In-Situ Structure and Property Correlation of Nanostructured Materials by Using a TEM-SPM Platform

by Jianyu Huang

Motivation—While excellent tools exist for the study of nanomaterials, such as scanning probe microscopies, high resolution TEM (HRTEM), near-field optical microscopy, etc., these tools generally lack the ability to correlate the measured properties with the material's structure, especially in real-time or under dynamic conditions. We are focused on developing research platforms and techniques that enable simultaneous structure-property correlation in nanoscale materials.

Accomplishment—1. *In-situ studies of batteries.* We are developing electrochemical cells inside a TEM with nanometer to atomic scale spatial resolution. We are also using the existing TEM-scanning probe microscopy (SPM) platform to study the battery process in real time and at an atomic scale. The research provides fundamental understanding of the science in batteries.

2. We are developing micro-electro-mechanical systems (MEMS) platforms that isolate individual nanowires/nanotubes and permit thermal property measurement while simultaneously presenting those materials inside a HRTEM for atomic-scale structural studies (see Figs. 1, 2).

3. *Cold-welding of ultrathin Au nanowires.* Collaborating with a CINT user from Rice University, we realized for the first time cold welding of individual Au, Ag,and Ni nanowires. We demonstrate that single-crystalline gold nanowires with diameters between 3 and 10 nanometers can be cold welded together within seconds by mechanical contact alone, and under relatively low applied pressures. In-situ measurements reveal that the welds are nearly perfect, with their electrical and mechanical properties approaching that of single crystalline nanowires, which is never achievable in macroscopic weldings. The high quality of the nano welds is attributed to the nanoscale sample dimensions, oriented attachment mechanisms and mechanically assisted fast surface atom diffusion. The welding of metals at the nanoscale is likely to play an important role in the bottom-up fabrication of electrical and mechanical nanodevices. The results will appear in **Nature Nanotechnology**.

4. *Discovered fractal sublimation of graphene and graphene bilayer edges* (see Fig. 3). The results are reported in **Proceeding of National Academy of Sciences** (Vol. 106, 10103 (2009)), and may provide important insight into the electronic application of graphene.

5. *Discovered for the first time superplastic deformation of salt and carbon nanotubes.* The results may guide the development of high toughness carbon nanotube composites for structural applications. These results are published in **Nano Letters** (Vol. 9, 2295 (2009)) and several **Physical Review Letters** (Vol. 97, 075501 (2006); 98, 185501 (2007); 100, 035503 (2008)) papers.

Significance—These studies provide unprecedented details regarding size, defects and surface effects on the electron, phonon, chemical, and mass transport processes in nanowires, nanotubes, and other nanomaterials (e.g. graphene), and to open a new path to study nanoscale transport phenomena in general. The results provide the fundamental science in tailoring the nanostructure fabrications for integrated device applications.

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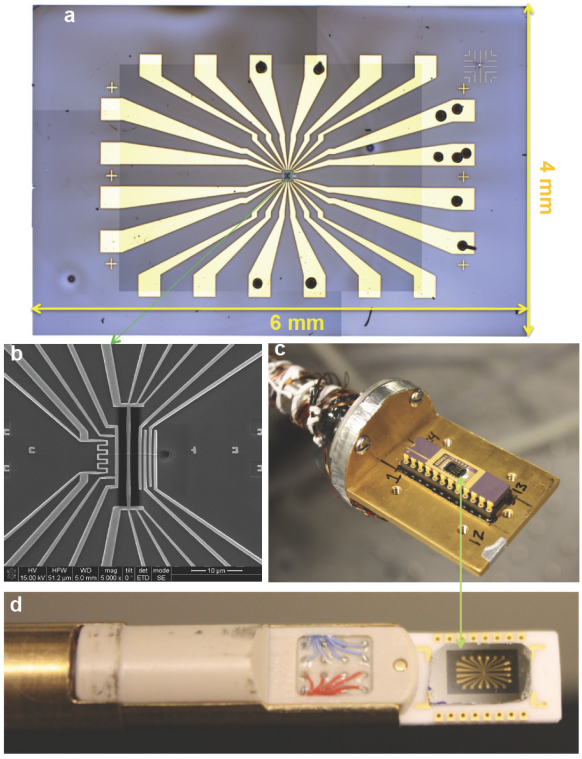

Figure 1. A MEMS device to enable both thermal/thermoelectric property measurements and TEM microstructure studies of nanomaterials.

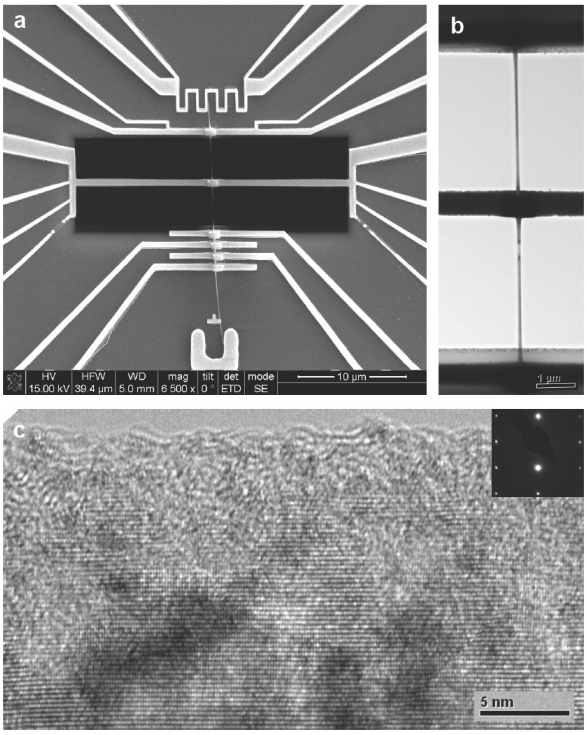

Figure 2. A GaN nanowire (NW) is mounted in a thermal microscopy platform. The atomic structure of the NW was determined.

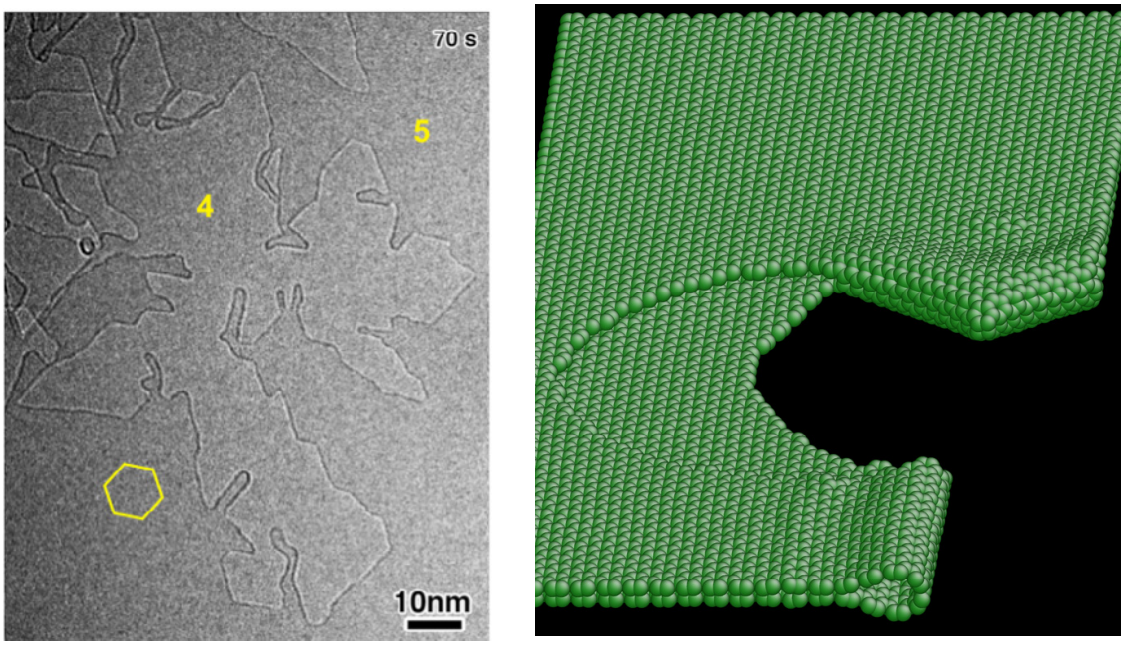

Figure 3. (Left) A snow-flake-like graphene sublimation pattern formed by in-situ Joule heating. (Right) The graphene edges formed by sublimation are bilayer edges rather than monolayer edges. The yellow hexagon marks the six sets of zig-zag planes, and the numbers mark the number of layers.