

Electronic Supplementary Information

Ultrathin corundum-type In₂O₃ nanotubes derived from orthorhombic InOOH: synthesis and formation mechanism

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Experimental section

In a typical experiment, 0.8grams of InCl₃·4H₂O was dissolved in 15.0 mL anhydrous ethanol, then 0.5 mL formamide was added to form a clear solution. Under a vigorously stirring, into the solution 1.2g of SDBS was added to form a slurry, and 15.0 mL slurry was poured into a 20.0 mL stainless steel autoclave with a lined Teflon, and heated at 140 °C for 8 h. The formed precipitate was obtained with centrifugation, washed with water and dried at 80 °C. After then the precipitate was annealed in a tubular oven at 300 °C for 0.5 h under ambient pressure.

The phase structures of the samples were identified using XRD technique on a Rigaku D/Max 2200 PC diffractometer with CuK α radiation ($\lambda=0.15418\text{ nm}$) and graphite monochromator (scan rate: 10°/min, scan step: 0.02°), and their morphology and microstructures were characterized using transmission electron microscope (TEM, JEM100-CXII) and high-resolution TEM (HRTEM, JEM-2100). The Fourier transform infrared (FT-IR) spectra were recorded on a Nicolet 5DX FF-IR spectrometer using KBr pellet technique, and the thermogravimetric analysis (TGA) was carried out on a thermal analyzer (Perkin-Elmer Diamond TG/DTA instrument) at a heating rate of 10 °C·min⁻¹ under air atmosphere.

The electronic properties of the field effect transistors (FET) devices were measured on the Keithley 4200-SCS Semiconductor Characterization System under Ar and air atmospheres, respectively. To measure the electronic properties, the samples were firstly dispersed in anhydrous ethanol (2.5 wt %) to form the suspension solution and then three drops of the suspension were dropped onto an *n*-doped silicon wafer covered with 500 nm thick SiO₂, the nanotubes self-aligned to form a thin film after the solvent evaporation, then the gold electrodes with the dimension of 28.6 mm width \times 0.24 mm length were evaporated by using shadow mask, in which the n-type Si was used as a back gate.

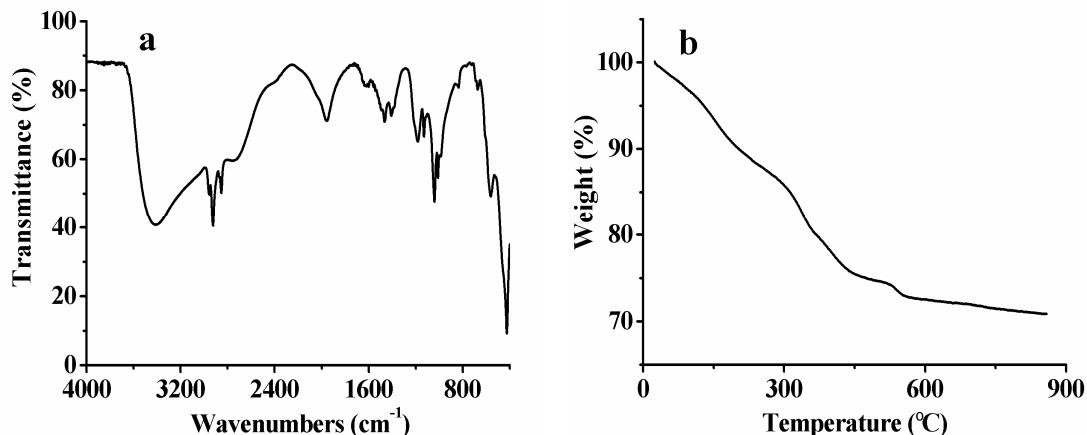


Fig. S1. FT-IR spectrum (a) and TG curve (b) of InOOH nanotubes. The hydrothermal reaction conditions: temperature: 140 $^{\circ}\text{C}$, reaction time: 10 h.

The IR spectrum shows that the InOOH nanotubes contain the surfactant molecules as well as the absorbed water besides the abundant hydroxyls. The band around 500 cm^{-1} is attributed to the In-O vibration absorption,¹ and those at *ca.*3000 to 2800 cm^{-1} are assigned to the C-H vibrations of alkane chain.² The absorptions at 1600 to 1400 cm^{-1} are due to the C=C bands of benzene ring, and that at 831 cm^{-1} results from the C-H vibration of 1,4 substituted benzene ring.³ The bands at 1200 to 1008 cm^{-1} are the characteristics of the S-O symmetry and asymmetry vibrations of alkyl benzene sulfonate.⁴ These confirm the existence of absorbed DBS on the InOOH nanotubes. A little absorbed water is also proved by the peak at *ca.*1610 cm^{-1} . In addition to the strong absorption at 3400 cm^{-1} , the O-H vibration of InOOH can also be confirmed by the broad band at 1957 cm^{-1} and the weak one at 2720 cm^{-1} .^{1,5} TG analysis shows a whole weight loss of *ca.* 29% when the product is heated from room temperature to 860 $^{\circ}\text{C}$, from which it is estimated that the content of the surfactant molecules together with the absorbed water is *ca.* 24.4%.

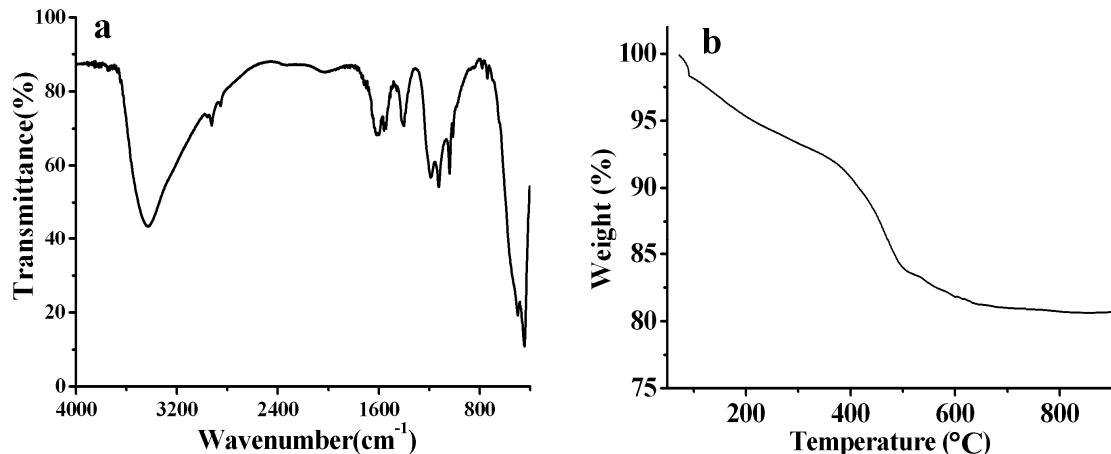


Fig. S2. FTIR spectrum (a) and TG curve (b) of the In_2O_3 nanotubes.

The annealed In_2O_3 nanotubes still contain the surfactant molecules as well as the absorbed water, which is *ca.* 18% estimated from the TGA curve. Moreover, the O-H adsorptions ((1957 cm^{-1} and 2720 cm^{-1}) of InOOH (Fig. S1a) after annealing treatment disappear (Fig. S2a).

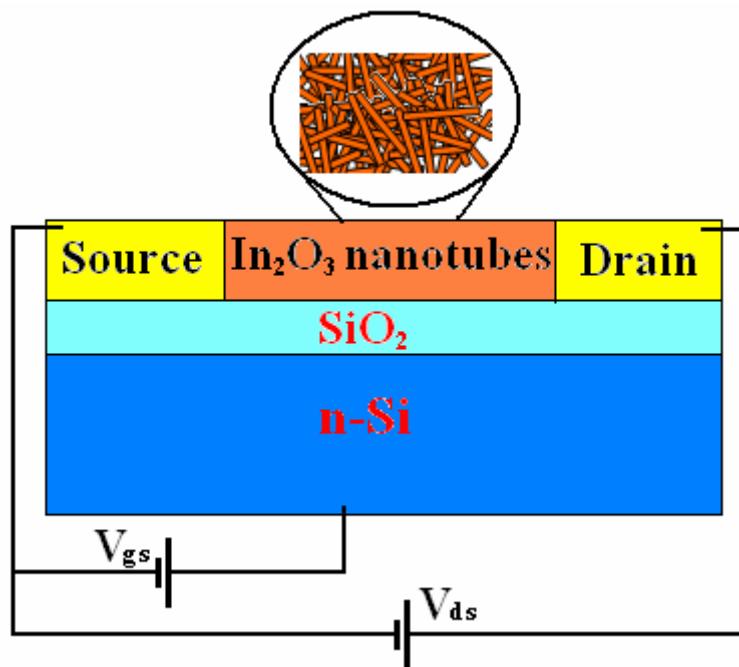


Fig. S3. Schematic cross-sectional view of the device.

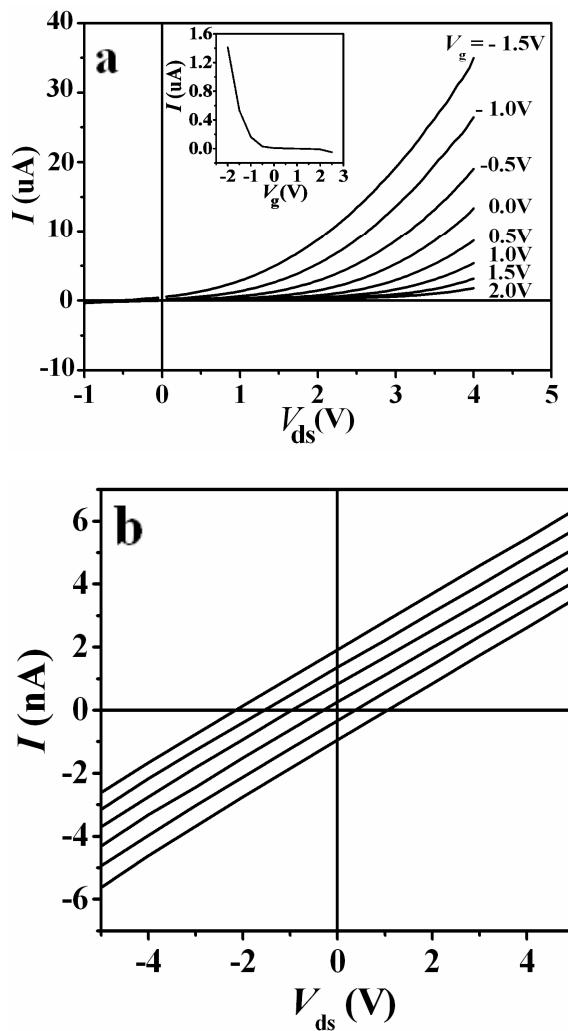


Fig. S4. (a) The output characteristics of the In_2O_3 nanotube FET devices measured in Ar gas at room temperature. The inset is the transfer characteristic of the device measured at $V_{ds}=50$ mV. (b) The I - V curves of the InOOH nanotube FET measured in Ar gas at room temperature. The gate voltages are 5, 3, 1, 0, -1, -3, and -5 V from bottom to top.

The output characteristics of FET device measured in Ar gas at room temperature exhibit the p-type channel behavior demonstrated by the decreasing drain current (I_{ds}) as the gate voltage (V_g) steps to more positive values (Fig. S5a). It indicates the formation of strong inversion layer on the surface of the *n*-type In_2O_3 coating, in which the concentration of carriers (holes) is much larger than the equilibrium concentration of the electron in the bulk In_2O_3 coating. The transfer characteristic of the device measured at $V_{ds}=50$ mV shows that the hole carriers are depleted when the gate voltage increases to *ca.*-0.5 V. The mobility of the

carriers is estimated to be $20.8 \text{ cm}^2/\text{Vs}$ from the slope $dI_{ds}/dV_g = 1.24 \times 10^{-6} \text{ A/V}$ at $V_{ds}=0.05 \text{ V}$ as determined from the linear portion of the I_{ds} - V_g curve according to the linear region transistor equation, $I_{ds} = (W/L)\mu C_0(V_g - V_t)V_{ds}$.⁶ Such electronic properties are similar to the single-walled carbon nanotubes (SWNTs) FETs,⁷ in which the p-type channel conduction behavior is attributed to the O₂ molecules adsorption on SWNT side walls for their electron-withdrawing capability.⁸ However, the measurement after exposure the FET devices in air for several days demonstrates that the p-type channel conduction characteristics still remain, indicating that the O₂ molecules doesn't affect the transfer characteristics. The results are highly reproducible with tens of independent In₂O₃ nanotubes' FETs, showing that the devices possess stable p-type channels conduction behavior.

References

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