

Element/Site-Selective Microanalysis Using High-Angular Resolution Electron Channeled X-ray/Electron/Light Spectroscopy

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The current state-of-the-art scanning transmission electron microscope (STEM) equipped with aberration correctors allows us handy for atomic scale imaging and elemental/electronic structural analysis, due to its high-brightness electron source and highly focused subatomic probe size available. The technique inevitably leads to a drawback associated with a high density of focused electron probe and its small illuminating area (i.e., a small number of sampling points), so that the probe can drill the sample or otherwise the obtained data may contain high level noise accordingly. We have hence been engaged in an alternative microanalysis method, instead of exploiting the atomic resolution in real space, using inelastic scattering by channeled electrons in a crystal. The method takes advantage of the high angular resolution in reciprocal space intrinsic to TEM, and we have developed an ‘integrated electron spectroscopic STEM’, where electron energy-loss spectroscopy (EELS), energy/wavelength dispersive x-ray spectroscopy (E/WDXS) and cathodoluminescence (CL) are implemented in a single STEM, as shown in Fig. 1.

The concept of the present element/site selective microanalysis is schematically shown in Fig. 2, where the incident electron beam is rocked about a pivot point on a sample, acquiring the spectroscopic data as functions of the diffraction condition (and the momentum transfer vector in EELS) with respect to the incident beam direction. The sample orientation and diffraction condition is monitored by the beam rocking pattern recorded by the ADF detector. The present method is an extension of HARECX(E)S [2,3], thereby exploiting element/site selective chemical information of the material associated with the different electron densities propagating along the specific atomic planes/columns by varying Bloch wave symmetry excited in the crystalline sample even from nanoscale areas. The acquired datasets of fluorescent x-ray intensities, core-loss spectra and light intensities bear information on the local spatial/electronic structures around the excited elements of interest, which can be quantitatively analyzed by comparing with theoretical simulations, based on the dynamical elastic/inelastic electron diffraction theories [4].

We have analyzed rare earth dopants in a metal oxides for a novel red light-emitting material (Eu/Y co-doped Ca_2SnO_4) as one of the representative applications of the present method: the occupation sites of Eu and Y were quantitatively determined by EDXS [5] (cf. Fig. 3), the valence states of the rare earths by EELS [5] and the active light emitting site by CL [1].

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References

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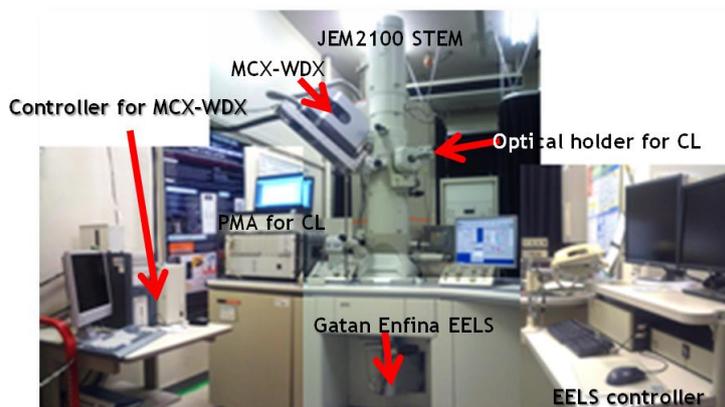


FIG. 1. Photo of current integrated electron spectroscopic STEM equipped with EDX, WDX, EELS and CL detectors.

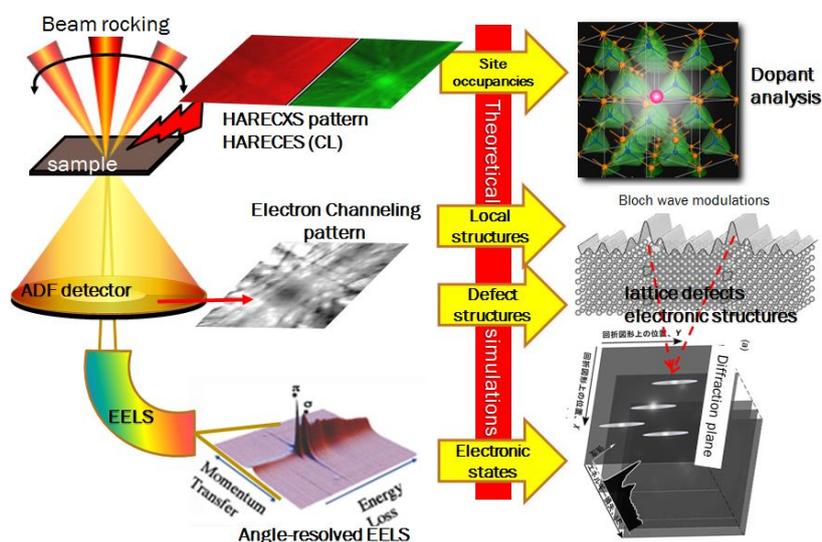


FIG. 2. Schematic diagram of element/site selective microanalysis using integrated spectroscopic STEM.