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Atom Probe Tomography allows to map routinely 3D composition of a specimen at the near atomic scale for an increasingly wide range of material systems. In APT, the information is composed to the list of coordinates of analyzed atoms in the probed volume, with elemental information get from time-of-flight mass spectrometry. This means that no real local chemical information (local chemical bounding for instance) is usually extracted from the tomographic data. To access partially this crucial information, intensive post-treatments of the tomographic data are necessary, with a coupling of APT dataset to simulations models [1, 2].

APT data have been indirectly mined in the past looking at the mechanism of field evaporation of surface atoms [3, 4]. Field evaporation is the fundamental mechanism involved to peel off the sample atom by atom. It is known for decades that field evaporation showed some differences from one sample to another related to the atomic local environment, which could help to explain their properties [4, 5]. However, this information remained very indirect and difficult to address. In a more recent study, we have shown how energetic considerations in the field evaporation process are in fact intrinsically present within the data, but have until now been ignored. Mass spectra shapes using voltage pulse analyses are indeed related to the activation energy required by the field evaporation mechanism that depends on the material and analysis temperature, allowing us to extract local energetic information. We demonstrate how this information can be mapped in 1D, 2D and 3D dimensions with nanometric resolution in a few experimental cases. We term this approach "Field Evaporation Energy Loss Spectroscopy". FEELS can be adapted to any straight flight path atom probe instrument using voltage pulses, and can even be used on the thousands of existing data sets to retrieve further information about the sampled material.

In this paper, we show that this information reveals fine scale energetic considerations in the field evaporation process. This information was in fact intrinsically present within the data, but have until now been ignored or even suppressed using ion optics devices. Mass spectra shapes are related to the activation energy required by the field evaporation mechanism that depends on the material and analysis temperature, allowing us to extract local energetic information in pure metals with nanometer resolution. An example in a pure Al dataset (Figure 1) is presented. The field evaporation activation energy depends on the local crystallography of the specimen and is universal to the material nature and atomic organization.
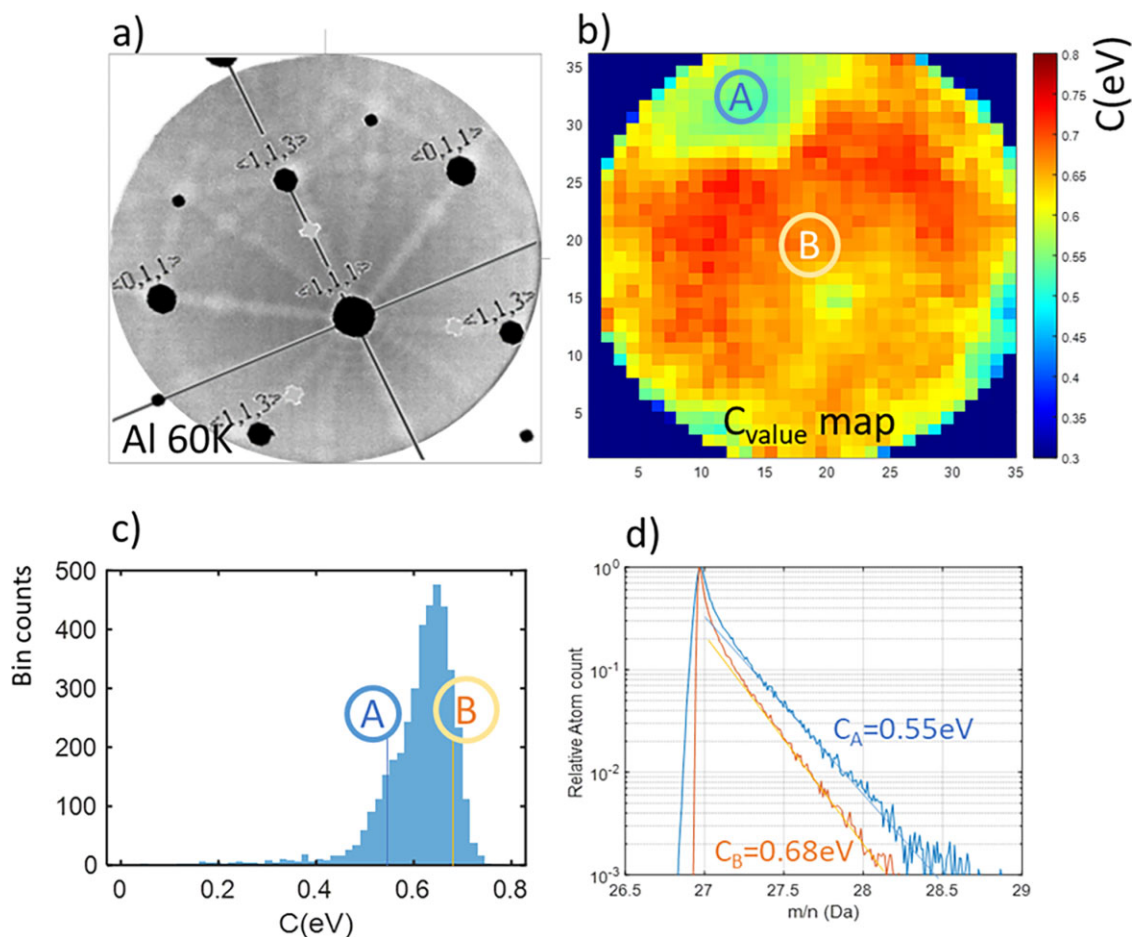
Fig. 1. a) Detector Hitmap (each dot is a detected Al atom) for an analysis of an Al sample at 60K using a LEAP 5000 in Voltage pulse mode (15% pulse fraction). b) Measurement on a detector of the C-value (slope of the Q(F/Fe) function, with Q~C(1-F/Fe) the field evaporation activation energy assuming a linear model), color scale indicates C constant in eV. c) Histogram of the values found in b) showing the presence of a multimodal distribution. Each count is an individual bin in this 35x35 binned map. D) local mass spectra from region A and B of b), showing the different mass tails enabling the calculation of the C-values.

References

1. M.P. Moody *et al.*, *Nat. Commun.* **5** (2014) 5501.
2. A. Prakash, *et al.*, *Acta Materialia* **92** (2015) 33–45
3. M.K. Miller, A. Cerezo, M.G. Hetherington and G.D.W Smith, *Atom-Probe Field Microscopy*. Clarendon Press, Oxford (1996)
4. Williams Lefebvre-Ulrikson, François Vurpillot and Xavier Sauvage, *Atom Probe Tomography. Put Theory Into Practice*. Academic Press 2016.
5. T. Boll, and T. Al-Kassab, *Ultramicroscopy* **124**, 1–5 (2013). doi:https://doi.org/10.1016/j.ultramic.2012.09.003.
6. M. Zhu *et al.*, *Advanced Materials* **30**(18), e1706, 735 (2018). doi:https://doi.org/10.1002/adma.201706735