Absolute chemical analysis of nanoscale carbide precipitates within steel matrices

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Using DualEELS, we have accurately quantified nanoscale carbonitride precipitates within a steel matrix. To make this possible, a two-step method has been developed. This is applied after the initial processing from the as-acquired dataset to a deconvolved spectrum image, as described by Bobynko *et al.* [1]. For the first step, binary standards of well-known compositions are needed to obtain cross-sections for use in the quantification. In the second step, these cross-sections are then used to accurately quantify the composition of the precipitate using a multiple linear least squares fitting approach without using background subtraction as far as possible. This is preferred over the traditional approach (i.e. subtracting backgrounds and integrating counts under each edge) as the closely spaced edges affect the background fitting.

DualEELS datasets were recorded from carbide precipitates inside a high manganese steel matrix using a JEOL ARM200cFEG electron microscope equipped with a GIF Quantum ER spectrometer, and were processed by the method described above [1]. Titanium, vanadium, carbon and nitrogen cross-sections were determined from highly pure binary carbides and nitrides using a least squares fitting technique [2]. These cross-sections were used in combination with local matrix shapes to quantify the precipitate composition using multiple linear least squares fitting of the spectrum from before the C-K edge until after the Fe-$L_{2,3}$ edge.

This method allows for accurate compositional analysis of precipitates down to 6nm in size embedded in a matrix of up to 40nm thickness. The estimated thickness sensitivity is in the range of 1 to 2 unit cells at this matrix thickness [3]. While we have applied this method to a specific material system, the approach we developed can be of use in many other systems where two or more chemically distinct phases are present.

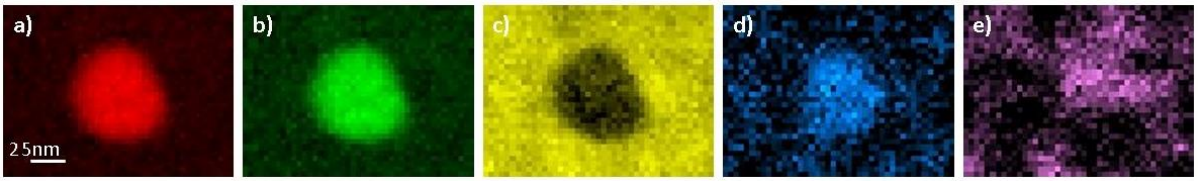

Figure 1: *Plots of coefficients in the MLLS fit a) TiC, b) VC, c) C from VC, d) N from VN and e) a-C.*

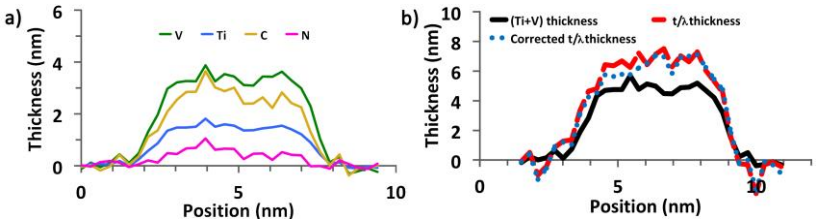

Figure 2: *Line profile plots of a) equivalent thickness for each element; b) total (Ti+V) thickness compared to that calculated from t/λ, with and without a correction for a-C.*

[1] J Bobynko, I MacLaren, A J Craven, Spectrum imaging of complex nanostructures using DualEELS: I. digital extraction replicas, *Ultramicroscopy* **149** (2015) 9-20

[2] A J Craven, J Bobynko, B Sala, MacLaren, Accurate measurement of absolute experimental inelastic mean free paths and EELS differential cross-sections, *Ultramicroscopy* **170** (2016) 113-127

[3] 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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