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Understanding Conditions Affecting Background in Atom Probe Tomography with Implications for Analysis of Hydrogen

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The ultimate analytical sensitivity of any mass spectrometry technique is strongly affected by the presence of background in the detected signal [1]. While methods and strategies exist for dealing with and correcting for this artefact [2], minimizing background contributions will always improve analytical precision. In atom probe tomography (APT), some spectral background originates from the pulsed ionization of residual gasses (in-time peaks in the mass spectrum) as well as random evaporation between pulses (out-of-time), while another background component is ionization events from sample atoms that occur between pulses. Optimizing APT sensitivity requires understanding the variation of each of these background components within the available analysis condition ranges.

Microstructural characterization of light elements is a challenge for many microscopy techniques because low atomic number elements interact weakly with electrons and x-rays. This is very inconvenient because hydrogen, as one example, is responsible for reducing the service life of many technologically important metallic components through enhanced creep and embrittlement, and so characterizing the phenomenology of hydrogen in metals is quite important [3]. Fortunately, APT is a near-atomic-scale analytical technique whose chemical sensitivity is independent of atomic number, so it has potential for characterization of hydrogen in material systems. Unfortunately, typical ultra-high-vacuum APT environments contain sufficient hydrogen that interpreting the material signal through the residual gas background has significant uncertainties. Some investigators have avoided this complication by working with deuterated samples where interferences with the H_2^+ ion background peak is significantly less pronounced [4].

In this study, we have investigated how analysis conditions affect the presence of background observed for standard APT specimens, which includes <100> silicon and <100> nickel. The availability of different materials allows us to more fully consider evaporation field dependence. We find that background trends are difficult to interpret unless they are expressed as ion flux-density-rates (or "flux" in ions/s/nm^2). Fig. 1 shows a relatively constant background hydrogen flux emission, independent of acquisition flux (i.e. the number of background H^+ ions per area, per time only depends on vacuum and not sample ionization rate). Results for various in-time background peaks and out-of-time evaporation levels in laser-pulsed mode are also shown in Fig. 2. Here a pre-sharpened microtip [2] was analysed under constant evaporation flux and charge-state-ratio (or evaporation field) conditions. The various background trends are reproducible and similar to those reported in the historical literature [5].

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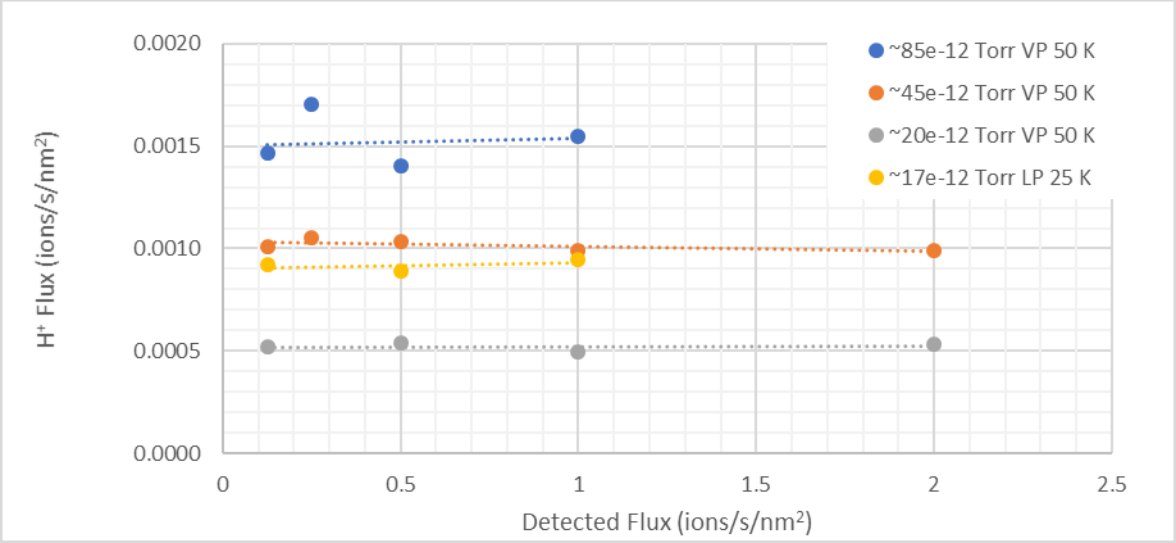

Figure 1. Intensity of the hydrogen peak emitted from a silicon <100> sample at various vacuum levels, and under both voltage-pulsed (VP) and laser-pulsed (LP) conditions. The hydrogen emission is relatively constant for a range of specimen ionization voltages (specimen sizes) and analysis rates (here reported as detected flux).

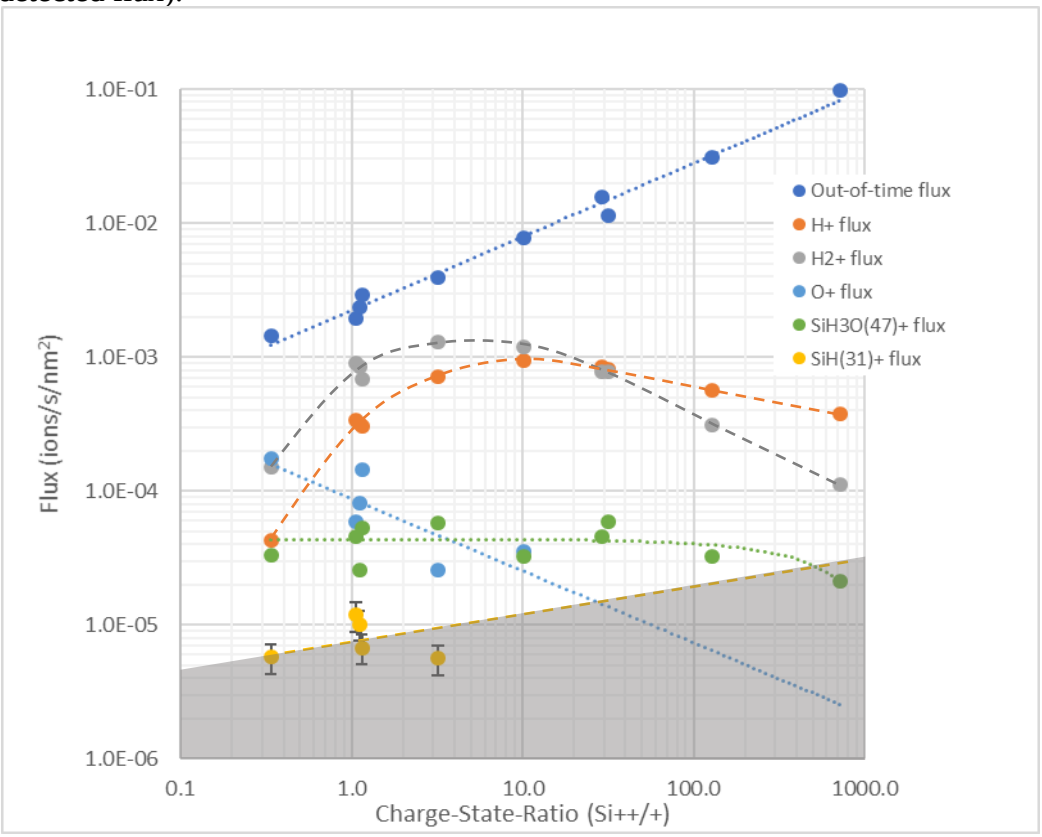

Figure 2. Background levels observed from a silicon <100> sample at various constant charge-state-ratio (constant field) conditions. Total detected flux was held at 0.5 ions/s/nm^2 for all measurements. The hydrogen background signal is observed to increase and then plateau at higher field conditions while the out-of-time background increases with field. The detection limit is approximated by the gray region.

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