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Cathodluminescence in a (S)TEM – Exploring Possibilities and Limits

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We hereby present first results from a newly-installed JEOL 2200FS (scanning) transmission electron microscope (S)TEM equipped with a Gatan XiClone CL detection system. The use of a cryo-stage pole piece with a large gap allows insertion of a 3 mm parabolic mirror for efficient light collection, which can be detected either in pan or monochromatic mode on a Peltier-cooled PM-tube, or with a liquid nitrogen cooled CCD camera for parallel spectral (para-CL) acquisition. The FEG provides emission currents up to ~160 μA with an accelerating voltage range of 80–200 kV; STEM probes range in size from 0.2 nm high-resolution (HR) to 2 nm analytical (i.e. high current). An array of STEM detectors (BF, ADF, HAADF, SE, BSE) allows imaging from low magnifications to HR with good contrast for samples of all thicknesses, giving high flexibility. Adding to this flexibility is the in-column Ω-filter, which can be used for STEM electron energy-loss spectroscopy (EELS) with a sub-1 eV energy resolution simultaneously to para-CL acquisition, and a CCD camera that can record STEM diffraction patterns. Liquid nitrogen cooled and cryo-transfer stages are available. The piezo-mechanical stage affords excellent stability; thus mapping over day(s) is possible [1]. Spectral and spatial resolutions of better than 2 nm are observed (Fig. 1).

Compared to SEM-CL instruments, STEM-CL offers many interesting capabilities and research possibilities. Our current work spans samples ranging from geological rocks to plasmonic particles, through semiconductors, from nano-scale particles to bulk, and photonic crystals. A main strength of CL in STEM is its ability to be recorded simultaneously with other signals: BF or low-angle ADF STEM for defect visualization; HAADF for compositional contrast; EELS for plasmon response, thickness measurement, compositional profiling and bonding-state analysis. Other advantages are the small interaction volume of the beam with the sample [2], which can lead to improved CL spatial resolution for e.g. plasmonic mapping [3] or panchromatic imaging of geological samples, and the possibility of using fast electrons to stimulate and measure Cherenkov radiation. Such advantages are, however, balanced against inherently lower CL signal compared to SEM-CL, which, for instance, necessitates long mapping times.

We present examples for simple simultaneous CL-EELS measurement by measuring the thickness of GdO particles along with their CL spectra, showing that ratios between their luminescence peaks are size dependant. Another example illustrating the complementary nature of the two signals comes from mapping bright plasmons with CL (due to their relatively low energy), and dark plasmons – which are of higher energy (5–10eV) and do not luminesce – with EELS.

By correlating results with SEM-CL, Group III nitride structures are investigated at several acceleration voltages (1–200 kV). It appears that, at high voltages (200 kV), the CL signal drastically drops and Cherenkov radiation dominates. However, high-spectral-resolution characterization of Cherenkov radiation using CL could be useful for determining and subtracting its contribution to low loss EEL spectra. Another application of Cherenkov radiation is the mapping of 2D Bloch modes of photonic crystal structures and their comparison with calculation [1].

STEM-CL is unmatchable in optical characterization of defects (in combination with BF and ADF imaging). We present emission dips at partial dislocations in ZnO films (Fig. 2) and threading dislocations in group III nitrides (including quantum wells). Further work is under progress, such as simultaneous CL and chemical composition mapping with EELS in group III nitride alloys (especially InGaN quantum wells), as well as optical property measurements of semiconductor nanostructures (CdSe and ZnO nanorods, silicon nanoparticles).

References

[1] Alexander et al., *"Cathodoluminescence mapping of Cherenkov-radiation generated Bloch-modes in planar photonic crystals by fast electrons"* submitted to MSA 2010, Portland.
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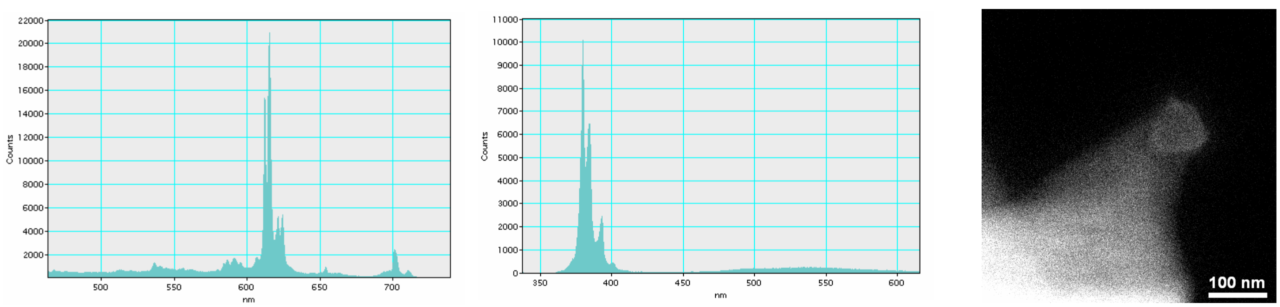

FIG. 1 Para-CL spectra acquired from NaEu(NoO4)2 luminescent crystals (left) and solid-vapour-liquid (VLS) synthesized ZnO nanowire (middle) show spectral wavelength resolution of < 2 nm. Panchromatic CL image of ZnO nanowire (right) shows a sharp change in CL signal at the edge of the gold catalyst particle over a distance of just 1 nm spatial resolution.

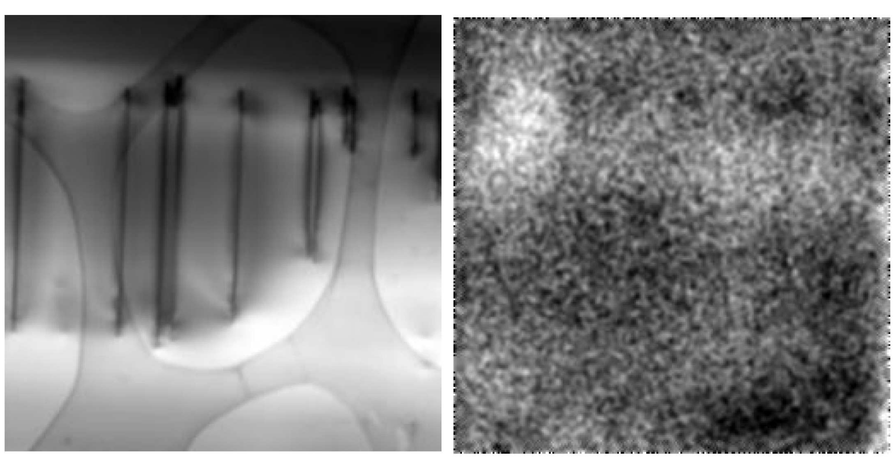

FIG. 2 Partial dislocations in a ZnO thin film and their CL emission map, showing a dip of luminescence at the dislocations, but not at the stacking faults.

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