In situ Electron Microscopy Characterization of Optoelectronic Nanostructures and Nanodevices

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One dimensional (1D) semiconductor nanostructures are considered to be promising building blocks for future electronic and optoelectronic nanodevices owning to their abilities to effectively transport electrons and photons. [1] The closely correlated electrical and optical properties of optoelectronic nanodevices often require co-measurements of microstructure, composition, and electrical and optical properties of the same individual nanostructures, especially when the dramatic inhomogeneity of as-grown nanomaterials is considered.

Two straightforward approaches have been developed to integrate optical techniques and *in situ* electron microscopy in order to achieve comprehensive characterization of individual 1D nanostructures. [2, 3] Firstly, we employ *in situ* electron microscopy and micro-photoluminescence (PL) techniques to locate and measure the same individual suspended nanostructure attached to sharp metal tips [Fig. 1(a)]. Secondly, we combine optical fiber probe detector and nanoprobe technique inside a scanning electron microscope (SEM) to assemble a comprehensive characterization system [Fig. 1(b)]. Above techniques have been applied to study the origin of "green" emission and the optical confinement in 1D ZnO nanostructures.

Figure 2 shows comprehensive measurements on an *in situ* burnt-out ZnO nanowire suspended between two tungsten (W) tips. During the *in situ* heating process by electric current, the composition (>5% oxygen loss) and the electrical conductance (first increases and then decreases before observable changes in the nanowire shape) are monitored. Micro-PL measurement of the unheated nanowire and along different locations of the burnt-out nanowire, shows a dramatically increased green emission and a noticeable UV emission redshift with the temperature increase. The correlation of PL, electrical transport and EDS measurements indicates that the UV emission redshift, green emission, and carrier density are closely related to the oxygen deficiency, which supports native defect complexes and/or hydrogen at oxygen sites serving as stable and effective shallow donor. The angular resolved cathodoluminescence (CL) measurement shown in Fig. 3 demonstrates the coexistence of Fabry–Pérot (FP) cavity and whispering gallery (WG) cavity in the same ZnO nanorod. The cavity qualities can be strongly affected by the microstructural surface defects.

The integrated characterization system also enables *in situ* assembly and characterization of nanostructures for optoelectronic device purposes. We will show its application in light emitter (Fig. 4) and photodetector based on individual nanowires. Using these examples, we demonstrate that the combination of optical techniques and *in situ* electron microscopy can be powerful for the studies of optoelectronic nanomaterials and nanodevices.

References

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FIG 1 (a) Comprehensive characterization of a suspended ZnO nanowire by *in situ* SEM and micro-PL. (b) An assembled characterization system based on SEM and *in situ* nanomanipulators.



FIG 2. (a) & (b) SEM images showing a suspended ZnO nanowire before and after *in situ* "burntout" by electrical current. (c) EDS spectra from position close to the "burnt-out" spot before and after the *in situ* heating. (d) I-V curves during the interval of *in situ* heating. The numbers indicate the cycle of the heating. The inset shows the variation of the electrical conductance. (e) Micro-PL spectra from different positions of the burnt-out nanowire (marked in Fig. 2b).



FIG 3. (a) Experimental setup of the angular resolved CL measurement of ZnO nanorod cavities. (b) CL spectra showing FP (parallel) and WG (perpendicular) cavity modes from the same individual ZnO nanorod. A spectrum without cavity modes is shown for comparison.



FIG 4. Assembly and characterization of an *in situ* light emitter based on a single ZnO nanorod. (a) and (b) Top-view SEM image and side-view schematic diagram of the light emitter. (c) Typical I-V curve measured as the W tip contacts the ZnO nanorod. (d) Tunneling luminescence (TL) spectrum obtained at the presence of a nanometer-sized gap between the ZnO nanorod and the W tip. A CL spectrum from the same nanorod is also presented for comparison.