Local Electrode Atom Probes

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The historical developments leading to the advent of Local Electrode Atom Probes (LEAP) are reviewed. An assessment of the state of the art is made, and the major advantages of LEAPs over conventional atom probes are described. The best implementations of these concepts and the remaining challenges for realization of LEAP’s potential are also described. It is concluded that LEAPs should be an important tool for materials characterization at the atomic scale. Modern materials-dependent industries as diverse as steel and microelectronics should benefit from this technology. © Elsevier Science Inc., 2000. All rights reserved.

INTRODUCTION

Much of the current activity and excitement in materials science involves processing and understanding materials at the atomic scale. Accordingly, it is necessary for materials scientists to control and characterize materials at the atomic level to optimize a variety of materials systems from ferrous metallurgy to multilayer thin films. There are only a few microscopies that are capable of providing information about the structure of materials at the atomic level. The atom probe field ion microscope (APFIM), particularly the three-dimensional atom probe (3DAP), is the only instrument that determines the 3D location and elemental identity of atoms in a sample at the atomic scale.

WHO NEEDS LEAP?

As most of the articles in this volume attest, the atom probe is one of the most spectacular and uniquely capable analytical instruments ever developed. Given its extraordinary capabilities and a burgeoning need for this capability, it is curious that atom-probe technology has not enjoyed more widespread adoption. There are many ways to explain this but, in the authors’ opinion, it is a problem of applicability. If the results available from the atom probe were more generally useful, there would have been more eager customers for the instrument. Three-dimensional atom probes have greatly improved the situation, but the underlying problem remains. The tedium and constraints of sample preparation could be cited as the reason, though the situation is no worse than for transmission electron microscopy. Nonetheless, it is very true that easier and more powerful sample preparation methods are the key to application of either technique to a broader spectrum of specimen types. The problem then, as we see it, is twofold: today’s atom probes do not analyze very large volumes of material (limited to about $10^6$ atoms), and the specimen geometries that are readily accessible (principally needle-shaped specimens of electrically conducting material) limit the technique. As a result, atom probes can be applied only to those problems for which knowledge of the limited analysis volume is useful. Based on today’s capabilities, that set of problems is not large.
The Local Electrode Atom Probe (LEAP) has the potential to greatly expand the reach of the atom-probe technique to much larger volumes (e.g., $10^9$ atoms) and more general specimen geometries (e.g., planar structures, where good electrical conductivity is not a requirement). The arguments for this conclusion are many and are described in detail in this paper. The essential ideas are as follows. (1) With a local electrode mediating the extraction of ions from a particular tip (Fig. 1) the specimen may consist of many tips rather than a single tip. These tips may be formed on a planar surface. (2) With the extraction electrode close to the tip, much lower extraction voltages are required compared with conventional atom probes. Pulsing systems can achieve much higher repetition rates at lower voltages, and so the extraction process may achieve several orders of magnitude higher rates. (3) Because primary acceleration of ions occurs in a spatially and temporally small region, the spread in ion times of flight is small initially. With appropriate design, if the spread is kept small relative to the total time of flight, the mass resolution of the instrument can be excellent. (4) Because of the low extraction voltage in LEAP, secondary acceleration of the ions to higher final energies is one strategy that can be applied to achieve improvement in the mass resolution.

Taken together, these advantages add up to major improvement in the key operating parameters of atom probes. They can change the way that the instrument is perceived and utilized. Thus, LEAPs could greatly expand the reach and commercial viability of atom-probe technology.

**RECOGNITION OF THE NEED FOR THE TECHNOLOGY**

Recently, a U.S. National Science Foundation Panel Report on Atomic Resolution Microscopy was commissioned to access the opportunities in materials research utilizing atomic resolution microscopies [1]. This report states “The continuing miniaturization of electronic devices requires the invention of manufacturing tools and processes to operate on scales that are much below current micron-size technology . . . At length scales of 20 to 100 atoms, there is an urgent need for instruments with resolution at the atomic level and with a capability for chemical identification for processing and characterization.” This need is evident today, as several industries are developing products where subnanometer layers are utilized and must be understood. With regard to how atomic resolution microscopies are needed to bring about a revolution in materials processing, the report states, “The holy grail of semiconductor analysis is the ability to identify, anywhere in a crystal and with full three-dimensional atomic resolution, an impurity or dopant atom. Such analysis will require sub-Å resolution utilizing tomographic techniques.” This statement sounds like an endorsement of 3DAP, in general. A LEAP performing 3D analyses, as described in this paper, can result in a user-friendly instrument that delivers this needed capability.
HISTORICAL DEVELOPMENTS

THE SCANNING ATOM PROBE

As described in the following sections, there were several research efforts which, in retrospect, appear as natural precursors to the breakthrough that established the concept of the LEAP or scanning atom probe (SAP). Clearly, however, it was the work of Nishikawa, with the help of Kimoto, who first presented their work at the 1993 International Field Emission Symposium in Nagoya, Japan [2] that established the idea of using a positionable small extraction electrode relative to multitude of sharp points. Nishikawa reviews his work in this area separately in this volume [3].

By applying a high voltage between planar microtip specimen and a flat screen, the field at any one tip will not be high enough for field evaporation. In addition, control over which tip is evaporating could not be controlled even if the field could be made high enough. Nishikawa and Kimoto [2] suggested that a small aperture could be scanned over the surface at high potential until ions were extracted from one of the protrusions; hence, the name, “scanning atom probe.” They evaluated the electrode geometries needed to achieve the electrostatic fields required for field evaporation of the anode while avoiding field electron emission from the cathode. They suggested that the SAP might be used to analyze any type of (planar) specimen that had protrusions on its surface such as fracture surfaces and particulate materials. They also suggested that specimens of planar materials might be made by cutting many parallel grooves in each of two perpendicular directions on a surface.

Nishikawa continued development of the SAP, and in 1995, his group presented initial results concerning a needle-shaped specimen that had protrusions on its surface such as fracture surfaces and particulate materials. They also suggested that specimens of planar materials might be made by cutting many parallel grooves in each of two perpendicular directions on a surface.

Nishikawa continued development of the SAP, and in 1995, his group presented initial results concerning a needle-shaped specimen that was positioned relative to a flat circular aperture of 10μm diameter [4]. They showed that the field electron emission current could be used to align the tip relative to the aperture. They have performed one-dimensional atom-probe analyses of protrusions in diamond-like coatings [5, 6] and silicon emitter tips [7, 8]. The instrument uses a local aperture, and is configured as a one-dimensional atom probe. Thus far, the instrument operates at room temperature. The extracted ions are sent through a reflectron [9, 10] to improve the mass resolution of the instrument.

THE SCANNING ATOM PROBE AND THE LEAP

At that 1993 IFES meeting, one of the authors of this paper (T.F.K.) was particularly impressed by the SAP concept and the possibilities that this new specimen geometry might open. This engendered a lot of thinking about the ramifications of the small extraction electrode. T.F.K. was familiar with the work on field emitter arrays where small extraction apertures are patterned around arrays of sharp tips. It was well known at the time that the voltage needed to achieve field electron emission from field emitter arrays in much lower than the voltage needed for field electron emission from isolated needles. It is the proximity of the extraction electrode to the tip that localizes and enhances the field. The major advantage of the scanning atom probe is that it makes it possible to work with planar microtip specimens. However, the local extraction aperture makes performance improvements possible. Thus, the name “Local Electrode Atom Probe” is used to emphasize the instrumental technique where proximity effects play a major role in defining the instrument. Note that the advantages that result from a local electrode apply to all atom-probe specimen types including conventional needle-shaped specimens.

It is worth considering the development of the SAP/LEAP in an historical context. Several avenues of development have contributed to this technology, and specimen preparation is paramount. The following sections describe some of the most important contributions to specimen fabrication work for SAP/LEAP.

DEVELOPMENT OF MICROTIP SPECIMENS

To investigate a materials system by any characterization technique, whether it is...
transmission electron microscopy, scanning electron microscopy, field ion microscopy, etc., an absolute requirement is the ability to fabricate a good specimen. This is particularly important for field ion microscopy and atom probe microscopy, where the specimen must be in the form of a sharply pointed “tip” having a radius of curvature at the apex of about 10 to 50nm. Although the fabrication of a specimen from a metal wire is relatively straightforward and is generally performed by electrochemical polishing methods, preparing a field ion specimen from a material that does not electropolish well can be difficult or impossible. If a particular physical or crystallographic orientation of the specimen is required as well, the chances of success diminish further. This is especially true for the case where the specimen must contain thin films on a planar structure with tips normal to the surface. The planar specimen requirement has either limited or precluded the application of field ion and atom probe microscopy to a number of materials systems. As stated above, the main advantage of an atom probe employing a local electrode is the ability to analyze microtips on such a specimen.

The planar microtip geometry ideally consists of small cones formed from a flat substrate in a direction normal to the plane of the substrate (Fig. 2). Some of the advantages of this type of specimen include the capability to: (1) fabricate multiple specimens from a single small piece of material, (2) form specimens with tip axes oriented along a specific direction, (3) form tips at specific positions on the sample surface (at interfaces, precipitates, etc.), and (4) take advantage of methods not commonly used for specimen fabrication, such as ion beam techniques.

**Ion Sputtering of Surfaces**

Ion sputtering may be defined as the removal of material from a surface due to the impact of ions. Sputtering was reportedly first observed in a gas discharge as the removal of a cathode impacted by energetic ions from a plasma [11]. (For a brief historical overview of sputtering, see [12].) Ion sputtering has many applications, which may be divided into two general categories: (1) those that remove or expose material for the purpose of analysis, and (2) those that remove material with the goal of modifying the structure of the substrate. Examples of the former include secondary ion mass spectrometry, sputtered neutral mass spectrometry, Auger electron spectroscopy, X-ray photoelectron spectroscopy, low-energy ion scattering spectrometry, and Rutherford backscattering spectrometry. Examples of the latter include substrate cleaning, specimen preparation for various microscopy techniques, thin-film deposition, and various forms of lithography. The specific sputtering application that is of interest here is the structuring of a material to form cones from a surface for use as field ion specimens. It should be noted that the concept of fabricating “multipoint” emitters using ion beam techniques for use in field emission or field ion microscopy was proposed as early as 1981 by Auciello [13], who pointed out that ion bombardment-induced arrays of cones might
be used as an alternative field ion specimen geometry.

**Cone Formation**

It has been known for quite some time that it is possible to form “cones” on a surface by the use of ion sputtering under certain conditions (Fig. 3). Many reviews discussing this type of cone formation have been published including those by Auciello [12, 13], Witcomb [14], Navinsek [15], Hauffe [16], and Ghose and Karmohapatro [17]. If foreign particles are present on a surface during ion sputtering, these particles will mask the substrate from sputtering and cones will be formed at these locations. This process is known as ion beam mask etching (IBME). Sputter yield is known to depend on the angle of incidence and to go through a critical angle ($\theta_{\text{max}}$) somewhere between 60° and 80° [15], where $\theta$ is defined as the angle between the surface normal and ion beam incidence. At angles greater than $\theta_{\text{max}}$, the ions are reflected from the surface and do not contribute to sputtering [18]. Ions that are reflected from the surface of a forming cone will impact at the cone base and form a “moat”-type structure, as illustrated in Fig. 4. The critical angle for such ion reflection is given by [19] [Eq. (1)]:

$$\theta_{\text{max}} = \frac{(\pi/2)((5\pi\rho/\rho_{0})^{2/3}Z_1 Z_2 E_r / (Z_1 + Z_2 E_i))^{1/2}}{a_0}$$  (1)

where $a_0$ is the Bohr radius, $\rho$ is the substrate atom density, $E_i$ is the incident ion energy, $E_r$ is the Rydberg constant, and $Z_1$ and $Z_2$ are the atomic numbers of the ion and substrate, respectively. As material is eroded away from the substrate, a structure forms that exposes those faces having the maximum sputtering rate. A cone develops with its axis parallel to the direction of the incident ion beam, and has a shank angle given by [18] [Eq. (2)]:

$$\alpha = \pi - 2 \theta_{\text{max}}$$  (2)

Note that this analysis assumes a random polycrystalline orientation, that is, no crystallographic dependence (channeling is ignored). Polycrystalline surfaces that are sputtered will result in height contrast due to different sputtering rates from random grain orientations [20]. The size of the particle and the relative mill rates of the particle and substrate will determine the final length of the cone.

With respect to the formation of cones for use as a field ion specimen, it is of interest to examine a typical shank angle of such a structure formed by IBME. Using copper as an example substrate, and Ar ions of 4keV energy at normal incidence ($\theta = 0$), the equation given above predicts a shank angle of about 32°. This compares favorably to an experimentally measured angle of about 30° [21]. Although this angle is higher than that normally observed in field ion specimens (about 1 to 10°), it is expected that the use of a local electrode geometry for electric field production will minimize the detrimental effects of the larger shank angle [22]. In addition, smaller shank angles may be obtained using IBME with angles other than normal incidence and a rotating specimen (Fig. 5) [23, 24]. In general, milling conditions must be selected such that a final geometry suitable
for field evaporation is obtained. Ion sputtering of the mask must produce tips that have an apex radius of curvature of <100nm, be at least a few microns long, have a shank angle <45°, and be spaced such that the nearest neighbor tips do not interfere with the extraction electrode used for field evaporation.

**Mask Material and Positioning**

Particles of many types including polymer spheres [25], metallic spheres and ceramic
particles [26] can serve as masks for IBME. Diamond [21] works especially well for this application, due to its very low sputtering rate [Figs. 2 and 4(bottom)]. The type of mask material used will determine whether the mask can be placed on the sample in random positions, in arrayed positions, or in specified locations. Random particle masking is relatively straightforward, but does not offer specific positioning capability. Another possibility is to use electron-beam–fabricated carbon contamination spikes to form a mask [21, 23]. This type of spike has been successfully fabricated for use as a scanning tunneling microscopy tip [27]. It should also be possible to deposit other mask materials using an electron beam. For example, aluminum carbide can be deposited from the breakdown of trimethyl aluminum gas by an electron beam (T. F. Kuech, University of Wisconsin, private communication, 1995). Similarly, a metallic mask can be produced using a focused ion beam instrument, and this technique has been used to protect specific regions in the fabrication of samples for transmission electron microscopy (D. T. Foord, University of Cambridge, private communication, 1998). These approaches would make it straightforward to control the mask position and to interactively select features of interest to be contained in the field ion specimen.

It is also possible to deposit masks lithographically. These masks can be positioned both with and without regard to the underlying microstructure. In this manner, tips could be fabricated, for example, from a particular metallization region in a device or in a linear array across a grain boundary. This method holds promise for the precise placement of tips on specific features of interest if the required instrumentation is available.

Field Ion Specimen Preparation by IBME

Preparation of planar microtip specimens The feasibility of fabricating microtips by IBME for LEAP has been investigated by Larson et al. [21]. In this work, tips were formed on planar samples using 3 and 6μm diamond particles as masks for ion beam sputtering at normal incidence. Samples of copper, 304 stainless steel, a metal–oxide semiconductor structure, and a BiSrCaCuO superconductor were studied. It was found that tips could be formed from all materials examined. The tips were many microns tall with a radius of curvature at the apex of less than 100nm and shank angles down to about 20° (see Fig. 2).

A random-placement IBME can be utilized in a larger area to create multilayer specimens from even complex structures.
Figure 6 shows the results of ion beam mask etching of an Intel 286 processor [28]. Many tips from a large area of the specimen are shown in Fig. 6(top). This image illustrates that within the same specimen fabricated by IBME, there can be a range of tip outcomes from unfinished to overmilled. Some tips may also be poorly shaped for analysis. Figure 6(bottom) shows a sharp microtip with many layers of the structure visible in the image. Note also that the cone angle, $\alpha$, changes slightly at each layer. A range of tip outcomes can be due either to variations in the size and shape of the mask particles or to variations in the ion beam current. This fact can be used to advantage.

In Fig. 6(top), it is apparent that the upper right-hand side of the image has been milled more than the lower left-hand side. The ion beam creates this circular region, and the center of the region corresponds to the center of the beam. A typical instrument used for ion milling of transmission electron microscopy specimens produces an ion beam of about a millimeter diameter with a Gaussian current profile. When using the IBME technique, an effective approach is to ion mill until the region at the center of the beam is slightly overmilled. Under these conditions, there is a gradient away from the center in the amount of ion milling exposure. Far from the center of the beam will necessarily be undermilled. There will be a ring about the center where “perfect” microtips will be found. The inherent range of tip exposure can be used to study material at different depths in a structure. Using mask particles with a range of size can create the same effect. In principle, if the particles are attached to the substrate with a binder, this technique could be used to analyze the particles themselves.

In all of this work, it is clear that the ability to image the microtips can be essential to productive analysis of a specimen. As discussed in the Current State of Development section, an SEM can be an important integral part of a LEAP, and it can serve the important function of tip imaging for preview.

**Preparation of specimens for conventional atom probe geometries** Liddle et al. [25] employed the IBME technique in attempts to prepare field ion specimens from a III–V epitaxial-layered material. This work resulted in the fabrication of cones that were about 1$\mu$m tall. Due to the small height of these tips, no successful atom probe analyses of these materials was obtained. It was determined that a combination of small tip height and large substrate area resulted in a reduction of the electric field formed at the apex of the tips. Silicon tips of height ~50$\mu$m were later fabricated by Larson et al. [24], and this work was partially successful as it successfully lead to field ionization and field
evaporation from the silicon specimens. Kvist et al. [29] have also used a multistage procedure involving IBME to prepare specimens with 10μm of the surface in WC–TiC–Co-based materials and a CVD multilayer-coated cutting tool.

One of the primary applications of a LEAP is the analysis of thin-film structures. The large amount of research in this area indicates its importance and the potential advantages associated with successful thin-film field-ion specimen preparation. Initial work on preparation of field-ion specimens from thin-film structures was done by Melmed [30] and Camus et al. [31] on homogeneous thin films. Further work has been performed by various researches including Hono et al. [32], Pundt and Michaelsen [33], Cerezo et al. [34], and Al-Kassab et al. [35]. The typical method for fabricating multilayer-film field-ion specimens is to evaporate individual layers onto a prepared specimen of the proper needle geometry. The disadvantage of this technique is that it is not directly possible to correlate structure and composition results from this type of sample with magnetic measurements carried out on “flat substrate”-type films. Another preparation method for field-ion specimens containing a single-layer film has been reported by Hasegawa et al. [36]. This technique involves a combination of photolithography and pulsed micropolishing techniques, and results in a specimen with the film parallel to the tip axis. Although the Hasegawa technique results in the fabrication of a field-ion specimen from a flat-substrate multilayer film, it still requires that the material used to form the films can be easily electrochemically polished. Difficulties arise when the multilayer film materials do not polish well or have highly different polishing rates or if the final blank geometry is of nanoscale proportions. In such cases, another method of sample preparation, such as ion-beam milling, must be used.

**Focused Ion-Beam Milling**

*Needle-shaped specimens by FIB* Recently, progress has been made in the fabrication of field ion specimens from thin film structures using focused ion-beam milling. The application of a focused ion-beam instrument that allows concurrent imaging and modification of a specimen was initially proposed by Waugh et al. [37]. Advances in the capabilities of focused ion beams have recently enabled their successful use in the fabrication of scanning probe tips [38] and field ion specimens from various materials including nanoscale thin film structures [39–41]. In this work, focused ion-beam milling has been used to fabricate field-ion specimens from a multilayer-film structure containing alternating layers of 2nm-thick copper and 2nm-thick cobalt. This method of specimen preparation has made it possible to observe these layers by both field-ion imaging and three-dimensional (3D) atom-probe analyses [42–44]. The importance of this result is that it is now possible to correlate directly the 3D morphology and composition data obtained from atom-probe analyses with magnetic measurements from the same film.

In addition, atom-probe analysis of specimens fabricated by focused ion-beam milling has made it possible to quantify the implantation and damage induced by preparing samples in this manner [41, 42]. Field evaporation through the region damaged by gallium ion implantation (<30nm thick) has been demonstrated. This work shows the feasibility of using focused ion-beam milling for field-ion specimen preparation and this technique could be applied to the fabrication of samples for LEAP. A disadvantage of this technique when used to fabricate needles, however, is that it does not readily lend itself to imaging the layers with the layer normal parallel to the tip axis. With multilayer films in cross-section, it is possible to get local magnification effects that distort the ion trajectories significantly from different layers and limit the spatial resolution to a nanometer or more. This limitation is especially deleterious in thin multilayer films because they are typically on the order of a few nanometers thick, and the degree of interlayer mixing is of particular interest. In atom probe analyses, the tip...
axis direction is currently the only direction where atomic layer resolution is achieved, and thus, it is preferable that the layer normals are parallel to the tip axis. Although FIB could be used to fabricate needle-shaped specimens with their layer normals parallel to the tip axis, planar microtip specimens analyzed with LEAP should be much simpler and more expeditious. Planar microtip specimens of multilayer materials could be made by random-placement IBME if the area of interest is large, or site-specific FIB could be used as described below.

Site specific planar microtip specimens by FIB
With the amount of control available from a FIB instrument, it is possible to fabricate microtips from a specific location on a flat surface in a relatively short time like 1 h or less [45]. Higher energy ions like 50keV will shorten the milling time markedly. However, the higher beam energies can lead to significant ion implantation and damage [39]. For this reason a dual-beam FIB, which uses an electron beam for imaging, is preferable. A hollow-cone illumination pattern of the focused ion beam is used to sputter away material around a central tip. It is possible to locate the center of the microtip to within a few hundred nanometers. If this technique is to be used for specimen fabrication for LEAP, then sufficient material around the tip must be removed to allow an extraction electrode to be positioned above the microtip. Sharp tips can be fabricated with this technique. The possibility of using site-specific FIB to fabricate blunter tips may be especially appealing for analysis of very thin layers as described in the next section. A potential difficulty with this technique is that ion implantation and damage to the underlying material may intersect the analysis volume and must be considered.

The effect of counter electrodes on field
The use of an extraction electrode to localize and enhance an electric field is certainly not new. In 1968, the first results from microfabricated field emitter arrays were published [46]. These devices consist of many sharp tips (cathodes), each having its own metallic extraction electrode or “gate.” The radius of these tips is comparable to AP specimens, and the gate opening is about 1 to 2μm. Spindt et al. [47] found that field emission currents of 50 to 150μA can be achieved from single tips of radius 50nm using 100 to 300 volts compared to 1 to 30kV for etched wire emitters. In this case, the voltage is approximately a factor of ten less than what is normally required for a conventional field emitter with the anode essentially at infinity. Spindt et al. [47] also concluded that when the tip radius is in the 50 to 150nm range, rather than an inverse relation between tip radius and field, the radius appears to have only a second-order effect on the field. This fact will be considered further with regard to analysis of blunt tips in the next section. These structures have also been the subjects of theoretical modeling [48, 49]. It should be noted that there is a vast amount of work and ongoing developments in field emission arrays, and we have not attempted to assemble a complete review. The interested reader is referred to [50] for more details.

Spindt has also experimentally demonstrated field ionization using an extraction electrode geometry at voltages on the order of 1kV [51], which is much lower than the usual 5 to 30kV. Again, as might have been expected, this value is about a factor of ten less than what is normally required for field ionization. Even though, in this case, the field ionization is not used for FIM or field evaporation, this proves that not only does an extraction electrode localize the electric field, but it also leads to a significant enhancement in the local electric field so that low extraction voltages can be employed.

Extraction electrodes have been used in conventional APFIM [52, 53], and have recently been employed frequently by several groups [54, 55]. Although these electrodes are not “local” by the definition employed herein (see below), they still provide a significant field enhancement and an improvement in mass resolution when compared to the case where there is no counter
Local Electrode Atom Probes

electrode. Huang et al. [56] have recorded field ion images and AP data from a single tip and an array of FE tips where the applied voltage was less than 300 volts. The group at the Université de Rouen, France, have measured reductions of a factor of three in the applied voltage needed for field evaporation with a counter electrode that is on the order of a millimeter in diameter relative to a remote electrode [57]. The effects of the proximity of a local electrode on the electric field near a tip can be examined analytically using a simple paraboloidal model [58]. For example, a typical arrangement for AP evaporation of Rh$^{2+}$ consists of a tip of radius 45nm, an electrode at 1mm from the tip, a detector at some distance from the electrode, and an applied voltage of 11kV between the tip and the electrode [59, 60]. The electric field at the specimen apex calculated with the paraboloidal model is 45.6V/nm. A simple approximation for the field in a local electrode case can be made by taking the paraboloidal detector to be 1nm from the same tip. To duplicate the field of 45.6V/nm, only 3.95kV needs to be applied to the specimen. Hence, the proximity of the local electrode results in a field enhancement factor of slightly less than 3 for this particular case. This model only approximates the real case of a local extraction electrode. In our experience with detailed numerical modeling based on finite element methods, which is expected to be much closer to actual values, the actual field is always greater than this parabolic model by as much as another factor of 3 [61], which agrees with the experimental observations noted above.

CURRENT STATE OF DEVELOPMENT

EXTRATION ELECTRODE DESIGN AND FABRICATION

Because field ion evaporation from the anode requires a field that is about a factor of 8 greater than the field required for field electron emission from the cathode, the extraction electrode must be designed carefully to prevent field emission from occurring. Nishikawa and Kimoto [2] paid special attention to this issue in their first paper on SAP. Actually, if field electron emission does occur during a voltage pulse, the emitted electrons will be attracted to the specimen tip, which may help stimulate field evaporation. This process could be utilized as a means to accomplish pulsed-field evaporation. However, the creation of extraneous electron signals in the vacuum chamber during the pulse is a concern, because they can make it more difficult to detect ion arrivals at the detector. Constructing the extraction electrode with no protrusions that might act as a source of field electron emission will help minimize this possibility. In addition, the extraction electrode should have a high work function inherently, or it should have a coating that suppresses field electron emission. For example, Mousa [62, 63] has experimentally shown that field electron emission will be suppressed from aluminum and magnesium by their native oxides as long as the oxide is at least 2nm thick. This may be readily accomplished by native oxide formation, but could be enhanced with an oxidizing treatment. In addition, Mousa has experimented with epoxies [64] that are also successful at minimizing field emission. The issue of field electron emission from the cathode is one that will require attention on any instrument that is built, but it appears that this issue may be solved satisfactorily with existing technology. We note that Nishikawa [5–8] has successfully produced results with the Scanning Atom Probe with no obvious difficulty from field electron emission.

Nishikawa and Kimoto [2] suggested the use of silicon microfabrication techniques for fabrication of the extraction electrodes. In this approach, a hole would be created lithographically in the apex of a square pyramid of silicon. A limitation of this approach is that the height of the pyramid is limited to about 25μm. Bajikar et al. [65] developed a technique based on machining of a conical form on the end of a metal rod using a lathe and then replicating this form
with a removable coating. An example of this type of electrode is shown in Fig. 7 [66]. The machined rod is first coated with a polymeric release layer that also serves to smooth out the machining marks. A metal coating is then applied, and is released using a solvent. A hole is cut in the apex of the cone by a focused ion beam instrument [65–67]. Aluminum metal was used in this case for the electrode material.

The size of the aperture that is required for a SAP/LEAP has been considered. Nishikawa and Kimoto [2] looked at particular aperture diameters with electrostatic modeling and found several geometries that would work. Bajikar et al. [61] used electrostatic modeling to seek an optimal aperture size and position for a given tip (Fig. 8). They found that for a given aperture diameter (in this case, 1.5 μm), the field on the tip went through a maximum as a centered tip was moved axially (the z-direction in Fig. 1) relative to the aperture [Fig. 8(a)]. The maximum field in this case corresponds to a tip location just inside the aperture (position = +0.7 μm). The maximum in the tip-field-to-electrode-field ratio, however, occurs when the tip is just outside the aperture (position = +0.2 μm) [Fig. 8(b)]. This latter curve is slowly changing near the maximum, and taking these two figures together; they suggest that an optimal position for this case is just about where the tip enters the aperture (position = 0). The effect of aperture diameter on the field was also considered. Figure 8(c) shows that the field on the tip increases monotonically as the aperture diameter decreases. However, according to Fig. 8(d), the tip-field-to-aperture-field ratio goes through a maximum at around 2 μm diameter in this case. Because both a high tip field and a high tip-field-to-aperture-field ratio are needed, this particular set of electrostatic models indicates that a 1.5 μm-diameter aperture positioned with the tip just at the entrance plane of the aperture will achieve the best results for LEAP. Further detailed electrostatic modeling is warranted, but the general conclusions from this work should hold up.

**EXTRACTION ELECTRODE POSITIONING**

To position an extraction aperture above the tip of the specimen, it is necessary that the positioning stage has three axes of motion; lateral centering in two dimensions, and height adjustment. This positioning could be accomplished with a stage outside...
the vacuum system to place an aperture relative to a tip for insertion as a prealigned unit. Alternatively, a stage may be built for operation inside the microscope vacuum. The former approach would offer, at least, greater simplicity of the UHV instrument, while the latter would offer the opportunity to examine multiple tips on a single specimen in rapid succession. The following discussion will consider primarily the case of a stage that is internal to the vacuum system. This case is the more challenging, and such a stage can be used outside of a vacuum.

If a 1\,\mu m aperture is to be positioned precisely with respect to a tip of 0.1\,\mu m radius of curvature, then the positioning precision should be at least 0.1\,\mu m or finer. Consider further that a planar microtip specimen may be several millimeters across, and it may be necessary to change extraction apertures on occasion, which may be a few millimeters across. Thus, the demands on the stage that positions the tip relative to the extraction aperture are that it must be able to cover large distances (millimeters) in a short amount of time (tens of seconds) with high precision (tens of nanometers). Rotary motor-based stages can meet these motion requirements, especially the speed requirements, but for ultrahigh vacuum applications, they will be bulky and complex. Most of the technologies that are used to realize this positioning precision in ultrahigh vacuum are based on piezoelectric actuation. Because piezoelectric actuators achieve very precise motion in submicron steps, a slip/stick mechanism is used in almost all

**FIG. 8.** Calculated dependence of the field at the apex of a microtip 10\,\mu m tall with 100nm radius of curvature and $V_{\text{extraction}} = 3.5\,\text{kV}$ (from Bajikar et al. [61]). (a) Dependence of the field at the tip on axial position of the aperture (electrode) for a 1.5\,\mu m-diameter aperture. (b) Dependence of the ratio of the tip field to the electrode field on axial position of the aperture for a 1.5\,\mu m diameter aperture. (c) Dependence of the field at the tip on aperture (electrode) diameter. (d) Dependence of the tip-field-to-electrode-field ratio on aperture diameter. The solid line in (c) and (d) corresponds to the position where the field is maximum in (a), while the dashed line corresponds to the position where the tip-field-to-electrode-field ratio is maximum in (b).
of the actuators that meet these requirements. Inchworm actuators [68] may be used to move linear slides [69] on multiple axes at 250μm per second with 1nm precision. Compact stages are available [70] that have the slide and actuator assembled as a unit, and they achieve 1nm precision over 5mm travel with 100μm per second speed. Linear micropositioners [71] are a similar technology that can achieve very high linear speeds (10mm per second) with 50nm precision. These later actuators may be a bit too coarse for final positioning but could be useful, nonetheless, for aperture changing or coarse specimen movement.

Some form of feedback is needed for positioning the tip relative to the aperture. Specimen/aperture sets that are prealigned outside the vacuum would need to address this issue. Light microscopes have been shown to work adequately for this purpose [65]. This solution is difficult to implement inside the vacuum. Once the tip is close to the aperture, Nishikawa et al. [4] have shown that field electron emission may be used to center the tip in the aperture. In principle, the field ion image may also be used for fine alignment. However, prior to fine alignment, it is necessary to find a usable tip and achieve at least-course alignment. A visual feedback mechanism appears to be the most expeditious solution at this time. Light microscopes might be used, but they cannot readily provide the submicron resolution needed at long working distance to align and inspect the tips. Scanning electron microscopy offers submicron resolution at long working distances and appears to be a preferable solution. With resolution on the order of 20nm, the SEM may be used to select the best tips, based on their image, in addition to providing information for positioning the aperture. A single SEM imaging from the side perpendicular to the tip axis will make it possible to align laterally relative to one axis of motion. The second axis of motion, along the optic axis of the SEM, could be scanned in the field-electron emission mode until the signal indicates that the tip is aligned. Alternatively, a second SEM column located at 90° to the first and in the plane of the specimen can be used for alignment of the second axis. This adds additional cost and complexity, but should make the alignment process very simple. These various scenarios will be tested both technologically and economically in the years to come.

MASS RESOLUTION

From its first inception, there has been interest in improving the mass resolution1 of the atom probe. Pulsing of the voltage applied to the tip is used to achieve the pulsed-field evaporation required for time-of-flight measurements. The voltage pulses applied must have a subnanosecond rise time and a few nanoseconds duration. This pulsing introduces a time-varying electric field during ion acceleration. Because the mass spectrometry in atom probe is based on time of flight, mass resolution is improved by ensuring that ions of the same mass-to-charge ratio have a small spread in their time of flight. Ions that evaporate early in the pulse as the voltage is rising (a) begin their journey earlier than others, and (b) are accelerated by an electric field that is increasing initially. After the peak in the voltage pulse, ions that evaporate get a later start, experience a decreasing electric field, and achieve a lower speed than the early ions. They continually fall further behind in the race to the detector. Thus, the measured timing resolution, and hence, mass resolution, suffers in voltage-pulsed atom probes due to aspects that are inherent to the pulsing. The spread in time of flight for ions of the same mass-to-charge ratio can be 1 to 3%, and so the mass resolution can be as poor as 1 part in 50. One part in 500 is commonly considered adequate mass resolution for a large fraction of materials analyses, and one part in 1000 or better is preferred for a general-purpose instru-

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1The term mass resolution will be used here instead of the technically more correct “mass resolving power” because the former is widely used. The mass resolution cited here is defined as \( \frac{m}{\Delta m} \).
ment. Better resolution is always desired for separating closely spaced isotopic peaks.

If the timing electronics of a given instrument cannot fully resolve the time spread of identical ions, then higher mass resolution can be achieved by increasing the flight distance (longer total time of flight) at the cost of a decreased solid angle in both linear AP and 3DAP. Conventional atom probes may be fitted with devices that compensate for the kinetic energy spread of the ions such as a Poschenrieder lens [72] or a reflectron [9, 10]. The resulting mass resolution can be as high as 1 part in 2000. Good mass resolution for 3DAP is a topic that received attention at a workshop on 3DAP [73]. Cerezo et al. [74] have applied a reflectron to a 3DAP, and achieved very good mass resolution while passing a 3D image with minimal distortion. Further improvement of the mass resolution, perhaps without the need for a reflectron, remains a goal for 3DAP.

To realize high-mass resolution in a LEAP, it is important to either minimize or reduce (ideally to zero) the time spread of like ions. Thus, two approaches to improving mass resolution in LEAP are suggested: one based on minimization, and one based on reduction. Both paths to higher mass resolution are considered below. Note that the entire discussion below applies regardless of whether the ions actually depart during the same pulse or during different pulses.

Minimization of the Time Spread

Kelly et al. [22] looked at mass resolution of the LEAP with a first-order model based on ion energies. Assuming that the energy spread could be taken as comparable for LEAP and conventional APFIM, their conclusions were that secondary or post acceleration of the ions could be used to achieve a relative improvement in mass resolution. That is, with the extraction field as the primary acceleration field, a second acceleration field is used to bring the ions up to a high total energy so that the small energy differences between like ions becomes negligible relative to the total. In Fig. 9(a), a contour plot of the mass resolution of a conventional atom probe is shown as a function of flight path length and total voltage. At longer flight times and higher total voltages, the mass resolution is maximized at about one part in 200. In Fig. 9(b), this same plot is shown for a LEAP with secondary acceleration. In this case, the mass resolution is significantly higher under all conditions. The calculated mass resolution
for LEAP is severely limited in this case by the timer resolution of 1 ns, which has been used in the calculation.

This first-order study of the potential for secondary acceleration to improve the mass resolution of LEAP was followed by both further analytical study [75] and experimental work [76]. Bajikar et al. [75] developed an analytical model that treated two separate sections of the LEAP independently. The results of this work are similar to the earlier work of Kelly et al. [22]. They show explicitly, however, the effect of timer resolution on mass resolution for a given energy spread (in this case 0.5%). A LEAP with a 155mm flight length operating at a total voltage of 10kV will be severely limited by timer resolution of 1 ns to a mass resolution of about one part in 245 [Fig. 10(a)]. With a timer resolution of 156 ps, the calculated mass resolution would improve to about one part in 600 [Fig. 10(b)]. Commercially available multi-hit timers have historically offered 1-ns timing resolution [77]. Faster timers are available with time resolution on the order of 100 ps, but they are not capable of properly encoding multiple hits with a pair resolution on the order of nanoseconds or better. Thus, development of a 100-ps multi-hit timer would bring significant benefits to LEAP technology. In addition, these results suggest that the time spread for like ions that is desirable in a LEAP is on the order of 100 ps or less.

Bajikar et al. [75] found that it is best to accelerate the ions to high speed as early as possible after the extraction electrode. A secondary acceleration electrode such as the one shown in Fig. 1 could be used to accomplish this effect. This work also considered image magnification for the case where secondary acceleration is used. They pointed out that the high magnification of an atom probe, which operates in a point projection mode, is reduced by secondary acceleration. Fortunately, it appears that adequate image magnifications can be obtained in a LEAP. When the field distribution between the extraction electrode and the detector retains a high divergence, this helps retain a high magnification.

Bajikar et al. [76] performed an experiment with secondary acceleration using self-aligned apertures in a silicon array. This geometry was not ideal, because the secondary acceleration field penetrated the tip/aperture region so that as the secondary acceleration field was increased, the field on the tip also increased. In addition, the capacitance of this geometry was high enough that it was not possible to apply temporally sharp pulses to the tip. Nonetheless, evidence was obtained that the mass resolution was improved by application of a secondary acceleration field.

More recent work [78] has focused on how to reduce the time spread of like ions to a minimum. Because it is the time spread...
that ultimately matters in time-of-flight-spectrometry, this is considered a better basis for analyzing the performance of a LEAP. The time spread of ions originates from at least four sources: (1) the time spread in the time of departure, (2) the time spread that develops during primary acceleration of the ion to the extraction electrode, (3) the time spread that develops during free flight inside the extraction electrode, and (4) the time spread that develops during secondary acceleration toward the detector. More segments could be added as necessary to treat, for example, additional control electrodes. The interest in this work is to find how to minimize the overall time spread.

The total time that ions spend in the primary acceleration zone in a LEAP is on the order of 50 ps, so the time spread from segment (2) will generally be negligible. Furthermore, a short extraction aperture will minimize the time spread that develops in segment (3) due to differences in speed of ions in the field-free zone. The shortest that the aperture can be and still perform as a local aperture is the length where the field spillage from the secondary acceleration zone becomes significant. We have found for a 1 μm aperture that about 10 μm length is needed to ensure against field spillage. Nishikawa and Kimoto [2] found the same result in their initial study. Assuming a 1 kV extraction voltage, a typical ion of mass-to-charge ratio of 30 will be traveling at \( 8 \times 10^4 \text{m/s} \) through the extraction aperture, and will spend only about 100 ps in a 10 μm-long extraction aperture. This does not leave much time for time spread to develop. However, if the extraction aperture is 5 mm long, then the resident time inside the extraction aperture is 50 ns, and a time spread of several hundred picoseconds can develop.

Construction of a very short extraction aperture might be accomplished by forming a dielectric material into a cone and then coating only the apex region with a metal. A secondary acceleration electrode could also be introduced along the remainder of the extraction electrode.

The time spread that develops in segment (4) is minimized by minimizing the total flight time after the extraction aperture. This says that the flight distance must be as short as possible, and the ions must be accelerated to as high energy as possible. However, remember that it is the relative time spread, \( \delta t/t \), that is to be minimized. If we treat this segment as a region of constant acceleration throughout, then the equation of motion is [Eq. (3)]:

\[
L = \frac{1}{2} at^2 + v_o t + \text{constant}
\]

where \( L \) is the flight distance, \( a \) is the constant acceleration, \( t \) is time, and \( v_o \) is the initial speed of an ion entering the segment. The relative time spread is [Eq. (4)]:

\[
\frac{\delta t}{t} = \frac{-\delta v_o}{at + v_o}
\]

For a given speed spread, \( \delta v_o \), and initial speed, \( v_o \), the relative time spread is minimized in segment (4) by making the product, \( at \), as large as possible. So, a high acceleration and a long flight time (distance) are suggested. The length of the flight path is constrained by the desire to make the solid angle of the position-sensitive detector as large as possible. For a given timer resolution, the acceleration magnitude should be as large as possible such that the actual time spread is matched to the timer resolution. For the position-sensitive detector described below, a flight length of about 200 mm is chosen.

Finally, there is the question, What is the optimal voltage pulse shape? In the ideal case, ions would all evaporate at the same instant and would experience a constant primary acceleration field. This suggests two extreme cases: a short impulse, and a long square pulse. In the first case, a sharp voltage impulse [100 ps full-width half-maximum (FWHM)] can result in a short spread in time of departure (about 33 ps). (For a 100-ps FWHM impulse, the rise time and fall time are each about 100 ps. Because ions evaporate within only the top 3% of the applied field, for a pulse fraction of 20%, this corresponds to the top one-sixth of the pulse. In time, this is one-sixth of the
rise and one-sixth of the fall or 33-ps total.) However, ions will depart over a range of voltages near the peak. Furthermore, primary acceleration of the ions takes only about 50 ps in a LEAP. Thus, the extraction voltage is still changing rapidly during primary acceleration and ions with different departure times are accelerated differing amounts. Thus, in this first case, the time-of-departure spread is low but the speed spread is high.

In the second case, a square pulse would be such that ions could evaporate any time during the pulse, but they would all see the same constant acceleration field. In this case, the time-of-departure spread is large but the speed spread is small. However, a perfectly square pulse does not exist, and the ions that depart on the rise or the fall of the voltage will be accelerated differing amounts. Furthermore, if we are trying to keep the total time spread to about 100 ps, then a longer time-of-departure spread will defeat the whole purpose. From this standpoint, it appears that it is best to use a fast impulse for voltage pulsing the LEAP and use secondary acceleration to minimize the overall relative time spread and thus maximize the mass resolution. Voltage pulsers are available that can deliver sub-200-ps FWHM pulses of up to several hundred volts (S. Fulkerson, Lawrence Livermore National Laboratory, private communication, 1996) [79, 80]. Note that many commercially available pulsers are designed to deliver very clean pulses with no ringing or other structures. For pulsing a LEAP, an impulse need only be clean on its top one-third or less of the pulse height. Faster pulses can be produced when it is not necessary to design against pre- or postringing of the pulse.

Reduction of the Time Spread

Once a finite time spread exits between two ions, this time spread can be reduced only by changing (a) their relative speeds, or (b) their relative path lengths to the detector. Poschenrieder lenses and reflectrons operate on both (a) and (b) simultaneously. Nishikawa [6] has used a reflectron on his SAP to reduce the time spread of the ions.

Because there is a field-free zone early in the flight path of a LEAP (inside of the extraction aperture), there is a possibility for reducing the time spread with a tailored pulse shape. If an impulse were to evaporate two ions at different start times, then the first ion has a head start and a higher speed. If the primary acceleration field is continuously increasing, then once the first ion enters the field-free zone of the aperture, the second will be accelerated to higher energy without affecting the first ion. If the voltage rise is fast enough, this would boost the energy of the second ion relative to the first, and provide time focusing at some point in the path. Ideally, the ions should be time focused at the detector. The challenge here is to achieve this time focusing with realistic aperture lengths and pulse shapes. Because the transit time of the ions to the aperture is on the order of 50 ps, this scenario may only be feasible if the aperture, or a separate correction field, is moved further away to increase the time available for the rising pulse to act on the ions. Furthermore, it is not clear that this can be made to work on more than one ion type at a time. If this time focusing can be achieved, then the other approaches to improving mass resolution become less important.

Data acquisition rate

Higher data collection rates are of interest for atom probes because the sensitivity of the technique is directly improved as the number of atoms collected increases. Because the pulse magnitude in a conventional atom probe needs to be on the order of 20% of the standing voltage [81a], a pulse generator must produce on the order of 3 to 4 kV pulse magnitudes. Historically, the prevalent device for producing these pulses with the required performance is a mercury-wetted reed switch. Reed switches are mechanical devices that produce up to 7 kV [82], 1 ns rise time, 5 ns-long pulses at frequencies up to about 200 pulses per second. Recently, Behlke switches [83] have been used in com-
commercial high voltage solid-state pulsers [84, 85]. These latter pulsers can operate at a 1.7-ns rise time, 8-ns long pulses at frequencies up to $2 \times 10^3$ pulses per second. Because ion flight times do not normally exceed $20 \mu s$ (2$\mu s$ in 3DAP), atom probes could be operated at much higher speeds, like $10^3$ pulses per second, if higher repetition rate were available.

Because the voltage needed to create the high field for evaporation in a LEAP is much smaller than in a conventional atom probe, the pulse voltage required for a 20% pulse fraction is also much smaller. With the technology available today, as the voltage magnitude of a pulse decreases, it is possible to make the temporal length of the fastest pulse smaller and the maximum repetition rate higher. Solid-state voltage pulsers are commercially available (S. Fulkerson, Lawrence Livermore National Laboratory, private communication, 1996) [79, 80] that can produce pulses of several hundreds of volts with a 100 ps rise times, a 100 ps FWHM, and repetition rates up to $10^5$ pulses per second. Note that some attention must be paid to minimize the capacitance between the tip and the extraction electrode if 100-ps impulses are to be realized in practice at the tip. At repetition rates of $10^5$ per second and greater, the power supplies for solid-state voltage pulsers become large and expensive. It is reasonable to expect, however, that solid-state pulsers can be pushed to provide pulsing rates up to $10^6$ per second if necessary (S. Fulkerson, Lawrence Livermore National Laboratory, private communication, 1996).

**Detector technology** In a conventional atom probe, the needle-shaped specimen is manipulated on a goniometer through two axes so that, for example, a feature of interest in a FIM image may be rotated into coincidence with the probe hole for analysis. The requirements on eucentricity of this goniometer are low because movement of the specimen apex on the order of a fraction of a millimeter is insignificant. If we consider that the extraction aperture in a LEAP is on the order of 1$\mu m$ in diameter, then it is clear that an unrealistic degree of eucentricity would be required in a LEAP stage to allow real-time rotation of the specimen relative to the aperture. There are four alternatives apparent: (1) use a multistep process where the tip is retracted, rotated, then repositioned in the aperture; (2) rotate/translate the entire aperture and tip assembly as a unit to sweep the image across the detector; (3) rotate/translate the detector to sweep the image across the detector; or (4) make the detector large enough to capture the entire field ion or field-desorption image. Alternatives 1 through 3 do not require any new technology, and can be implemented based simply on which is the best option in a given circumstance. Alternative 4 offers several significant advantages if it can be realized. They are explored below.

Making a detector large enough means that it must have enough pixels to fully record the field ion and field-desorption images. If we assume that a wide-angle field ion image is 500 atoms across, then a detector with about $2,000 \times 2,000$ pixels is required to record such an image with high quality. There are several detector technologies that can reach this level of resolution including charge-couple devices (CCDs), and some single-particle detectors [86–89]. A detector based on delay-line technology has the potential to provide this level of resolution. One advantage of delay-line technology is that the same type of timer used in the position determinations can also be used for measuring ion times of flight. The projected performance of a detector under construction by the lead author is $3 \times 10^6$ properly encoded hits per second at $2,000 \times 2,000$ resolution. The high data rate is needed if the large number of pixels is to be populated with data in a short amount of time. It can be made to any physical size from $75 \times 75$mm active area to $200 \times 200$mm active area.

Consider the advantages of this detector for a LEAP. First, the high data rate means that it can be used to record real-time field ion images in addition to field desorption images. This greatly simplifies the design
of the instrument by eliminating the need to switch specimen positions between two or more detectors. Furthermore, because there is no need to rotate the specimen into a final analysis position, a simple fixed mounting can be used. This greatly facilitates the connection of cryogenic cooling to the specimen (see below). The specimen could be mounted directly on the cold finger of the cryo-cooler if a vibration-free cooler is available (see below).

The high data rate of the detector makes it possible to consider high pulsing rates on the field evaporation process, which, as mentioned above, should be possible with LEAP. With the high resolution of the detector, a large swath may be recorded through the sample in the field desorption mode. A large swath diameter means high sensitivity per atomic plane, high sensitivity to localized 3D concentrations, and high efficiency of specimen utilization. The complexity in this case goes into the detector and the rest of the instrument is, therefore, greatly simplified.

Cryo-cooling of the specimen To freeze diffusive movement of atoms on the surface of a specimen, it is necessary to cool the tip to cryogenic temperatures in an atom probe [81b]. The resolution and contrast in FIM images are both markedly improved at low temperatures relative to room temperature. Different materials require different temperatures for best operation. Aluminum alloys typically require very low temperatures like 20 to 40K, while steels are typically analyzed at 60K. A LEAP should be no different in this requirement. It may be slightly easier to reach the low temperatures with a LEAP geometry because the specimen is planar, but this is countered by the fact that the nearby extraction aperture is at room temperature.

Vacuum requirements for LEAP LEAP, like the conventional atom probe, is an ultrahigh vacuum technique. Because surface atoms are analyzed, it is necessary to keep that surface clean from contamination. Base pressures of low $10^{-9}$Pa (low $10^{-11}$mbar) are desirable to keep tramp gas atoms off the specimen. Actually, during a typical operation, an imaging gas like neon or argon is admitted into the main chamber to a pressure of about $10^{-3}$Pa ($10^{-5}$mbar) for field ion imaging. Upon completion of the imaging, the imaging gas is pumped out to a pressure of about $10^{-7}$Pa ($10^{-9}$mbar). This latter pressure is best for atom probe analysis because it has been found that the field needed to evaporate atoms decreases slightly as the pressure rises from $10^{-9}$ to $10^{-7}$Pa ($10^{-11}$ to $10^{-9}$mbar). The lower field helps reduce the occurrence of catastrophic mechanical specimen fractures.

In a LEAP, these same considerations apply for the most part. There are two possible exceptions. First, if the data rates of a LEAP are much higher than conventional atom probes, then the relative rate of contamination of the data by tramp elements in the vacuum is decreased proportionately. By this argument the base pressure could afford to be higher for a given contamination rate, but it seems more prudent to keep the base pressure low and realize a lower contamination rate in the data. Second, the shorter shank length of tips and the different field distribution along the shank due to the local extraction aperture should lead to different mechanical stress distribution in microtip specimens relative to needle-shaped specimens in a conventional atom probe. Whether these stresses are lower or higher has yet to reported in the literature. If they are lower, then the pressure during analysis could be lower, perhaps at base pressure. The shorter shank length of microtips will mean a lower probability of a fatal mechanical flaw in the tip. At this point, however, it remains to be shown whether microtip specimens in a LEAP have a higher or lower propensity for mechanical failure than needle-shaped specimens in a conventional atom probe. The conclusion at this time is that a LEAP should be designed as an ultrahigh vacuum system with the same considerations as a conventional atom probe.

Mechanical vibration Conventional atom probes are insensitive to mechanical vibration.
This is not the case for LEAPs, however. The relative vibration of the tip and the extraction electrode has the potential to alter the magnitude of the field at the tip, alter ion trajectories, and in the extreme, make operation impossible. Indeed, one method of pulsing the field evaporation would have the tip mechanically oscillating along the tip axis direction such that the peak field encountered is sufficient to induce evaporation. Furthermore, in any three-dimensional atom probe, the field distribution must be axially symmetric along the tip axis. Otherwise, asymmetric fields will lead to anisotropic magnification of the desorption image. It is, therefore, essential that relative vibration of the tip and aperture, which would introduce field asymmetries, be eliminated in the instrument.

The main concerns are isolation from ambient vibration sources and prevention of vibration from vacuum pumps and cryo-coolers. There are three prominent technologies for isolation of the vacuum chamber from vibration: pneumatic systems [90, 91], mechanical systems [92], and active systems [93]. Pneumatic isolation systems are probably adequate for the job. They have been used extensively with electron microscopes that achieve subnanometer resolution. The main advantages of mechanical systems are that they achieve greater isolation of the system from ambient and they never need servicing. Mechanical systems can be more expensive than pneumatic systems, however. Active systems are the most complex and tend to be the most expensive solution. Their performance is excellent in general.

The stage should be designed to be insensitive to external vibration to the maximum extent possible. The fixed specimen design mentioned above is desirable from this standpoint as long as the cryogenic cooling system does not introduce vibration. In general, the aperture and tip assembly should be mounted on the same basic mount assembly so that they vibrate as a unit and not differentially.

Magnetically levitated turbomolecular pumps have been used on electron microscopes for over a decade, and should be adequate for this application. Ion pumps on the main chamber can be used, but they should be coupled with a throughput pump such as a turbomolecular pump to pump the inert gases often used for field ion imaging.

Convenient cryogenic cooling of the specimen without vibration is perhaps the biggest challenge. Multistage helium compressor cryopumps are able to cool to about 14K, and are the standard in conventional atom probe technology. However, they produce far too much vibration for the LEAP application. Vibration-isolated helium compressor cryopumps have been built by individual research teams from commercially available components (R. Dykhuizen, Sandia National Laboratory, private communication, 1998; and H-O Andrén and A. Kvist, Chalmers University of Technology, private communication, 1998). This solution looks promising for this application. It requires that the cryohead be mounted independently of the chamber, and compliant connections to the vacuum chamber are used. Heat is transferred by helium gas between the vibrating cryohead and the vibration-isolated specimen holder. A complete commercial system of this type is available from Advanced Research Systems [94].

In those cases where liquid helium is available, gaseous helium boil-off from the helium bottle can be directed through the specimen holder. This approach creates minimal vibration and excellent cooling, but requires a supply of liquid helium. Similarly, liquid neon cooling is a straightforward and inexpensive method of cooling that will reach about 27K. Liquid nitrogen is even less expensive, but it will reach only 80K, which is not a sufficiently low temperature for most AP analyses.

Electromagnetic fields Conventional atom probes are insensitive to low-level electromagnetic fields in the environment. This is not necessarily the case for LEAPs, however, if a SEM is used to image the tip and aperture region. A typical SEM is sensitive to time-varying electromagnetic fields that are on the order of a few milligauss or greater in magnitude.
Depending on details of the shielding and design, a LEAP using SEM imaging will most likely have similar constraints.

**PROSPECTS FOR FUTURE DEVELOPMENTS**

In addition to instrumental developments, there are several specimen geometry developments particular to a LEAP that are worth considering.

**BLUNT TIPS**

In a conventional APFIM, the specimen must have a radius of curvature at the apex that is less than about 100nm to achieve the high electric field needed for field evaporation. The electric field on the tip is generally recognized as being inversely proportional to the apex radius of curvature [81c]. With a local electrode, the dependence of the high field on the radius of curvature at the apex is reduced, and the high electric fields can be produced even at modest voltages. Not only does this make it possible to continue analysis of tips to a blunter condition to get more material removed, but it also makes it possible to consider utilizing blunt tips purposefully, as illustrated in Fig. 11. For example, using numerical modeling, Bajikar [95] showed that within the limits of expected LEAP operation, a tip radius of curvature up to 0.5µm will still allow electric fields high enough for evaporation to be achieved.

The consequences are important for work on interfaces and other 2D structures. At a 0.5µm radius, the field desorption image will contain about 5,000 atoms across the diameter. This corresponds to about 25 million atoms per atomic plane. Note that at this image size, the detector resolution would not be sufficient to detect atomic spacings laterally (x-y directions in Fig. 1). Furthermore, a reconstruction of the three-dimensional image over such a wide area may not achieve atomic resolution in the longitudinal direction (z direction in Fig. 1) either. However, this tradeoff of resolution for sensitivity can be beneficial in many applications. With this sort of 3D image, the sensitivity of the technique would exceed parts per million per atomic plane. A good example of where this sensitivity is needed can be found in the semiconductor industry today. The dopants in shallow implant structures are only about 100 atom layers deep. The total number of atoms detectable in a volume of this depth is about 2.5 billion. If we take 10 atoms as the minimum

![Fig. 11. Schematic illustration of the concept of using blunt tips to realize a greater number of atoms analyzed per atomic layer.](image-url)
number that represents a statistically significant sample, then this translates into a volume sensitivity of 4 atomic parts per billion or $2 \times 10^{14}$ dopant atoms per cm$^3$ of silicon. This is a very low concentration.

Another possible application of blunt tips will be for analysis of submicron structures. As device size diminishes, characterization of these structures, especially in three dimensions, becomes a harder problem. 3DAP could be used to study such structures but a lateral image size of hundreds of atoms would make it very difficult to obtain the data. However, large sections of a submicron device can be contained within a blunt tip. If tip formation is accomplished at a specific location as mentioned above, then targeted regions of a very large integrated circuit could be analyzed. These regions might be either deliberately fabricated test sections on a wafer or sections of a device that has been shown to possess a defect.

SPECIMENS OF LOW ELECTRICAL CONDUCTIVITY

Except in a few cases, atom probe analysis has been performed almost exclusively on specimens that have a high electrical conductivity. This is primarily because the high-voltage pulse that initiates field evaporation must travel down the length of a slender needle. In poor electrical conductors, the pulse is heavily attenuated before it reaches the specimen apex and it is inadequate to cause field evaporation. Two methods have been used to overcome this problem for poor conductors. In the first, a pulsed-laser atom probe was developed by Kellogg and Tsong [96] to thermally pulse low-conductivity materials like silicon. In the second, specimens of poor electrical conductors have also been treated with an electrically conducting coating like carbon [97, 98]. During analysis, the coating is field evaporated away from the analyzed region, and generally does not influence the results. This latter approach should also work for LEAP specimens. Figure 12 illustrates this concept schematically. After a planar microtip specimen is prepared, a thin coating of an electrical conductor can be applied to the top surface. One potential advantage of the planar microtip in this regard is that it may be easier to achieve the coating on any standard coating system, but this is a minor point. More importantly, because the thickness of a planar microtip specimen is on the order of a millimeter or less, the voltage pulse through the speci-

Fig. 12. Schematic illustration of the concept of coating an electrically insulating material with a conductive layer so that LEAP analysis may be performed.
men will be much less attenuated than in a needle-shaped specimen. Thus, for these reasons, it should also be possible to analyze uncoated specimens in LEAP that are lower in electrical conductivity than may be analyzed in a conventional APFIM. Handling of weak or brittle materials should also be easier with planar microtip specimens than with needles.

**LEAP AS A METROLOGY TOOL**

For an instrument to qualify as a metrology tool, it is necessary that analyses be completed in a time frame that allows the information to be used as feedback for further processing. In a conventional APFIM or 3DAP, no methodology has yet been developed where this is a realistic possibility. Complete analyses that do not destroy the overall viability of an entire wafer must be performed in a matter of hours or less. It appears, however, that a LEAP could be configured to suit this task. An FIB may be used to create microtips from specific locations on a wafer. These locations may be test sites that have been engineered into the wafer design. Facilities for handling and cooling wafers in ultrahigh vacuum have been developed by the semiconductor processing industry. A FIB could be incorporated in the metrology station. Depending on the information that is sought, 3D images could be recorded and analyzed from several sites on the wafer within the several-hour constraint. There would be considerable development work needed to make this a routine task, but it is no more daunting than similar metrology tasks that are currently performed by analytical instruments.

**CONCLUSIONS**

All of the scientific bases of LEAPs have been established. Although dedicated systems have not yet been completed, LEAP has the potential to reach:

1. pulsed evaporation rates of $10^6$ pulses per second;
2. mass resolution of one part in 500 or better; and
3. billion atom, three-dimensional images.

LEAP makes innovative new specimen geometries possible that could push the sensitivity limits of two-dimensional structures to parts per million per atomic plane. Part per billion analyses should be possible in volumes of 1-μm diameter by hundreds of atomic layers thick. With this type of performance, LEAPs will compete favorably with other analytical techniques like secondary ion mass spectrometry, bulk chemical analysis, electron probe microanalysis, and scanning auger microprobe.

LEAP can play a crucial role in characterization of materials with nanometer-scale compositional structure in three dimensions. In a user-friendly package, a LEAP could become a routine analytical instrument. The semiconductor and thin-film industries especially need a means for imaging structures in three dimensions at the atomic scale. 3D LEAP may be the only technique that can reach that goal in the near future.

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