Supplementary Material (ESI) for Chemical Communications

Vapor-Liquid-Solid Growth of Silicon Nanowires Using Organosilane as Precursor

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Supporting Information.

Experimental Details.

Si nanowire synthesis.

A 10 ml titanium grade 2 reactor was used for nanowire reactions by injection chemical vapor deposition (LICVD). The inlet and outlet of the 10 ml Ti reactor cell were both connected to a stainless steel (1/8" i.d.) tubing via a LM-6 HIP (High Pressure Equipment Co.) reducer. The inlet 1/8" stainless steel tubing was connected to a valve (valco) with a 10ml injection loop and the outlet of the 10 ml cell was connected to a micro control-metering valve (HF4-V, HIP), respectively. The microvalve was used to control the outlet rate of reactants with careful manual operation. A Si (100) wafer, sonically cleaned in acetone for 40 min, followed by a rinse in 2-propanol, rinsed in doubly distilled deionized water, was placed inside the reactor cell as a substrate to collect the nanowires produced. Heating tape and insulation tape cover the whole cell to maintain the reactor temperature deviation within 1 °C controlled by a temperature controller. A high-pressure liquid chromatography (HPLC) pump was used to transport water to push the piston inside the solvent cylinder and sequentially transport the anhydrous toluene to the reactor system. The system pressure was monitored with a digital pressure gauge.

Dodecanethiol-coated Au nanocrystals were prepared and size-selected according to the procedure outlined in the literature[1]. The used Au nanocrystals has an average diameter of approximately 3.9nm (Figure S1 and S2). The 10 mL precursor solution (Figure S3) with a MPS (Gelest) concentration of 630 mM in toluene (Aldrich), with dodecanethiol-capped and with a Au/Si molar ratio of 1:6000, was prepared in a glove box where the oxygen and water concentration below 1 ppm. Prior to synthesis, a 10 mL stainless steel reactor containing a cleaned silicon substrate inside was placed in the glove box to make the reactor free of oxygen and brought out from the glove box. The titanium cell was heated to 480 °C. The reactor

was flushed by anhydrous toluene at flow rate of 1 ml/min for 40 min to be filled with anhydrous toluene vapor atmosphere. The precursor solution was loaded into a 10 mL loading loop and injected by the HPLC pump. The liquid precursor feed passed through a stainless steel tube at 230℃ prior to its entry into the furnace. At this temperature, the precursor immediately volatilized and swept into the reaction zone. When the precursor solution was injected into the vapor phase toluene in the hot reactor, the micro-control metering valve was slightly manually opened to equilibrate the system pressure at 1 atm. When the reaction ended, the valve was closed and the injection flow and heater was turn off. Unreacted reactants and byproduct were condensed to the pipe line connected to the end of the reactor, collected in a glass reservoir, and disposed into waste barrels. Fresh air was used to cool the reactor cell until it reached room temperature. The Si substrate was removed from the reactor and rinsed with toluene. The Si nanowires collected from the reaction were for further characterization.

Si nanowire Characterization.

Reaction products were characterized by scanning electron microscopy (SEM), and transmission electron microscopy (TEM), X-ray energy dispersive spectroscopy (EDS), X-ray diffraction (XRD). SEM images were obtained on a HITACHI-S4700 field-emission scanning electron microscope with 3–10 kV accelerating voltage with working distances ranging between 10 to 20 mm, typically by looking at the deposition substrate with the collected product. HRTEM and EDS were performed on a field-emission Philips Tecnai F20 G2 transmission electron microscope equipped with an Oxford INCA EDS. For TEM imaging, the nanowires were dispersed in toluene with brief sonication followed by drop-casting onto a 200 mesh lacey carbon-coated copper grid (Electron Microscope Sciences). TEM images were obtained at 200 kV accelerating voltage. XRD was performed using a Phillips vertical scanning diffractometer at a 4°/min scan rate.

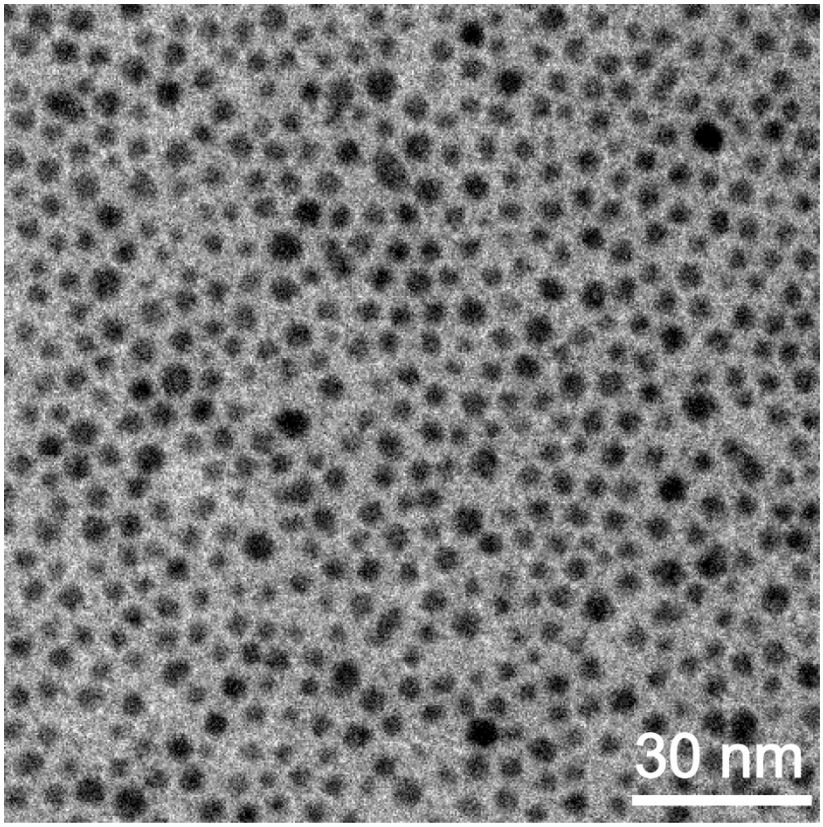

Figure S1. TEM image of dodecanethiol-coated Au nanocrystals.

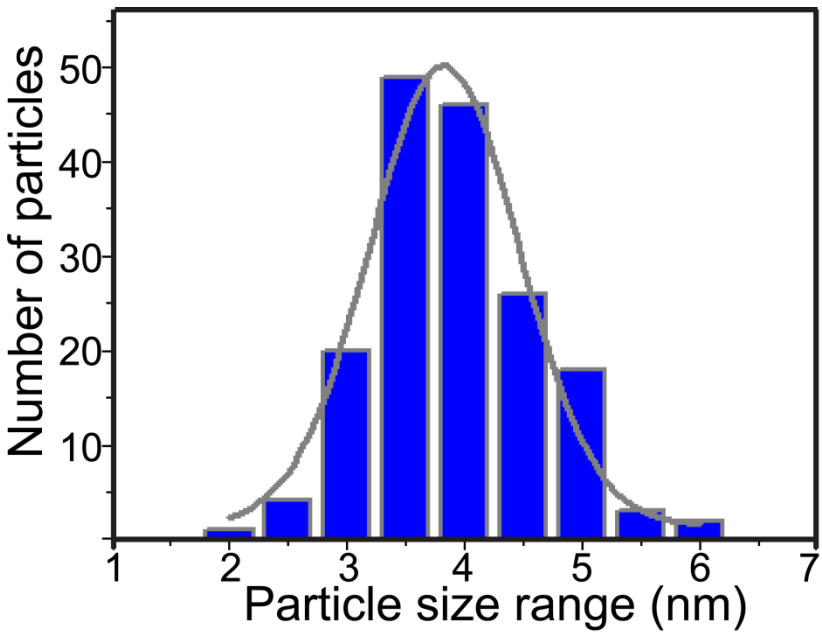

Figure S2. The size distribution of Au nanocrystals. The smooth curves show a Gaussian fit of the nanocrystals distributions.

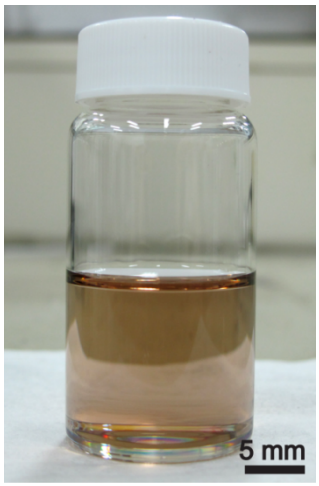

Figure S3. Photograph of precursor solutoin

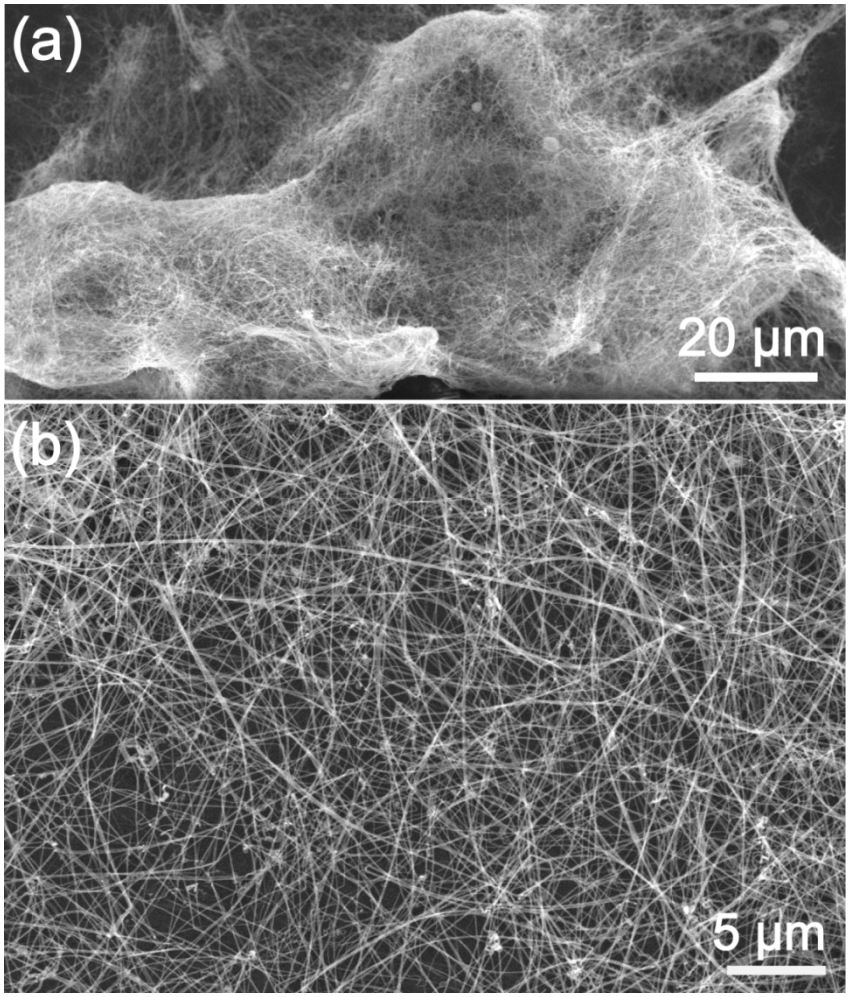

Figure S4. SEM image of Si nanowire synthesized by thermal decomposition of MPS in anhydrous toluene at 480°C by LICVD

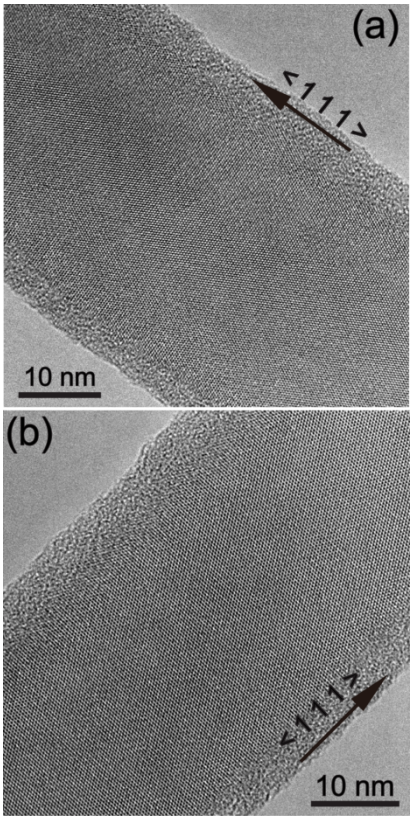

Figure S5. HRTEM images of two SiNWs with a diameter of (a) 38,(b) 41 nm, respectively.

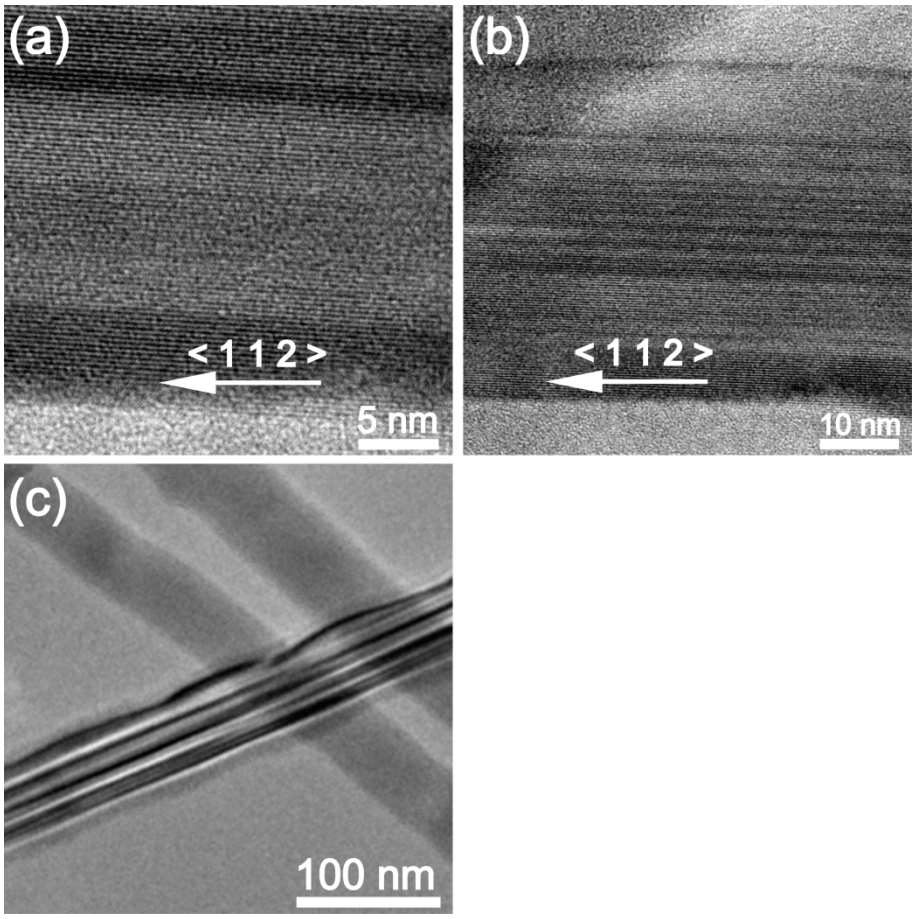

Figure. S6 TEM images of Si nanowires with ⟨112⟩ growth directions. These nanowires were grown by LICVD with Au nanocrystals. The nanowires have multiple {111} twins extending down their length oriented parallel to their ⟨112⟩ growth direction.

References

1. M. Brust, M. Walker, D. Bethell, D. J. Schiffrin and R. Whyman, *J. Chem. Soc., Chem. Commun.,* 1994, 801.