Appendix B

Optical Characterization in the Nanoprobe

The Nanoprobe system (Omicron NanoTechnology, GmbH) integrates electron, electrical, surface, chemical, and optical characterization into a single instrument operating in ultra-high vacuum (UHV) conditions. This enables simultaneous investigation of electrical transport, chemical composition, and light-emission properties in a single nanostructure. The system at Caltech is equipped with a Zeiss Gemini SEM column, a NanoSAM Auger analyzer, and four independent scanning probes, two of which are capable of mounting optical fibers for light excitation and collection as well. A photograph of the typical nanoprobe stage is shown in Figure B.1a, and an SEM image of two etched W probes contacting a Si nanowire in Figure B.1b.

At Caltech, we have integrated detection optics to enable optical characterization inside the nanoprobe. In particular, optical fibers permit laser light to be coupled into the UHV chamber for excitation in photoconductivity measurements, or for emitted light from cathodoluminescence or electroluminescence experiments to be collected and sent to an exterior monochromator, PMT, and CCD detector similar to the CL setup of Chapters 2 and 3. Traditional CL configurations use a mirror to collect the emitted light that, if properly designed, collects a cone of ±80° from the surface normal. Inside the nanoprobe chamber, there is not sufficient space for a mirror-based detection system, and for this reason we chose to integrate fiber-based collection. Coupling multimode optical fibers with 50 μm (for excitation) and 200 μm (for collection) cores to two of the piezo-driven probes allows precise positioning in lateral dimensions and in height from the sample surface.
Figure B.1. (a) Photograph of the stage in the Omicron Nanoprobe system (image courtesy Omicron, GmbH). (b) SEM micrograph of two etched W probes in electrical contact with a Si nanowire.

We used a sample of Ce-doped YAG (YAG:Ce) particles on an In foil to calibrate the fiber-based collection as seen in Figure B.2. This material is known to exhibit extremely bright cathodoluminescence. The position of the fiber was optimized to obtain maximum CL emission intensity. Figure B.2a shows the optimal in-plane position of the fiber with respect to the electron beam position (center of the SEM micrograph). Here, we use the 200 µm core fiber for collection. The CL spectra and images in Figures B.2b and B.2c show that, indeed, bright CL is observed and that the YAG:Ce particles are the source of this emission. Furthermore, these CL spectra match those obtained from mirror-based CL that were taken for comparison.

Adding this capability to the nanoprobe system enables a number of novel experiments to be performed. Not only can the electron beam from the SEM be used to induce light emission, but when the probes operate as scanning tunneling microscopes (STM), one can pursue the investigation of STM-induced light emission in metallic nanostructures. This provides even higher spatial resolution than the focused electron beam in an SEM. The nanoprobe allows precise positioning of the STM probes over a specific nanostructure for extremely localized excitation. Additionally, a variety of optical fibers could be coupled to the scanning probes, such as tapered or lensed fibers for near-field collection or excitation. Such schemes that decouple regions of excitation and collection are valuable for determining the mechanisms of light emission.
Figure B.2. (a) SEM micrograph of fiber-based CL collection in the nanoprobe. (b) CL spectra of YAG:Ce particles on an In foil and of a YAG:Ce substrate at low magnification (5 kX) and high magnification (100 kX). (c) SEM and monochromatic CL images of YAG:Ce particles. The CL image at \( \lambda = 550 \) nm shows that the particles emit at this wavelength, and not at \( \lambda = 800 \) nm.