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Point-by-point compositional analysis for atom probe tomography

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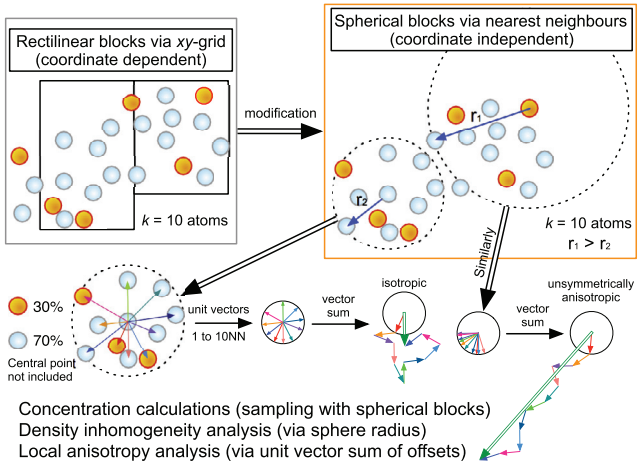
GRAPHICAL ABSTRACT

GRAPHICAL ABSTRACT FOR POINT-BY-POINT COMPOSITION ANALYSIS



ABSTRACT

This new alternate approach to data processing for analyses that traditionally employed grid-based counting methods is necessary because it removes a user-imposed coordinate system that not only limits an analysis but

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also may introduce errors. We have modified the widely used "binomial" analysis for APT data by replacing grid-based counting with coordinate-independent nearest neighbour identification, improving the measurements and the statistics obtained, allowing quantitative analysis of smaller datasets, and datasets from non-dilute solid solutions. It also allows better visualisation of compositional fluctuations in the data. Our modifications include:.

- using spherical *k*-atom blocks identified by each detected atom's first *k* nearest neighbours.
- 3D data visualisation of block composition and nearest neighbour anisotropy.
- using *z*-statistics to directly compare experimental and expected composition curves.

Similar modifications may be made to other grid-based counting analyses (contingency table, Langer-Bar-on-Miller, sinusoidal model) and could be instrumental in developing novel data visualisation options.

ARTICLE INFO
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Method details

Preparation of material

We demonstrated the new protocol using an atom-probe analysis of a Ni-based super-alloy [1]. In the broader study [2], the Al and Cr segregation is being investigated for its possible association with the nucleation of the γ′-phase precipitates within the matrix γ-phase. The material was processed in the following manner.

- As-cast Ni–8Al–8Cr at.%.
- Solution treatment 1150 °C for 30 min and quenched in liquid nitrogen.
- A further heat treatment at 600 °C for 5 min.
- The atom probe sample was prepared with FEI Nova Nanolab 200 SEM/FIB system.
- 10^6 atoms were detected using laser-assisted Cameca LEAP 3000XHR.

The operational parameters of the LEAP were a set temperature of 45 K with a pulse rate of 160 kHz and a target evaporation rate of 5 ions per 1000 pulses.

Construction of spherical blocks

The "binomial analysis" is widely used and provides a relatively rapid test for the presence of non-random compositional fluctuations [3]. The original protocol did this by dividing data into rectilinear *k*-atom blocks, aggregating solute contributions from each block and comparing the one-dimensional compositional histograms with binomial predictions. Later modifications to this protocol achieved many improvements [4], notably by rescaling the imposed (*x*,*y*)-grid to produce *k*-atom blocks that are, on average, more cubic ($z \approx x = y$). Highly anisotropic blocks were discounted from the analysis.

Our modifications to the protocol replace the rectilinear *k*-atom blocks with spherical *k*-atom blocks. The spherical blocks increase in radius as *k* is increased. This approach is similar to an earlier protocol to the calculations of atomic concentrations made on the atomic scale (concerning nearest neighbour shells) [5].

The user must select the parameter *k* while being mindful of the minimum size of nanostructural features that can be reliably discerned with this *k*-atom block. Features smaller that these blocks would be smeared with surrounding matrix. Reducing the value of *k* can increase spatial sensitivity but if *k* is too small the local concentrations are computed with smaller samples leading to larger measurement errors.

For each atom, an in-house produced algorithm (employed for earlier studies [6]) was used to perform the following steps.