The power of speed: DED EELS for analytical *in situ* TEM

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Introduction

In situ (S)TEM can provide detailed insight into dynamic processes in materials subjected to stimuli. However, a key condition for the use of a structure with dimensions in the micro- and nanometer range to illustrate processes in a bulk system is the exact knowledge of the local composition. Consequently, concomitant analytical analysis that is fast enough to observe changes in the chemical details of the sample during the experiment is extremely valuable for the reliable interpretation of *in situ* TEM results.

In this study, we illustrate the benefits of Direct Electron Detection EELS (DED EELS) to perform 2D chemical analysis during an STEM heating experiment and compare them to EDXS and EELS using a common CCD. The material that is described is AlCu4, an aluminum alloy with 4% of copper that can be strengthened by precipitation hardening [1,2]. We capture and describe the changes of the system during different stages of precipitate formation, using fast EELS techniques to track the chemical composition of the alloy system.

EDXS (SuperX on Titan³ G2 @300kV)

EDXS combines well with EELS for concomitant analysis of the process, especially for dynamic experiments that require thick samples e.g. for monitoring growth processes. Overall, it does not provide enough speed to be used to map fast processes, and the signal quality is susceptible to temperature changes [3].

+ Covers whole range of elements
+ Well suited also for thicker specimen
– Slow, with low signal yield with *in situ* double tilt holder
– Signal varies with tilt of holder (in dependence of the detector system)
– Signal quality is dependent on temperature
– Quantification for systems containing light elements is not very reliable

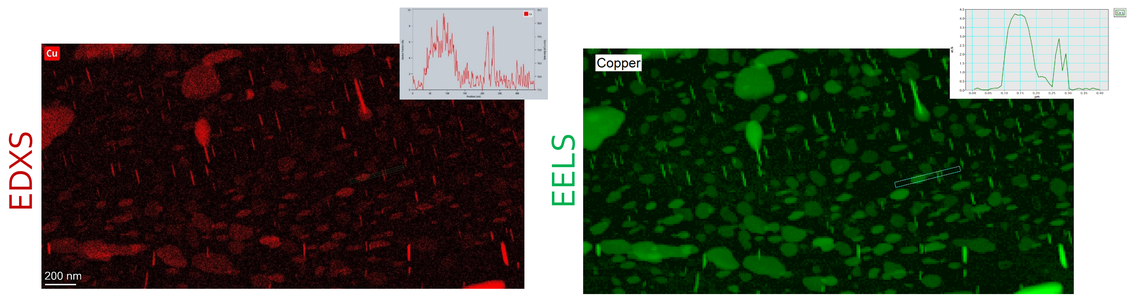

Figure 1: Comparison of Cu elemental maps from 90 min EDXs map (red) and 30 min DED EELS map (green). The SNR is evidently far better for the EELS map (see also profiles), although the recording time was three times lower.

EELS (Quantum ERS GIF on Titan³ G2 @300kV)

For EELS, we took into account that Direct Electron Detection EELS systems feature superior benefits in terms of speed when compared to systems with a CCD camera, in particular when using denoising techniques.

+ fast/high signal quality for high scanning speed especially for DED signals with PCA denoising
+ No dependence on tilt and temperature
+ Signals can provide additional information (ELNES, plasmons)
– Only for a pre-determined range of elements
– Preferably for light elements, due to decay of signal for large energy losses
– Decay in SNR for thick samples

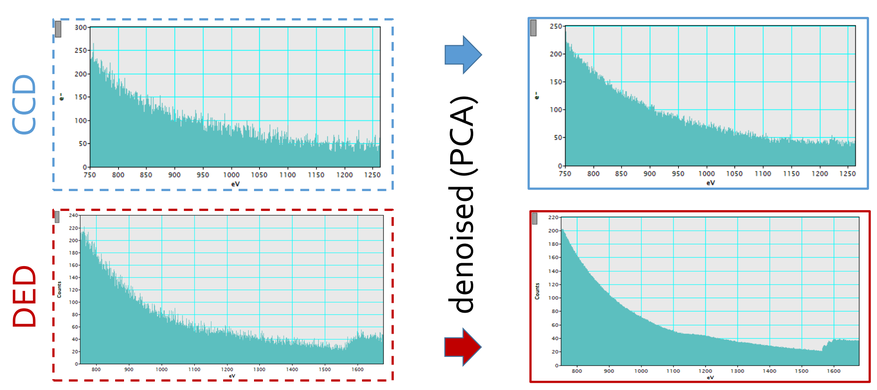

Figure 2: Comparison of CCD and DED single EELS spectra of the energy range of interest (energy loss > 800eV) captured at the same sample position with identical settings (pixel time 50ms, sample thickness was ~170 nm)

DED EELS: Providing speed for dynamic processes

Direct Electron Detection EELS systems like the Quantum ERS GIF with K2 camera (Gatan) used for these experiments feature suitable sensitivity and speed to deliver 2D elemental maps with sufficient signal quality even during *in situ* TEM experiements. They are fast enough to map dynamic processes, providing detailed insight in compositional changes, in this case triggered by a heat treatment with a DENSsolutions DT Wildfire holder [4].

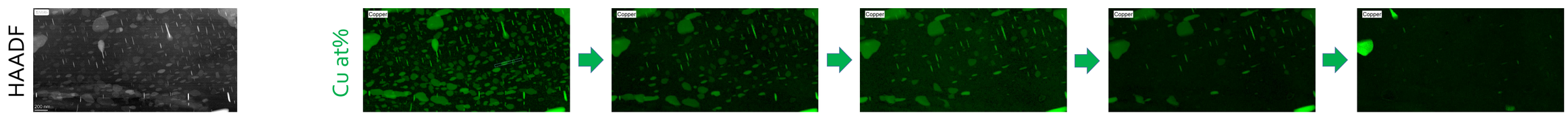

Figure 3: Evolution of the copper-containing precipitates during heat treatment illustrated by a series of elemental DED EELS maps showing the decomposition of the Guinier-Preston zones II [3] while heating from 350 to 410°C (map size 280x140 pixels, pixel time 50 ms, overall mapping time 30 min, HAADF of first map on the far left for comparison).

Conclusion

Using analytical signals during *in situ* TEM experiments is an essential factor in understanding the dynamic processes in a changing sample. Especially when compared to EDXS analysis, which is more suited for complementary overview evaluation, Direct Electron Detections EELS is the most powerful choice in terms of speed, providing the capability to perform detailed chemical analysis of a complex light element system at various stages of transformation.

References/ Literature

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[2] Zandbergen et al., Nature Scientific Reports 7 (2017) 2184

[3] R. Krisper et al., Ultramicroscopy 234 (2022) 113461

[4] https://denssolutions.com/products/wildfire

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