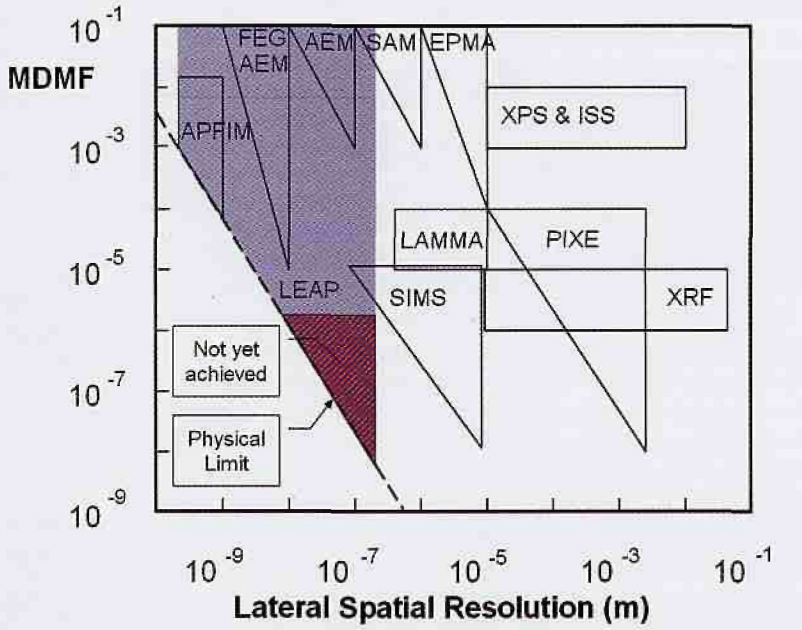

Figure 4, Map of analytical techniques depicting their ranges of applicability. The LEAP is shown in colored background. Adapted from a figure by C. E. Lyman.

Where are Atom Probes Headed?

There are many applications of the atom probe. A very large number of important applications have been demonstrated for metals of all sorts. Applications in the semiconductor industry are at an early stage, but will likely expand significantly in the next several years. At this time it is important that the material have sufficient electrical conductivity (<0.01 ohm-cm) to support voltage pulsing of the specimen though thin (<5 nm) dielectric films on good conductors can be run. Methods of pulsing poor electrical conductors are being developed and are expected to become available.

If atom probes are to be more widely adopted, and therefore more generally useful, it will be necessary to make good on the promise that specimen preparation will become simple and even automated. We are just at the early stage of this process; much

Figure 5, LEAP 3D image of a multilayer stack of PtMn layers with CoFe layers. a) Mn and Co atoms shown to depict the layers. b) Ga shown to depict the ion implantation from the focused ion beam. c) C shown to depict the impurity presence at low concentration and segregation to the CoFe layers. The Mn is shown in red, Co is shown in blue, Ga is shown in gold, and C is shown in green. The layer repeat distance is 12 nm. This image is from the work of Peter Ladwig and Austin Chang of the University of Wisconsin, David Larson of Seagate Recording Technologies and Imago personnel.

like transmission electron microscopy was in the 1960s. However, much of the progress made in transmission electron microscopy specimen preparation methods and tools since the 1960s is applicable to atom probe specimen preparation. Over the next few years many of these methods will be applied to atom probe specimen preparation for a wide range of materials. Indeed, we can expect to see the atom probe being applied to an increasingly broad range of materials including semiconductors, ceramics, and organics. The challenges for successful atom probe analysis are greater as the material complexity increases but the rewards are equally great. Every field of endeavor today from metallurgy to microelectronics and polymers to drug development could benefit from three-dimensional compositional images at the atomic scale.

While the utility of the atom probe to research and development for a wide spectrum of applications may be apparent, it is not as obvious that the atom probe may find application in quality assurance, failure analysis, and even metrology. The technique is destructive but only requires a few cubic micron of material. This amount can be extracted from most devices or wafers in microelectronic applications without sacrificing the entire device or wafer. Furthermore, the speed of specimen preparation and data collection is now sufficient in the LEAP atom probe to entertain these applications. Look for automated or semi-automated microtip specimen preparation instruments to appear for atom probes in the near future for these demanding applications.

Summary

Atom probe technology has taken a major leap forward with the advent of working LEAP atom probe microscopes. This development will benefit fundamental science since new questions can be answered with the expanded reach of the atom probe. It will also benefit technology because both development and manufacturing are increasingly dependent on information at the atomic scale for their basic operations. Although it is not yet a fully mature technology, the atom probe is on a new path that solves most of the earlier limitations and points to a future that is rich in discovery and benefit. ■

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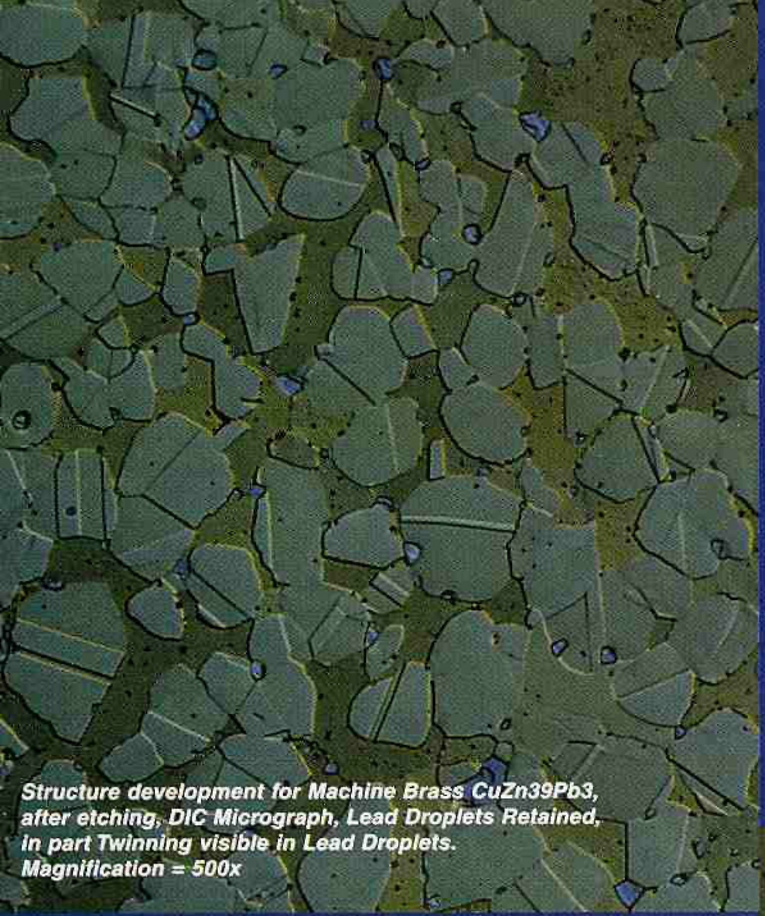

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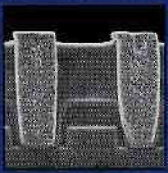

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