Development of a Cathodoluminescence Detection System for the (S)TEM Demonstrating Sub-nm Spatial and Sub-meV Spectral Resolution

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The development of nanomaterials with unique optical and electronic properties offers great potential in the advancement of optoelectronic devices, telecommunications and sensor technology. Cathodoluminescence (CL) microscopy in the SEM is a characterization technique which has enriched understanding of these materials through the correlation between morphology and luminescence, with spatial resolution approaching 10nm [1]. However, there remains a growing interest in performing CL microscopy in the (S)TEM where the morphology, microstructure and local chemistry can be attributed to the luminescence properties, at a spatial resolution potentially better than 1nm [2, 3].

Here we present a novel CL detection system suitable for a wide range of (S)TEM instruments and show exemplar results from semiconductor heterostructure nanorods and colloidal silver nanoparticles; luminescence information with a spatial resolution on the order of 1nm is demonstrated.

Highly efficient collection of the CL signal is achieved through miniature diamond-turned mirrors integrated into the tip of a conventional cryogenic side-entry TEM holder. Mirrors above and below the specimen provide a solid angle of collection of up to 7.3steradian; a significant increase over existing systems where a single collecting mirror mounted to the TEM column is employed [4]. Light is coupled out of the holder through two NA-matched optical fibres to a Czerny-Turner optical spectrometer fitted with a photomultiplier tube and CCD providing a spectral resolution better than 0.5nm (0.4meV). In addition, the holder design is compatible with standard STEM imaging modes and allows the simultaneous acquisition of CL and EEL spectral data.

Figure 1 shows a nanorod with a periodic GaN/AlN quantum disc heterostructure with quantum disc nominal thicknesses of 2nm. A CL spectrum-linescan along the length of the nanorod clearly reveals the luminescence properties of many of the individual GaN quantum discs. Figure 2 shows two localized plasmon resonance modes excited by the primary electron beam of the TEM to be spatially and spectrally resolved by CL microscopy.

References:

Figure 1. Left: HAADF STEM image of nanorod with GaN/AlN heterostructure (2nm quantum discs); GaN shows up bright. Centre: CL spectrum-linescan acquired along the length of the nanorod (indicated by the green line in the HAADF image); red arrows indicate quantum wells with directly attributable luminescence spectra; sample temperature: 90K. Right: selected CL spectra showing quantum discs 5 and 6 to be resolved spatially; specimen courtesy of Prof. R Myers, Ohio State University.

Figure 2. a) HAADF image of an egg-shaped colloidal silver nanoparticle; b) panchromatic cathodoluminescence image (acquired simultaneously to the HAADF image) displaying three ‘bright’ resonance nodes (indicated by arrow markers); c) cathodoluminescence spectrum with two peaks corresponding to spectrally discrete resonance modes at 430 and 510nm; d) and e) cathodoluminescence band pass images at 430 and 550nm ±40nm extracted from parent spectrum-image showing resonance modes are separated spatially and spectrally.