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Data Quality Improvements in the Voltage-Pulsed LEAP 5000 R/XR

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Performance advances in atom probe tomography (APT) in recent years have driven a dramatic increase in the number of APT articles published. One area of rapid research growth is analysis of fragile or insulating materials that require a laser-pulsed atom probe [1]. However, for multi-user facilities around the world, voltage pulsing is still used regularly (averaging ~40% of the experiments), and at some facilities voltage-pulsed analyses comprise greater than 60% of the experiments [2].

Local electrode atom probe (LEAP®) systems have provided substantial improvements in ease of use, speed, field of view (FOV), mass resolving power, imaging quality and sample preparation flexibility over previous generation TAP and 3DAP instruments. However, the voltage pulse performance has been somewhat limited by the maximum pulse rate (200 kHz) and pulse amplitude (1300-1700 V) in the earlier generation LEAP 3000 and 4000 systems. The pulse amplitude limitation restricted the ability to use the LEAP to its maximum specimen voltage and FOV because the necessary voltage pulse fraction (15-25% depending up on the material and run conditions) to assure a consistent measurement of composition through the entire experiment [3] could only be maintained over a fraction of the available specimen voltage. Figure 1 demonstrates situations where the pulse voltage is insufficient to maintain compositional accuracy [1]. Figure 2 illustrates the extended range of constant pulse fraction for the new LEAP 5000 R/XR systems over the previous systems (now 15% pulse fraction up to 15kV).

Although maximum data collection rates increased dramatically with the introduction of the first LEAP system in 2003, data acquisition times can still be significant (several hours or more) for complex specimens with large regions of interest. To improve specimen throughput, the maximum pulse rate has increased in the LEAP 5000 X/XR by 250% to 500 kHz. In addition, adaptive pulse frequency controls (available in both laser and voltage modes) have been developed to allow selection of a constant mass range during the entire experiment. In this mode, the pulse frequency is automatically increased as the specimen voltage increases and ion flight times decrease, which further improves throughput by as much as a factor of two.

An increased pulse rate can also result in improved signal-to-noise ratio (SNR) due to a higher duty cycle of pulsing, thus more signal relative to background per unit time if the background noise is uncorrelated with signal peaks. Some samples have long spectral tails following the main signal peak and these tails can extend into the TOF window associated with the next voltage pulse thus increasing the background noise level. Figure 3 demonstrates that, even on a log scale, mass spectra can appear to have a flat SNR by simple examination, but upon closer inspection can have long, exponential components.

Fortunately, the application of a high pulse fraction can assure that evaporation only occurs in association with the very sharp peak of the voltage pulse, suppressing correlated evaporation. Figure 4 demonstrates that the SNR rise with increased frequency can be nearly eliminated when using a large pulse fraction. The advanced voltage pulsing capabilities of the LEAP 5000 R/XR systems allow simultaneous improved throughput and improved detection sensitivity for low concentration species [4].

References:

[1] D.J. Larson *et al*., "Local Electrode Atom Probe Tomography" (Springer, New York 2014).
[2] Unpublished customer survey, CAMECA Instruments Inc., (2015).
[3] Miller, M. K. & G.D.W. Smith, Vac. Sci. and Tech. **19**, (1981), p. 57.
[4] R.M. Ulfig *et al*, Microscopy and Microanalysis **19** S2, (2013), p. 986.
[5] The authors would like to thank Montanuniversität Leoben, University of Sydney, Oxford, Alabama, and the University of California Santa Barbara for sharing their pulse mode statistics.

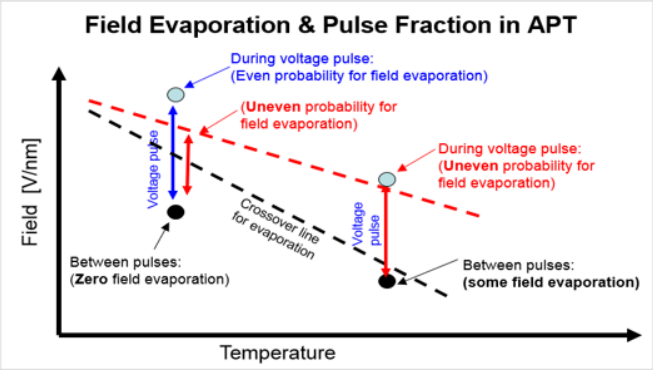

Figure 1. Schematic relationship between temperature and evaporation field for a multi component sample. At any base temperature, a sufficient magnitude pulse is required all species to have a similar probability of evaporation.

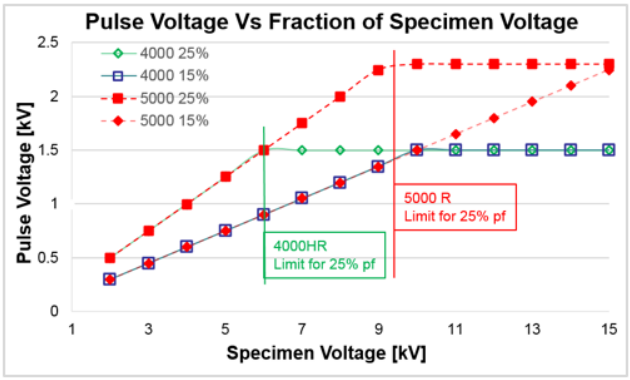

Figure 2. The extended range of operation for a constant pulse fraction for the LEAP 5000 to a maximum pulse voltage of up to 2350V (40% increase).

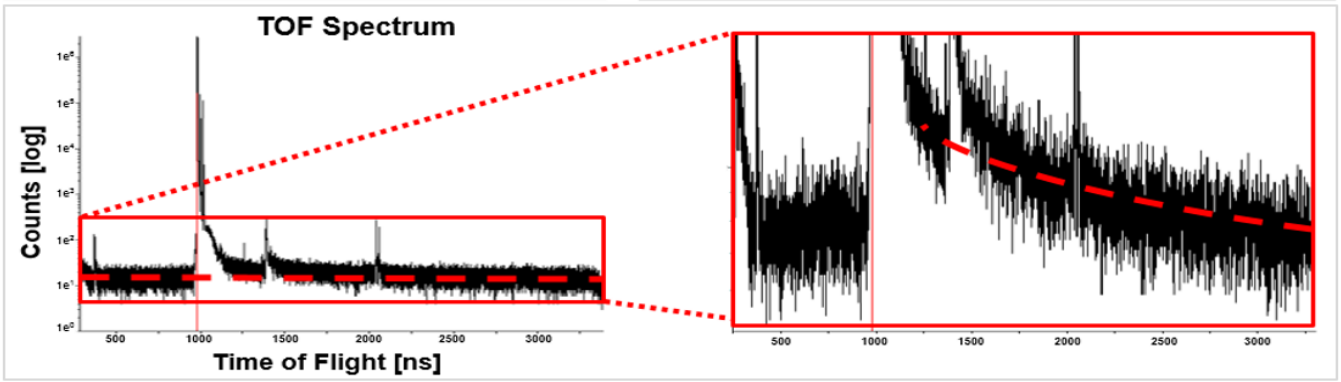

Figure 3. Although the background noise for a Sb-doped silicon sample appears to be flat in time, an expanded vertical axis demonstrates that the noise is correlated with the main peaks and may contribute to noise in the adjacent pulse window.

Figure 4. Higher voltage pulse fraction (30%) results in a significantly reduced noise floor, even with increasing pulse frequency. This enables higher sensitivity to low concentration species, down to ppm levels.