MICROSCOPY MILESTONES: FIELD ION MICROSCOPY, ATOM PROBE FIELD ION MICROSCOPY AND ATOM PROBE TOMOGRAPHY

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Two of the most significant microscopy milestones that were achieved in the last century were the imaging of individual atoms and the identification of individual atoms. Both these remarkable achievements were due to Prof. E. W. Müller and members of his group at Pennsylvania State University. Almost fifty years ago, Müller introduced a new type of microscope in which a sharp needle-shaped specimen was pointed at a fluorescent screen, Fig. 1.[1] By applying an appropriately high positive voltage to the specimen, image gas atoms near the apex of the specimen could be ionized and radially projected towards the screen where they produced highly magnified images of the specimen surface, Fig. 2. By cryogenically cooling the specimen and using helium as the image gas, the first images of individual atoms were obtained in a field ion microscope by Bahadur and Müller on October 11th, 1955. These researchers also discovered that by increasing the voltage on the specimen from that required to form the field ion image, the surface atoms could also be field evaporated from the specimen. Barofsky and Müller used this field evaporation process to identify the ions with a mass spectrometer. In 1968, Müller, Panitz and McLane incorporated a small aperture in the fluorescent screen and mounted the specimen on a sliding glass seal goniometer so that a specific atom on the surface could be selected and field evaporated with a high voltage pulse for identification in a time-of-flight mass spectrometer, Fig. 3.[2] This instrument was called an atom probe field ion microscope (APFIM). The usefulness of this type of instrument for high resolution metallurgical characterizations was immediately recognized and several atom probes were constructed in other laboratories around the world. In the following years, many improvements were made to the original design including the incorporation of image intensification, electrostatic lenses to provide energy compensation, high speed digital timing systems, and computer control. In addition, extensive data analysis software was developed. These improvements dramatically increased the number of atoms that could be collected, improved the mass resolving power of the instrument and transformed the atom probe into a routine metallurgical tool for the characterization of materials at the atomic level.

In 1972, Panitz at Sandia National Laboratory introduced a new variant of atom probe that was originally named the 10 cm atom probe and is now referred to as an imaging atom probe (IAP), Fig. 4.[3] This instrument eliminated the fluorescent screen with the probe aperture and featured a wide angle single-atom sensitive detector at the end of the time-of-flight mass spectrometer. By pulsing the specimen with a high voltage pulse and synchronously time gating the detector to image a selected element (or atom species), two-dimensional atom maps of a significantly larger area of the specimen surface could be obtained. As with the original atom probe, concentration profiles as a function of distance into the near surface regions of specimen could be obtained. In 1986, Miller at Oak Ridge National Laboratory introduced the concept of atom probe tomography (APT) and first prototype of a three-dimensional atom probe (3DAP), Fig. 5.[4] This instrument featured a position-sensitive detector at the end of the time-of-flight mass spectrometer. This wide angle detector enabled the impact positions of the ions to be determined. The 3DAP experimentally determines the spatial coordinates and elemental identities of the atoms in a small volume of material. These data may be rendered in three-dimensions on a computer to visualize the arrangement of solute atoms, Fig. 6. Since the introduction of this 3DAP prototype, several improved versions with different types of position-sensitive detectors have been developed. These instruments permit the atoms within a specimen volume to be analyzed. This volume typically contains up to ~1 million atoms and is ~10 to ~20 nm square by ~100 to ~250 nm deep. The type and spatial resolution of the information that is possible to obtain with the 3DAP has revolutionized the microanalytical characterization of materials.[5]

1. E. W. Müller, *Z. Physik*, 31 (1951) 136.
2. E. W. Müller, J. Panitz, S. B. McLane, *Rev. Sci. Instr.*, 39 (1968) 83.
3. J. A. Panitz, *Rev. Sci. Instrum.*, 44 (1973) 1034.
4. M. K. Miller, paper presented at the Microbeam Analysis Society, Meeting, Albuquerque, NM, 1986.
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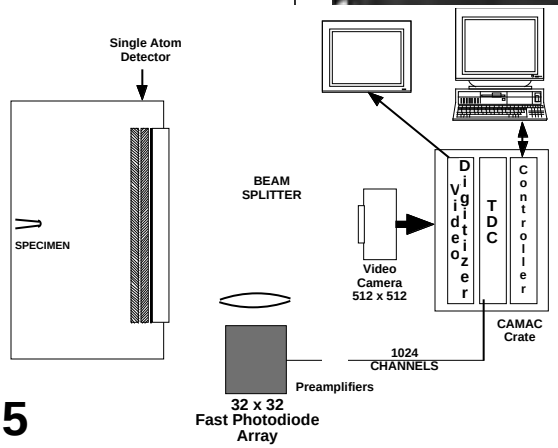

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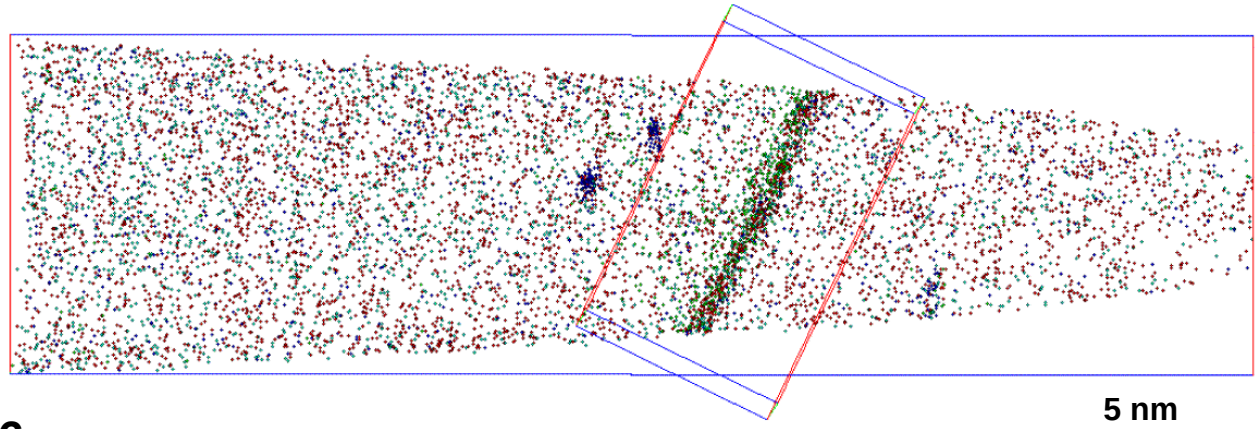

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FIG. 1. Prof. E. W. Müller with an early glass field ion microscope.
FIG. 2. Field ion micrograph of iridium.
FIG. 3. The original atom probe developed by Müller, Panitz and McLane. The field ion microscope section is at the top of the instrument and the single atom detector at the end of the mass spectrometer is at the bottom.
FIG. 4. The 10 cm atom probe (imaging atom probe) developed by Panitz.
FIG. 5. Schematic diagram of the prototype three-dimensional atom probe developed by Miller.
FIG. 6. 3DAP atom map of a lath boundary and 3 copper precipitates in a neutron irradiated pressure vessel steel.