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Original Article

My Life With Erwin: The Beginning of an Atom-Probe Legacy

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Abstract

The atom-probe field ion microscope was introduced in 1967 at the 14th Field Emission Symposium held at the National Bureau of Standards (now, NIST) in Gaithersburg, Maryland. The atom-probe field ion microscope was, and remains, the only instrument capable of determining "the nature of one single atom seen on a metal surface and selected from neighboring atoms at the discretion of the observer". The development of the atom-probe is a story of an instrument that one National Science Foundation (NSF) reviewer called "impossible because single atoms could not be detected". It is also a story of my life with Erwin Wilhelm Müller as his graduate student in the Field Emission Laboratory at the Pennsylvania State University in the late 1960s and his strong and volatile personality, perhaps fostered by his pedigree as Gustav Hertz's student in the Berlin of the 1930s. It is the story that has defined by scientific career.

Key words: atom-probe tomography, atom probe, microscopy, imaging

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The atom-probe field ion microscope (APFIM) was; arguably, Erwin Müller's greatest achievement, see Figure 1. Although the field emission microscope; or FEM, was revolutionary when he introduced it in 1937 (Müller, 1937) and the field ion microscope; or FIM, promised single-atom resolution when introduced in 1951 (Müller, 1951) the APFIM allowed one single atom to be selected from neighboring atoms on a metal surface and identified by mass analysis. To achieve this goal, an FIM was operated at a cryogenic temperature so that single atoms could be resolved (Müller, 1956). The sample (or tip) was moveable, allowing the image of the selected atom to be positioned over a small hole that provided entrance to a mass spectrometer. The process of field

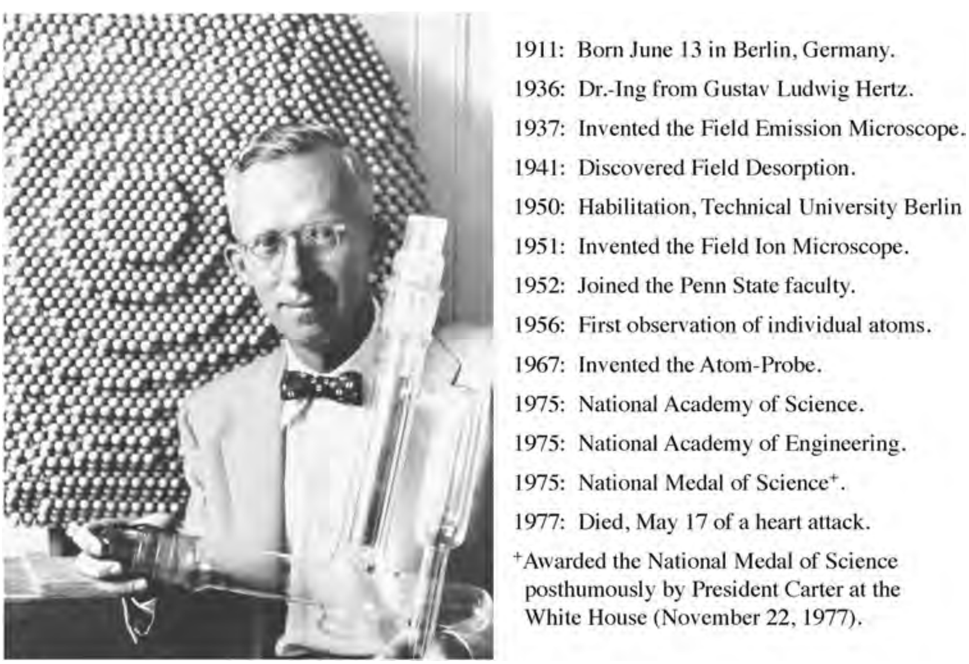

Figure 1. The Atom-Probe Field Ion Microscope was Erwin Wilhelm Müller's last major achievement.

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Figure 2. The Field Emission Lab at Penn State in 1966, celebration for the birth of Osamu Nishikawa's second son (Shoji) born May 9, 1966. Top Row: John Panitz, Douglas Barofsky, Klaus Rendulic, Brooks McLane. Middle Row: Jay Politzer, Myron Hicks, Tien Tsong, Gerry Fowler. Bottom Row: Sandi Mori, Osamu Nishikawa and son Albert Noboru Nishikawa, Erwin Müller.

desorption (Müller, 1941)—now called "field evaporation"—removed the atom as a positive ion that entered the mass spectrometer.

The saga of the APFIM begins in 1966 in Erwin's Field Emission Laboratory at Penn State University, where a dedicated group of students, faculty and staff were ready to take on, seemingly, any challenge, see Figure 2. Activity in the Field Emission Laboratory was centered on the FIM. At the time, atomic resolution imaging in the FIM was a routine procedure. Before and after events (e.g. before and after ion implantation) could be visualized by an optical color comparison of two black and white FIM images but the chemical identity of an atom could not be determined.

In the fall of 1966 Erwin had returned from European travel and suggested the concept of an "Atom-Probe", that he named in analogy with Raymond Castaing's "Electron Probe" (Castaing, 1960). I had just completed my Master degree in Physics under the direction of Professor Müller and was selected to oversee the prototype APFIM and create its single-atom detector. S. Brooks McLane, the lab's electronic technician, was to supply his expertise and Müller was to direct and involve himself in all aspects of the project. The prototype APFIM was completed in 1967, see Figure 3. Erwin presented the APFIM at the 14th Field Emission Symposium in Gaithersburg, Maryland and received a standing ovation for his accomplishment (Müller and Panitz, 1967). Shortly thereafter the APFIM project was funded by the National Science Foundation for two years, Erwin filed a patent application and therein lies the Douglas Barofsky story.

Douglas Barofsky was a graduate student, completing his PhD thesis on the mass spectroscopy of field evaporated ions with Erwin Müller when Erwin proposed the APFIM. Erwin was inclined to use Doug's magnetic sector mass spectrometer for the APFIM but Doug suggested using a time-of-flight (TOF) mass spectrometer, an instrument that was discussed in Bruce Kendall's ion optics course that Doug had attended in 1965. Not only did Doug propose using the TOF spectrometer (that was incorporated into the prototype APFIM), but he also spent considerable time and effort discussing and writing the patent application with Erwin. The final patent was granted in 1971 (US Patent 3,602,710) with Erwin listed as the sole inventor. Despite all of Doug's input his contributions were never publicly acknowledged.

Erwin and I had a somewhat "stormy" relationship that often involved assigning credit. An example is the only remaining piece of the prototype APFIM, a glass chamber that provided a ball joint for tip movement and a method to separate the FIM imaging gas (at a pressure of several millitorr) from the high vacuum environment of the TOF. This was accomplished by differential

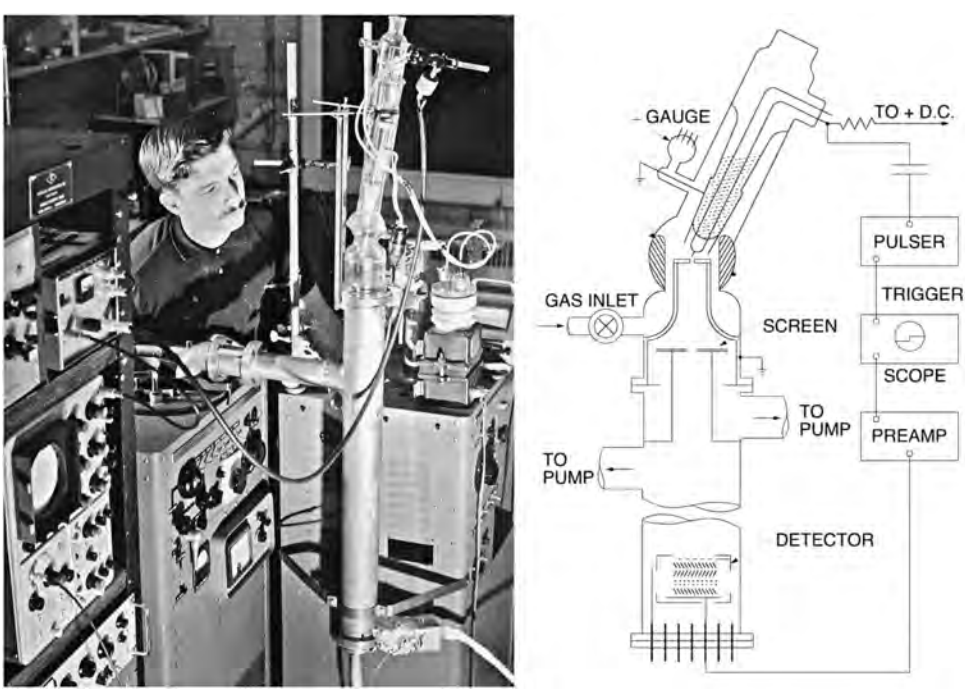

Figure 3. During 1966 the first Atom-Probe was constructed in the Field Emission Lab at Penn State (overseen by John Panitz).

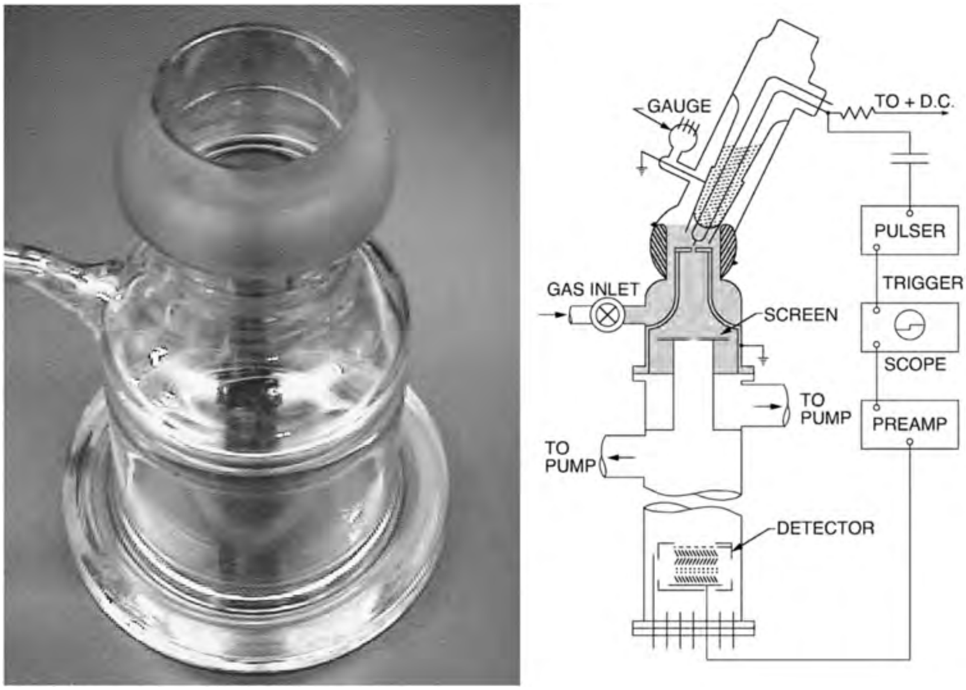

Figure 4. Only one glass piece of the Prototype Atom-Probe Field Ion Microscope survives and it's a story in itself!

pumping through a small orifice, see Figure 4. I was an accomplished glassblower at the time having worked during my high school years, building quartz sample cells for Harold Babrov at a division of the Warner and Swasey company in Flushing, New York. Erwin, Brooks and I had discussed several designs for the glass chamber, including the one shown in Figure 4 that I had

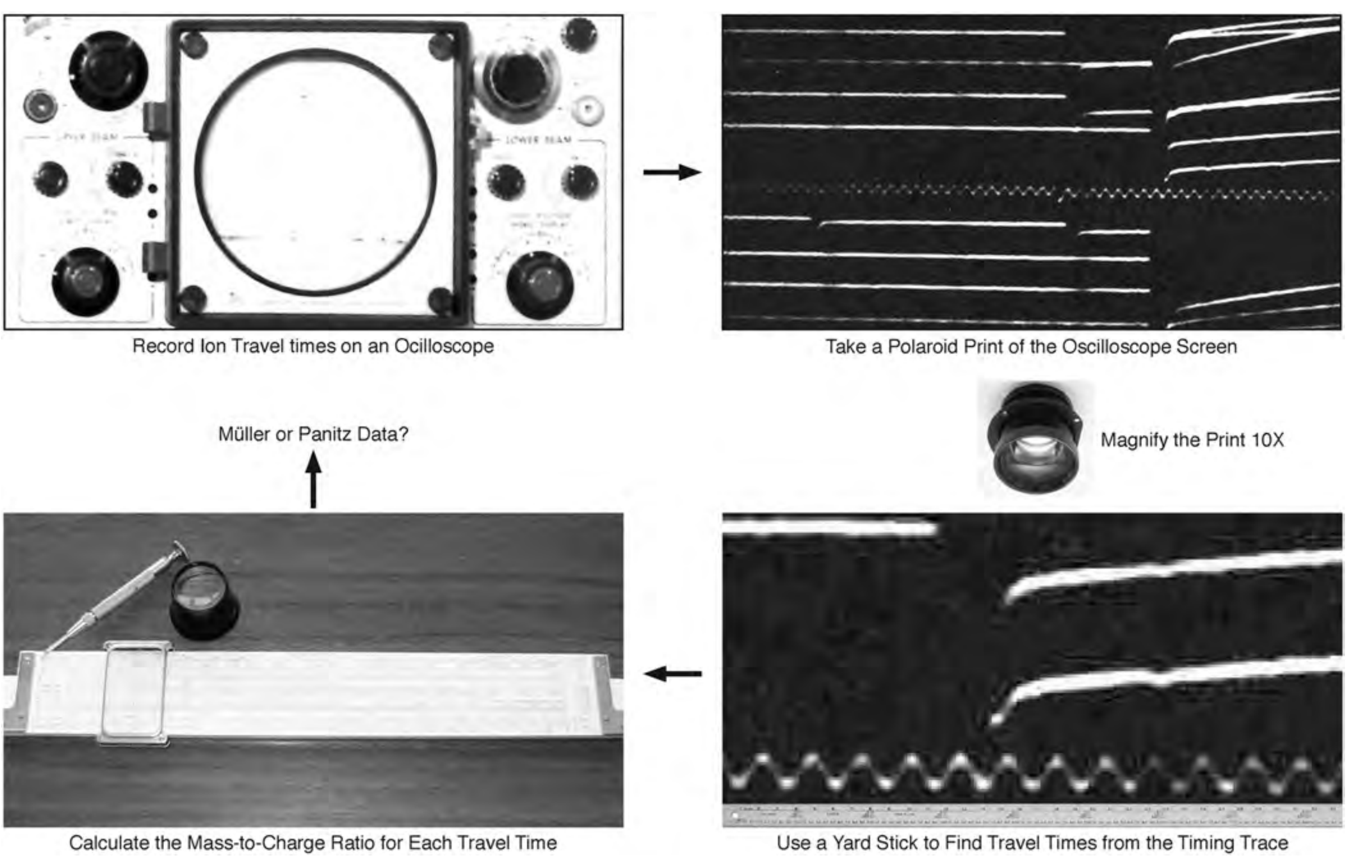

Figure 5. Data collection and analysis was always a challenge as summarized here.

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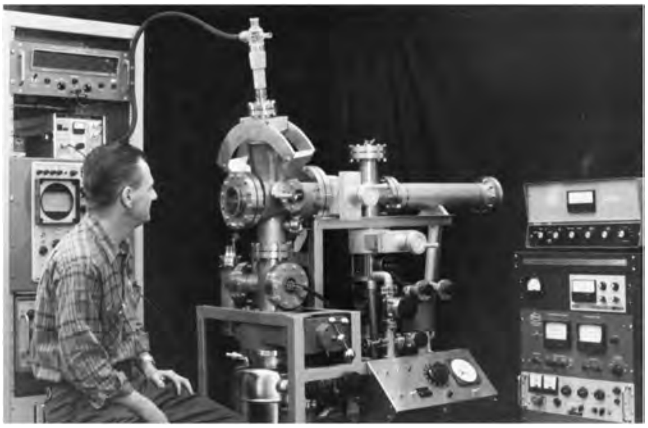
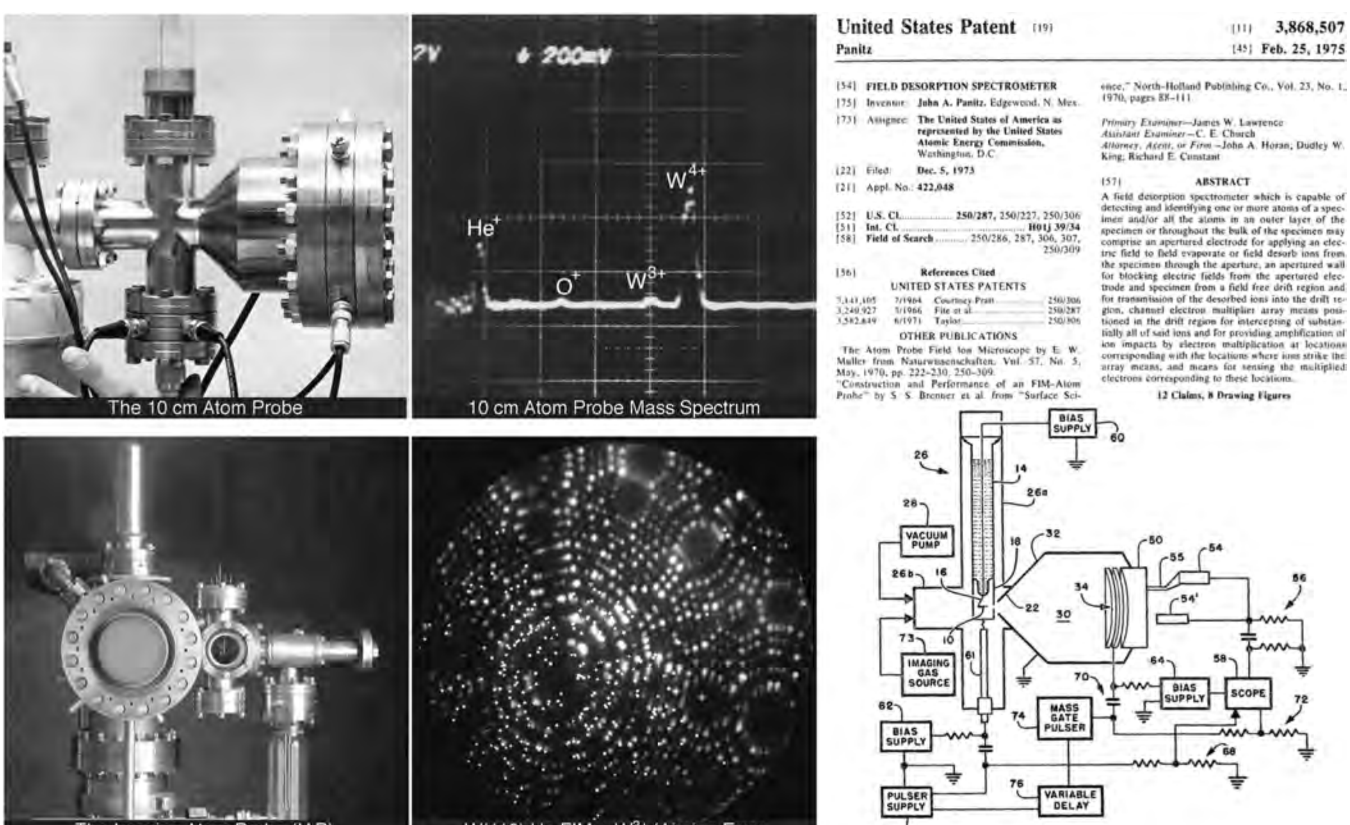


Figure 6. Gerald Leroy (Gerry) Fowler is shown here with the second Atom-Probe Field Ion Microscope that he constructed in 1968. Gerry was a technician in the Field Emission Laboratory at Penn State where he designed and constructed instruments and helped to educate a cadre of Erwin Müller's students. This Atom-Probe was wheeled out of sight if visitors were expected. Only two grooves in the floor led to a closed door!

sketched on a blackboard in Brook's office. Erwin, however, re-sketched the design a day or two later on the same blackboard, and Brooks could only shake his head in disbelief!

Another point of contention was who "owned" the data recorded from the APFIM. Erwin, Brooks, and I recorded several hundred TOF data records each Friday and by Monday the data had to be analyzed to find the mass-to-charge ratios from the travel times and voltages. If I had the data analyzed by 8 am on Monday morning it was considered "my data" for use in my thesis; if not, Erwin would have had the data analyzed and it became his data. This was a great incentive for a graduate student to acquire a work ethic! See Figure 5. Although I now relate these and similar stories as idyllic encounters with Erwin; at the time (I am told), I voiced considerable opposition in private.

In 1968 the prototype APFIM appeared in the literature (Müller et al., 1968) and I wrote the draft of the published paper and included Gerry Fowler as a coauthor. Although Gerry helped with the construction of the prototype instrument, Erwin removed his name from the final paper and Gerry's contributions; like Doug Barofsky's, were never publicly acknowledged. Gerry constructed the second atom-probe from stainless steel components that he built or purchased and that he tungsten inert gas welded (Fig. 6). The exception was a pyrex "cold finger" that held liquid hydrogen to cool the sample tip to 21 K via thermal conduction through the tungsten high voltage leads. The Field Emission Laboratory was situated on the top floor of the Osmond lab building because of the potential hazard of a hydrogen explosion (we all remembered the tragedy of the Hindenburg). Hydrogen sensors were placed throughout the lab to monitor the hydrogen-air mixture and Environment, Safety & Health (ES&H) was only a glimmer in the mind of man. There was a close call when Erwin, Brooks, and I were taking data. During an experiment a small "pop" was heard and a small blue flame was seen burning on top of the liquid hydrogen in the cold finger. Erwin immediately forced us out of the room while he extinguished the flame. When he called us back we resumed the experiment as if nothing had happened!



United States Patent [19] [11] 3,868,507
Panitz [45] Feb. 25, 1975

[54] FIELD DESORPTION SPECTROMETER
[75] Inventor: John A. Panitz, Edgewood, N. Mex.
[73] Assignee: The United States of America as represented by the United States Atomic Energy Commission, Washington, D.C.
[22] Filed: Dec. 5, 1973
[21] Appl. No.: 422,048
[52] U.S. Cl. 250/287, 250/227, 250/306
[51] Int. Cl. H01j 39/34
[58] Field of Search 250/286, 287, 306, 307, 250/309
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[57] ABSTRACT
A field desorption spectrometer which is capable of detecting and identifying one or more atoms of a specimen and/or all the atoms in an outer layer of the specimen or throughout the bulk of the specimen may comprise an apertured electrode for applying an electric field to field evaporate or field desorb ions from the specimen through the aperture, an apertured wall for blocking electric fields from the apertured electrode and specimen from a field free drift region and for transmission of the desorbed ions into the drift region, channel electron multiplier array means positioned in the drift region for intercepting of substantially all of said ions and for providing amplification of ion impacts by electron multiplication at locations corresponding with the locations where ions strike the array means, and means for sensing the multiplied electrons corresponding to these locations.

12 Claims, 8 Drawing Figures

Figure 7. Between 1973 and 1974 the Atom-Probe was reinvented.

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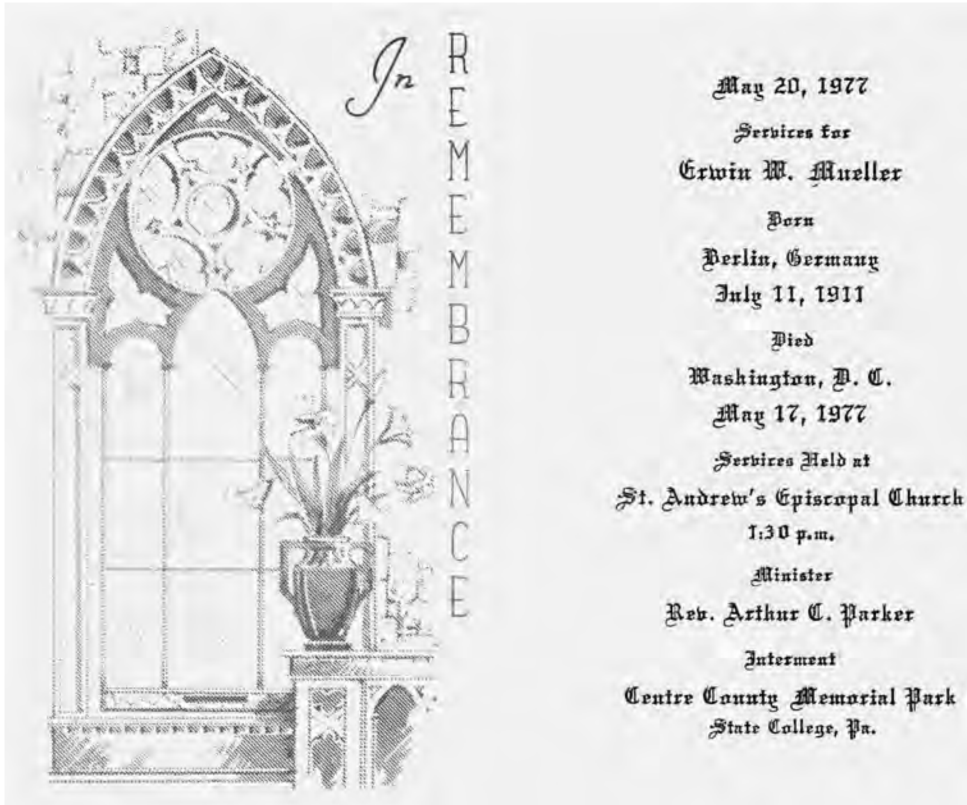

Figure 8. On May 20, 1977 funeral services were held for Erwin in State College, Pennsylvania.

In 1969 an "Aiming Error" was detected "If an image spot is placed over the probe hole, and a few atoms of the net plane edge are evaporated, no metal ion is ever detected. However, if the probe hole is placed approximately one image spot diameter from the selected image spot toward the center of the plane, a metal ion is recorded during essentially every pulse" (Panitz, 1969). This was due to different trajectories for the imaging gas ion formed about 4 Å above the surface and the metal ion from the surface.

Figure 9. Erwin Müller's gravestone at the Center County Memorial Park in State College, Pennsylvania.

Later in the same year the channel plate image intensifier was introduced with a gain of $\approx 10^3$ (Turner et al., 1969). It was an imaging revolution because this simple device could be placed within an FIM and the dark adaption required to see a faint FIM image was no longer necessary.

In 1970, I joined the Surface Physics Division (5114) at Sandia Laboratory in Albuquerque, New Mexico after a 1-year post-doctoral position at Penn State University overseeing Bruce Kendall's Ionosphere Research Lab, while he was on sabbatical. In 1973, I reinvented the atom-probe by incorporating a short drift distance and eliminating the probe hole by providing a desorption image of the entire surface (Panitz, 1973). In 1974 a "time-gating" feature was added allowing a preselected ion species to be chosen for imaging and its complete crystallographic distribution on the surface compared to an FIM image of the surface. The technique was called field desorption spectrometry (Panitz, 1974). In 1975 the field desorption spectrometer was patented, see Figure 7. Later the same year its moniker; the imaging atom-probe (IAP), was introduced by Waugh (1975). The IAP has been called the progenitor of atom-probe tomography (Seidman, 2007).

As I became recognized for the IAP, Erwin and I corresponded and, although when I first called him Erwin (rather than Professor Müller) his face blanched, I like to think we became colleagues, if not friends. On May 17, 1977, Erwin died from a heart attack while attending a National Academy of Science meeting in Washington, DC. Funeral services were held on May 20, 1977 in State College, Pennsylvania, see Figure 8. Note the spelling of Erwin's last name. The Mueller spelling appears because US typewriters at the time did not have an umlaut key! The most poignant memory I have of Erwin occurred in the Field Emission

Laboratory during the development of the prototype APFIM when Erwin remarked that his tombstone should read "Here lies a great idea in a rotten brain". That was not to be (see Figure 9), but it is how I like to remember this remarkable individual who defined my scientific career.

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