Advances in 3D Atom probe: the LEAP 5000

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1. INTRODUCTION

Atom probe tomography (APT) is based on time of flight mass spectrometry of the individual atoms of a specimen prepared as a sharp needle (end radius ~100nm). The atoms are extracted and ionized with high efficiency in a DC electrical field of a few tens V/nm.

The APT was initially applied to a wide variety of metallurgical applications such as nuclear structural materials and combustion and steam turbine development, allowing one to image in 3D and quantify the composition with a subnanometer spatial resolution without the need of standards [1].

The recent combination of UV laser pulsing to initiate the evaporation of atoms, and FIB-SEM technology to cut, extract and shape specimens from selected areas of bulk materials (very similar to TEM sample preparation) has revolutionized the technique, opening its application to new fields, including not only site specific metallurgical applications, but also analysis of dielectric materials, oxides and ceramics, microelectronics, spintronics, photovoltaics, lightning, geology and cosmochemistry [2].

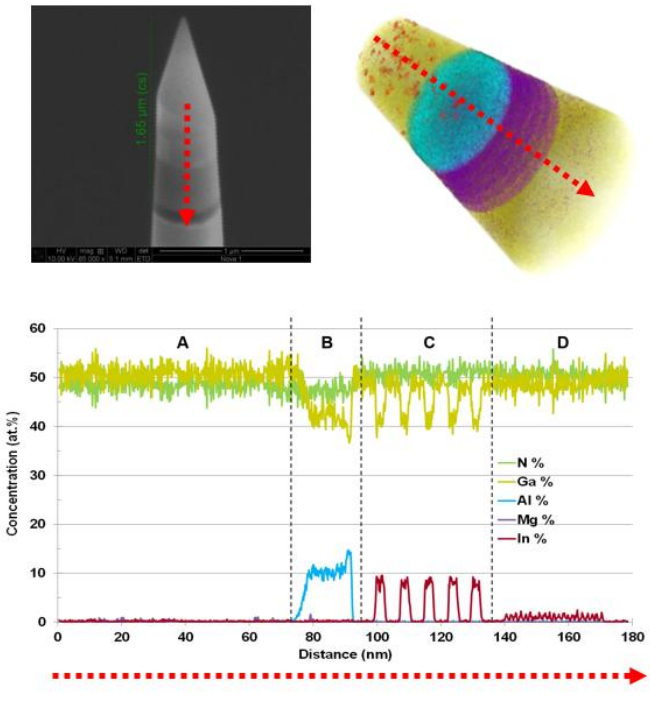

Figure 1. LEAP data of an OSRAM white LED including a composition profile from the p+ Mg doped region, through the Al-rich electron blocking layer into the active region containing a multi quantum well and an 11 period indium superlatice. [3,4]

2. THE NEW LEAP 5000

2.1 Origin

The LEAP 5000 is an evolution of the 3D atom probe; its unique design is based on the integration of a local electrode, a high frequency UV laser focused to a few µm spot size, and the choice between two types of flight mass analyzers (linear or energy-compensating reflectron).

2.2 Main instrumental advances

Among the new characteristics, three main instrumental improvements will be reviewed here:

1) APT being, by definition, a destructive technique, in order to characterize volumes of a few atoms only, the analyst relies on 100% ionization of all atoms and the highest analyzer transmission possible. The overall transmission has been improved from ~50% (on the former generation of atom probes) up to ~80% of atoms in the specimen for the new LEAP 5000 using a new detection design and new analysis algorithms. We will illustrate the direct benefit of this parameter through the detection of solute clusters of only a few tens atoms in nuclear nanostructured ferritic alloys.

2) One of the revolutions of the LEAP atom probe has been the increase of field of view *and* acquisition speed simultaneously. The LEAP 5000 is pushing this even further by increasing the repetition rate of the laser mode up to 1 MHz and of the high voltage mode up to 500 kHz. There are multiple benefits:

- a faster acquisition implies access to larger volumes in a short time: with the LEAP 5000 field evaporating up to 5 M ions per minute in laser mode, the analysis of a typical volume of 150 nm in diameter by 500nm in height is completed within a couple of hours. Positioning of rare objects in the specimen by FIB-SEM (example: a single given transistor) is greatly facilitated, and more statistics are available from numerous nano-objects (grain boundaries, clusters, etc.) inside large volumes.
- a lower mechanical stress on the specimen can be obtained by using a lower instantaneous field evaporation flux while keeping reasonable acquisition speed through a high repetition rate. Lower stress together with a more sophisticated real-time field evaporation regulation (new electronics & software) results in a higher success rate, pushing the LEAP 5000 atom probe nearer a failure analysis technique.
- In voltage mode used for metal analysis, a higher voltage pulse fraction (i.e. 30% of the DC voltage) together with a higher frequency (up to 500 kHz) produce an improved signal to noise ratio (less atom evaporation between pulses), an even evaporation probability for all atoms, and access to larger volumes in a given time.

3) the ion optics of the two available time of flight mass analyzers and detectors have been redesigned. This produces a constant field of view while the specimen radius is growing during the analysis, a uniform illumination of the detector area, and a more homogeneous mass resolving power over the full detector area.

3. CONCLUSION

The newest generation of atom probe tomography, the LEAP 5000, is incorporating new instrumental features improving sensitivity, quality of data, speed of acquisition and yield of success. It further advances the atom probe from a complicated technique limited to a few metallurgists to a widespread quantitative analysis technique for 3D, compositional measurement at the nanoscale in a very large and still growing number of applications: metallurgy, ceramics, glasses, minerals, catalysis, nanoparticles and nanowires, spintronic, LEDs, quantum dots, semiconductors, microelectronics, failure analysis, photovoltaic, and bio-mineralization.

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