1077
doi:10.1017/S1431927615006170 Paper No. 0538 Microsc. Microanal. 21 (Suppl 3), 2015

Channelling and atomic resolution STEM using X-ray emissions with absorption

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Although quantitative analysis using soft characteristic X-rays is beset by relatively high absorption effects, the concomitant skewing (depending on detector-specimen geometry) of the detected X-ray signal towards ionizations which have occurred closer to the surface may have advantage in retaining coherent information compared with an incoherent contribution. It is envisaged that strong absorption of soft X-rays may enhance coherent contrast in both lattice images and channelling patterns compared to that derived from more energetic X-rays, particularly with specimen-detector geometry at a grazing angle of detection to enhance this effect. A Bloch wave approach provides a framework in which both ionization localization and absorption of generated X-rays may be accounted for, and enables explicit separation of coherent and incoherent contrast. An initially unexpected outcome is an ability to quantify the degree of cross-talk between projected columns of atoms in lattice resolution mode. It turns out that the incoherent component is responsible for most of the cross-talk.

Fig. 1 compares thickness-averaged signals from an aberration-free focused 200 keV coherent beam of 1.2 Å resolution placed on an Au column in the <100> projection with a channelling response from a collimated beam. As the interaction becomes more delocalized, the variation in response is diminished, although this is somewhat masked by thermal smearing. Note that the signal is developed within the first 30 Å, after which it decays rapidly with thickness. The build-up of an incoherent background is solely a reflection of the absorptive potential and is invariant with crystallographic site and interaction delocalization. Geometry-adjusted effective mean free paths for X-ray absorption are assumed to be 10, 0.5 and 0.1 μm respectively for L, M and N excitations. <100> channelling patterns for L and N shell excitations as well as the N channelling ratio with a pair of X-ray detectors placed symmetrically above and below the specimen are shown in Fig. 2. The higher absorption of N shell excitations yields stronger top/bottom ratio contrast. Fig 3 shows calculated STEM lattice images for these excitations. A *gedanken* experiment allows isolation of a central column of atoms within the superlattice, and cross-talk is observed as the beam is scanned across neighbouring columns. With the coherent wavefunction ϕ injected onto atomic columns, the central maximum is rapidly attenuated at depths greater than 50 Å, and a hole is formed at about 100 Å thus eliminating almost entirely coherent ionization events at greater depths. This rapid and selective absorption of ϕ means the top few atoms alone yield coherent ionization intensity in Au. The ratio of contrast achieved by the top and bottom detectors is also shown in Fig. 3. Other materials with higher Debye temperatures and lower atomic numbers (such as Si or TiAl alloys) may allow coherent ionization effects to be monitored to greater depths, and this may also be helped by specimen cooling to inhibit incoherent scattering.

It is my view that quantitative analysis of X-ray channelling patterns may ultimately prove more robust and indeed more useful than the pursuit of atomic resolution in STEM. For instance, the determination of an interstitial site of 1% Cr in mullite [1] would be well-nigh impossible by STEM, but was readily achieved by channeling effects. STEM is compromised in both real and reciprocal space, whereas channeling is not compromised in reciprocal space and has a resolution determined by ϕ itself.

[1] C.J. Rossouw and P.R. Miller, American Mineralogist 84 (1999) 965-969.

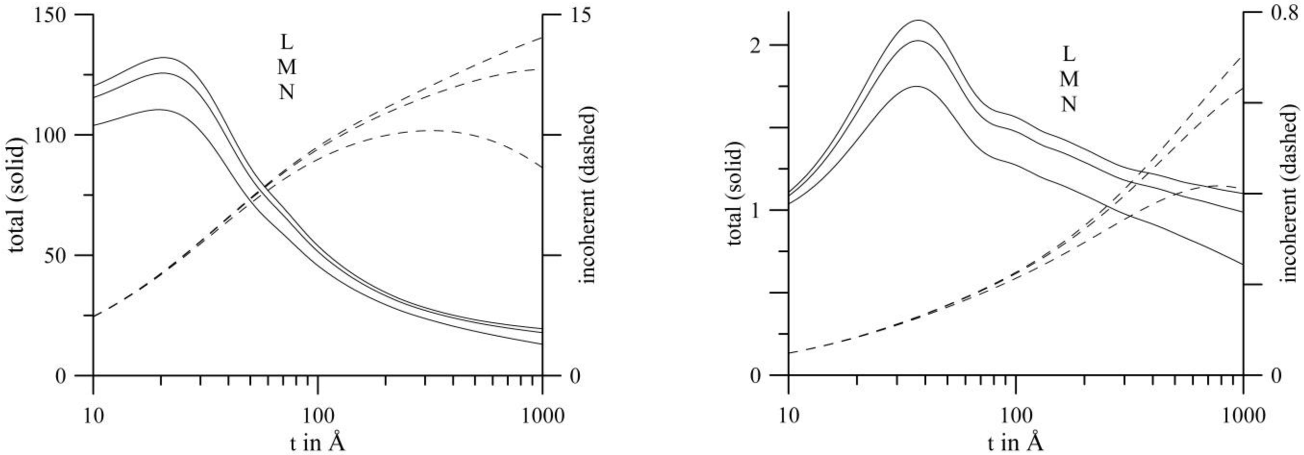

Fig. 1 Au L, M and N shell responses showing total and incoherent signals. The N shell X-rays are more strongly absorbed (see dashed lines). Left is a coherent beam focused onto an Au column and right is channeling response from a collimated beam (see text).

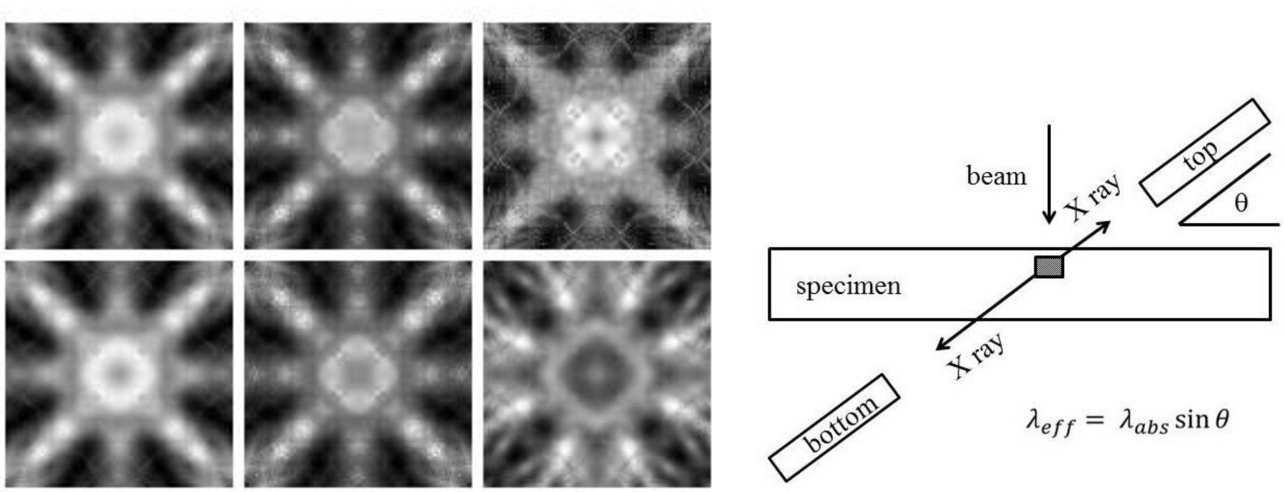

Fig 2 L- and N-shell Au channelling patterns 200 Å (top) and 500 Å (bottom). Right, incoherent pattern and bottom, ratio of N emissions top/bottom due to variation in X-ray absorption (7 % contrast).

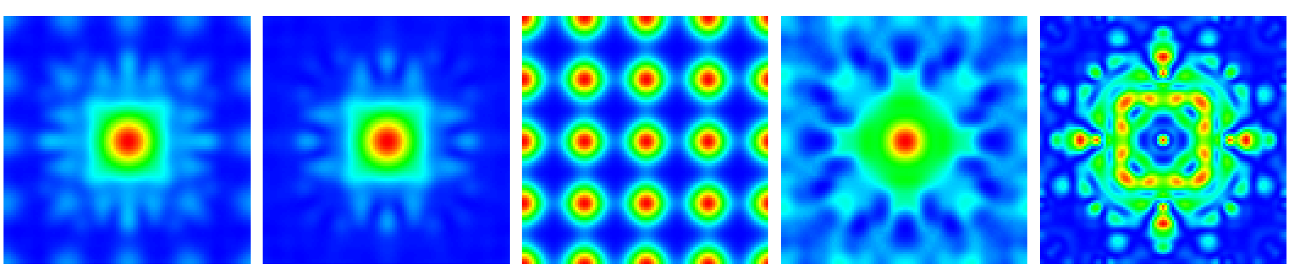

Fig 3 M-shell STEM lattice images 200 Å. Total, coherent, incoherent, total ratio top/bottom (25 % contrast) and wavefunction ϕ^2 at 100 Å. Note appearance of central hole in ϕ^2, log scale.