Experiment Brief

GIF Continuum with direct detection & drift correction

Title

Achieving ~1 Å resolution in $Tb_3Sc_2Al_3O_{12}$ STEM EELS mapping with GIF Continuum K3

Gatan instruments used

GIF Continuum® K3®

Background

Electron energy loss spectroscopy (EELS) spectrum imaging (SI) combined with aberration-corrected STEM enables the simultaneous characterization of structural and chemical changes at the angstrom scale. However, long acquisition times, sample drift, and beam-induced sample damage make atomically resolved EELS SI challenging relative to annular dark-field (ADF) STEM imaging. Direct detection single electron counting cameras improve the detection limit for ionization edges in EELS spectra due to their high sensitivity. This allows SI data to be collected at significantly reduced total dose, reducing the acquisition time necessary for high-spatial resolution data capture. Multi-pass SI collection enabled via Gatan's eaSI™ technology and DigitalMicrograph®, combined with the high spectral rate of the K3 camera fractionates the total dose over several passes, allowing the user to perform drift correction at a high enough frequency to avoid image distortion. Furthermore, the single electron sensitivity of the K3 camera prevents read-noise from compromising the spectra's signal-to-noise ratio (SNR) for sub-millisecond dwell times.

Materials and methods

EELS SI were acquired from a terbium scandium aluminum garnet ($Tb_3Sc_2Al_3O_{12}$) focused ion beam (FIB) prepared lamella, using a GIF Continuum K3 installed on a Thermo Fisher Scientific Spectra 300. All data was acquired at 300 kV with a probe current of 37 pA and EELS dispersion 0.9 eV/ch. This gave us a single spectral range >3,000 eV, enabling simultaneous acquisition of ionization edges from every element in the sample. Spectrum images were acquired at 3,000 spectra/s (~0.34 ms/spectra), with a step size of 0.2 Å/pixel. Each pass of the SI took ~3.4 s (100 x 100-pixel array), and a total of 50 passes were acquired, resulting in an effective acquisition time of ~170 s. Drift correction was applied every two passes. All data was processed using the built-in tools in DigitalMicrograph, allowing derivation of atomically resolved elemental maps as shown in Figure 1(a – d). Sc, Tb, and O in the overlaid image (Figure 1f) sit exactly in their respective positions predicted by the structure model (Figure 1g). The Tb trimer with a projected distance of ~1.2 Å (Figure 1e) is resolved (Figures 1b and 1f), indicating the high spatial resolution of the elemental map. Although the Al map generally corresponds to the Al atoms' location in the lattice, the precise location is not resolved. This is due to the strong overlap of the Al K and Tb edges, decreasing the signal- to- background ratio of the Al K edge and increasing the uncertainty in the fit of the cross-section model during quantification.

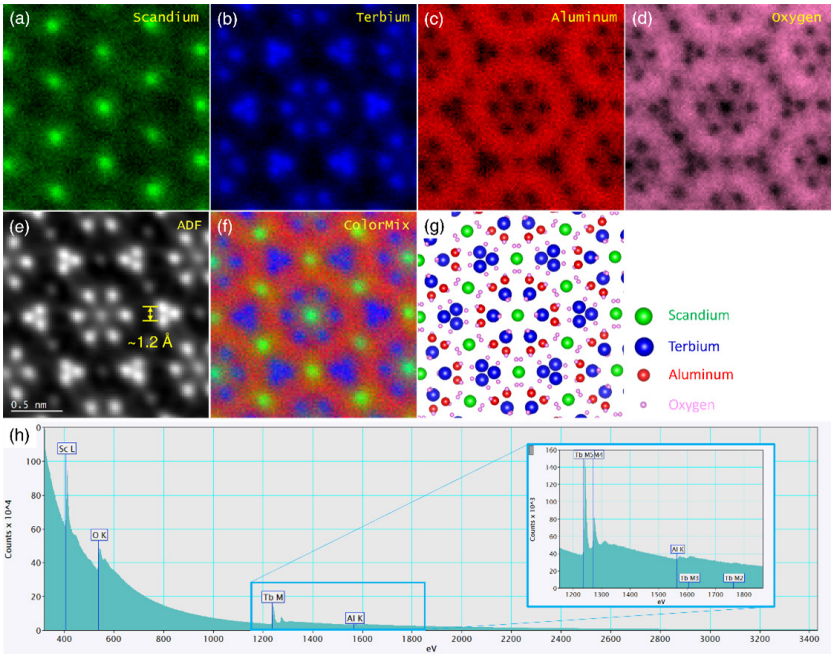

Figure 1. Elemental maps were generated from the a) Sc L, b) Tb M, c) Al K, and d) O K edges, and e) the simultaneously collected ADF image. f) The individual elemental maps were overlaid to create a composite map. g) The atomic positions correspond with the structural model. h) A plot of the average EELS spectrum extracted from the SI data.

Summary

We demonstrate that crystal lattices can be precisely mapped at ~1 Å level spatial resolution within minutes using the GIF Continuum K3. The high sensitivity and speed of the K3 camera, the remarkably low noise level of counting EELS, and the multi-pass SI function in DigitalMicrograph allow users to collect high SNR distortion-free EELS SI data with reduced total dose. The built-in analytical tools further simplify the procedure toward meaningful results.

Credit(s)

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