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Novel Nanoscale Tomography Modes in Materials Science

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Electron tomography of crystalline, inorganic, and heavy element containing nanomaterials is complicated by nonlinear, possibly non-monotonous, intensity relation during image formation, strong dependence on lattice plane orientation and potentially extreme exposure requirements. Very recent break-through results have been achieved in this field using new modes of image formation available in modern TEMs, comprising bright field, CTEM, STEM, HAADF, EFTEM, and EDX-mapping [1-5]. Three major groups of contrast seem to be most favourable (FIG. 1), sketched for two concentric cylinders of elements A and B (Z_A and Z_B) :

- binary tomography: $I_0 = 0, I_1 = I_2 = 1$ (1)
- Z-Contrast imaging: $I_0 = 0, I_1 = t_A(x,y) * Z_A^m, I_2 = t_A(x,y)*Z_A^m + t_B(x,y)*Z_B^m$ (2)
- Spectroscopic tomography: - tuned to (A): $I_0 = 0, I_1 = I_2 = t_A(x,y)*\sigma_A$ (3)
  - tuned to (B): $I_0 = 0, I_1 = 0, I_2 = t_B(x,y)*\sigma_B$ (4)

Here, $t(x,y)$ is thickness, σ an inelastic cross section, and m the atomic number exponent ($m<2$). In this simple picture, longitudinal coherence is neglected, and spectroscopic images are assumed processed into elemental maps (multi-window EFTEM). While HAADF-STEM is the most successful and universal "Z-contrast", other mechanisms such as weak-phase-object (WPO)-HREM, and various special cases of BF/DF-CTEM, e.g. high-angle hollow cone [1] , also classify as Z-contrast as of eq. (2). FIG. 1e sketches the topological class of objects which can be solved by binary tomography: Homogeneous phases ($\rho(x,y,z) = \text{const.}$) of strictly convex shape on all cross-sections perpendicular to the backprojection axis. Inversely, this is the class which can be sculptured by parallel beam tools (FIB, Laser cutter) from a rotating block. FIG. 2 shows experimental results obtained for spectroscopic tomography over $\pm 60^O$ tilt, using EFTEM/ESI, and $\pm 50^O$ EDX-mapping (step 10^O), as the imaging/ projection mechanisms [6, 7]. In FIG. 3, simulations demonstrate the theoretical capabilities of WPO-HREM at atomic resolution (ideal Si-crystal, cut into tip shape).

Outlook: The importance of spectroscopic tomography lies in eliminating thickness $t(x,y)$ as a parameter for quantification of composition in EELS and EDX. Furthermore, once the voxel size reduces (in future) to contain only one atom on average, interpretation will change: The element specific spectroscopic "4D-data space" after reconstruction would then consist of 1s and 0s only and can be largely compressed. The Z-contrast "3D data space" then contains no longer superpositions $Z_A^m + Z_B^m$, instead: $I(x,y,z) = Z_{i,j,k}^m$. With careful scaling and calibration of *m*, full spectroscopic information could then be claimed in 3D without a spectrometer; the 4D space becomes redundant.

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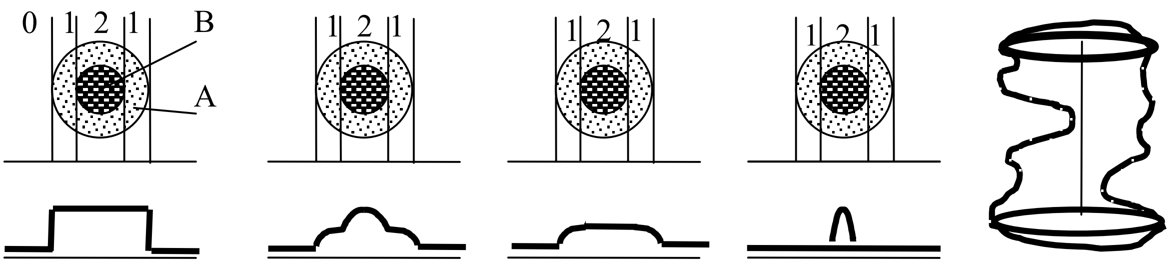

FIG. 1. (a-d) Four mechanisms of projected contrast (binary, Z-contrast, spectroscopic A and B). (e) class of objects with convex cross sections along backprojection axis.

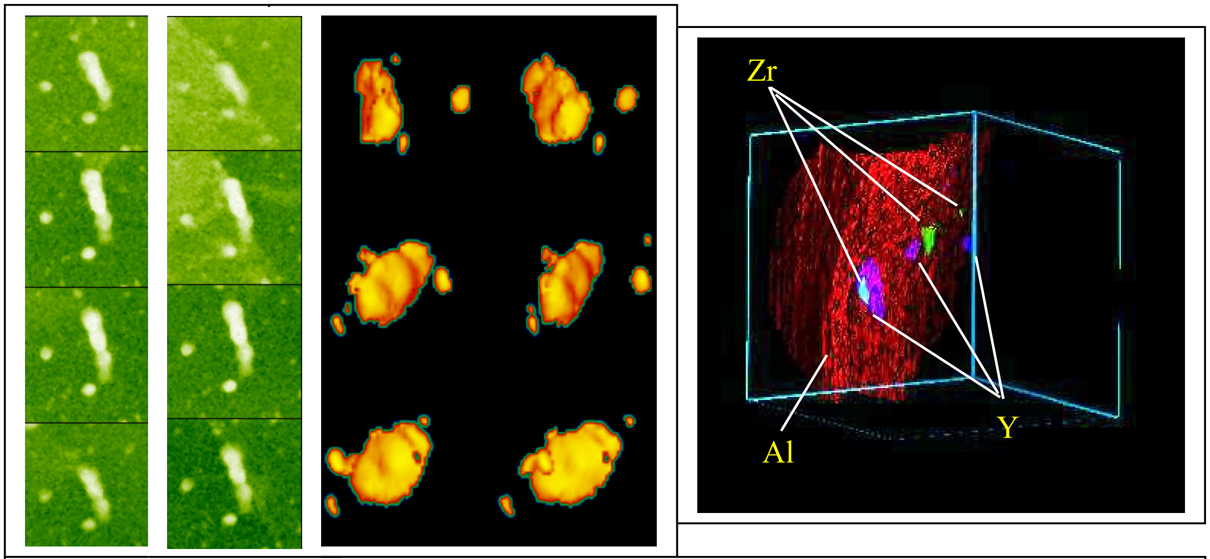

FIG. 2. (a) EFTEM tilt series of FeAl intermetallic alloy with Y_2O_3/ZrO_2 particles, recorded at Fe-L-edge inner shell loss; LEO-912Ω. (b) Reconstructed by filtered backprojection; threshold rending for Y_2O_3 particle shape. (c) Reconstruction of 3 spectroscopic energies from tilt series of EDX maps. B/W display (see CDROM for RGB-colour: red=Al, blue=Y, green=Zr) of a super-position of Al, Y, Zr volume maps; same material as in (a,b); JEM 2010. For details see [6,7].

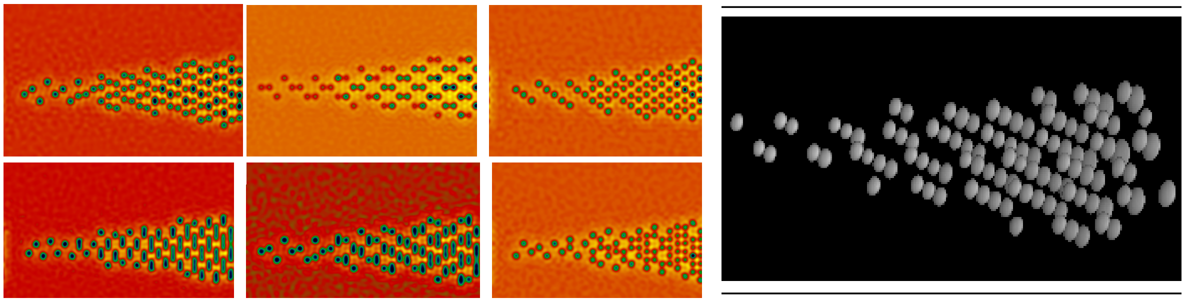

FIG. 3. (a) Silicon-tip, HREM simulations every 3^{O} over 180^{O} (6 shown). (b) Reconstructed by linear backprojection (Optical data for JEM-ARM1250, Scherzer focus).