Supporting Information

Two-phase Microwave-assisted Synthesis of Cu₂S Nanocrystals[†]

Ying Wang, ^b Xin Ai, ^a Dolores Miller, ^a Philip Rice, ^a Teya Topuria, ^a Leslie Krupp, ^a Andrew Kellock^a and Qing Song^{*a}

^a IBM Almaden Research Center, 650 Harry Road, San Jose, CA 95120, USA. ^b Department of Cancer, Shengjing Hospital of China Medical University, Shenyang, Liaoning Province, 110004 China

1. Characterization

Powder X-ray diffraction was performed on a Bruker D8 Discover diffractometer with a GADDS X-ray detector and using a Cu K_{α} irradiation ($\lambda = 1.5408$ Å), which was operated at 40 kV and 40 mA for all Cu₂S crystal structure measurements. All of XRD samples were prepared by drop-casting a concentrated Cu₂S nanocrystals solution on a silicon wafer substrate.

The size and shape of Cu₂S nanocrystals were analyzed using a JEOL-2010F Field Emission Transmission Electron Microscope (TEM) operated at 200 kV. All of TEM samples were prepared by drop-casting a dilute Cu₂S nanocrystals solution on a thin carbon-coated copper or nickel grid.

XPS analysis was done on a Physical Electronics Quantum 2000 ESCA Microprobe with a monochromatic Al K_{α} source. The 1000 eV survey spectra were taken at 188 eV pass energy, 100 µm spot size and 45° takeoff angle for each sample to determine overall surface elemental composition. High-resolution spectra (29.4 eV pass energy, 0.25 eV/step) were taken for copper (2p) and sulfur (2p) to investigate chemical environment and for quantitation. Charge neutralization (1 V, 20 µÅ, Ar⁺) was used. Binding energies were referenced to C (1s) _{maximum} = 284.8 eV.

The chemical composition of Cu_2S nanocrystals was measured using Rutherford Backscattering Spectrometry (RBS). The scattering was done using He⁺ ions at 2.3 MeV from an NEC 3UH Pelletron.

2. TEM Images of Cu₂S nanocrystals



Fig. S1 (a) and (c) are TEM image and size distribution histogram of $3.3 \text{ nm } \text{Cu}_2\text{S}$ nanocrystals, respectively; (b) and (d) are TEM image and size distribution histogram of $8.2 \text{ nm } \text{Cu}_2\text{S}$ nanocrystals, respectively.

3. XRD and SEM of PbS nanocrystals



Fig. S2 SEM image of PbS nanocrystals.



Fig. S3 XRD pattern of PbS nanocrystals, which can be indexed as rock salt structure. The markers are the standard XRD pattern of JCPDS 05-0592.