**Large area mapping for analysis of nanoscale carbonitride precipitates within the steel matrix**

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In recent work, we have shown that it is possible to measure the chemistry of carbonitride precipitates, a few nm in size, with high accuracy and precision while they remain within the steel matrix [1]. However, such work is limited to small numbers of precipitates because of the detail required in the analysis. In the analysis of dispersion-hardened alloys, it is critical to understand the size and spatial distribution of the precipitates, as well as their number density to seek links between composition, processing, precipitation and properties. Whilst existing techniques, such as carbon replica extraction, provide some information, they also have huge problems, specifically:

- the exact depth from which the precipitates are extracted is not known, so number densities cannot be accurately determined;
- the relationship between the precipitates and the microstructure is lost;
- precipitates can move during extraction, so the 3D distribution is lost;
- small precipitates may be dissolved, or their outer layers lost, resulting in incorrect size distributions.

This work shows major progress towards a new way of mapping precipitates in steels over much larger volumes using DualEELS in the scanning transmission electron microscope. Specifically, we record low- loss maps over large areas of FIB cross-sections of the steel, and then fit the spectra using standards from well understood binary carbides and nitrides, together with standards for the matrix, to extract large area maps e.g. Figure 1. Image analysis can be used to find particle size distributions. Additionally, the sample thickness is measured from the same dataset, allowing results to be expressed per unit volume of matrix e.g. Figure 2. This method can also be used to rapidly survey areas for subsequent more detailed compositional mapping of precipitates using the methods developed previously. Examples will be shown including cases where larger precipitates are found along a grain boundary, together with smaller precipitates of different composition nucleated within a grain in a bainitic steel.

While in this case the method has been applied to study precipitation in steels, it can be extended to other systems where the particles and matrix have different compositions, such as catalytic nanoparticles on a support.

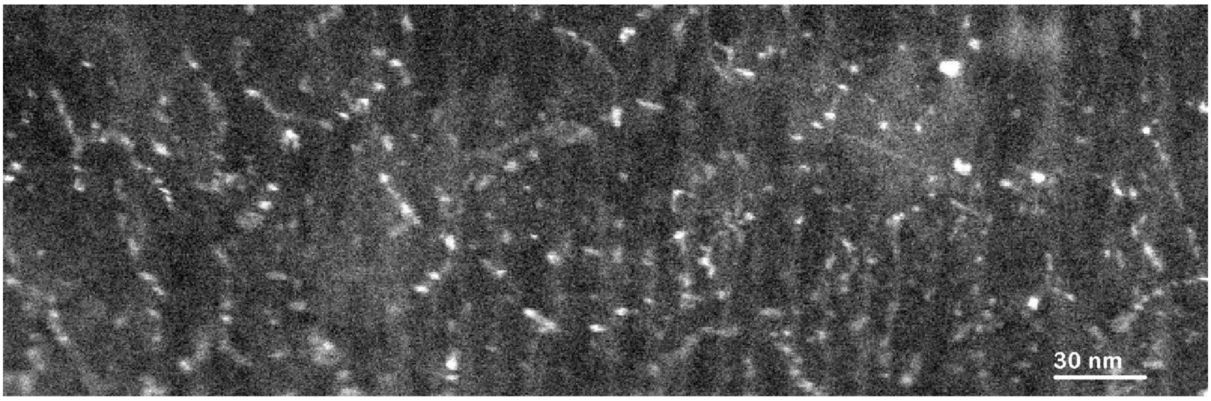


**Figure 1**: *Large area map of an area of the steel sample, showing a large number of small precipitates within the matrix and their clear connection to the microstructure*

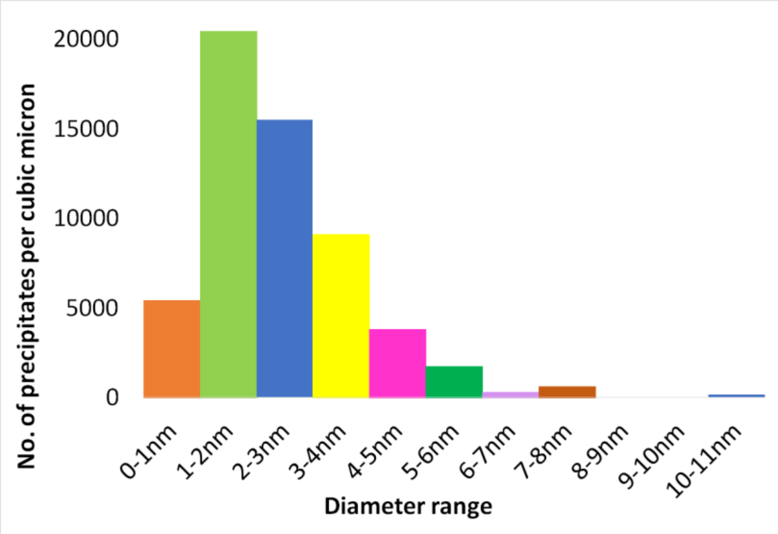


**Figure 2**: *Number of precipitates per unit volume within each diameter range*

[1] A J Craven, B Sala, J Bobynko, I MacLaren, Spectrum imaging of complex nanostructures using DualEELS: II. Absolute quantification using standards, *Ultramicroscopy* **186** (2018) 66-81

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