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Supporting Information for:

**Reproducible Layer-by-Layer Exfoliation for Free-Standing Ultrathin Films of
Single-Walled Carbon Nanotubes**

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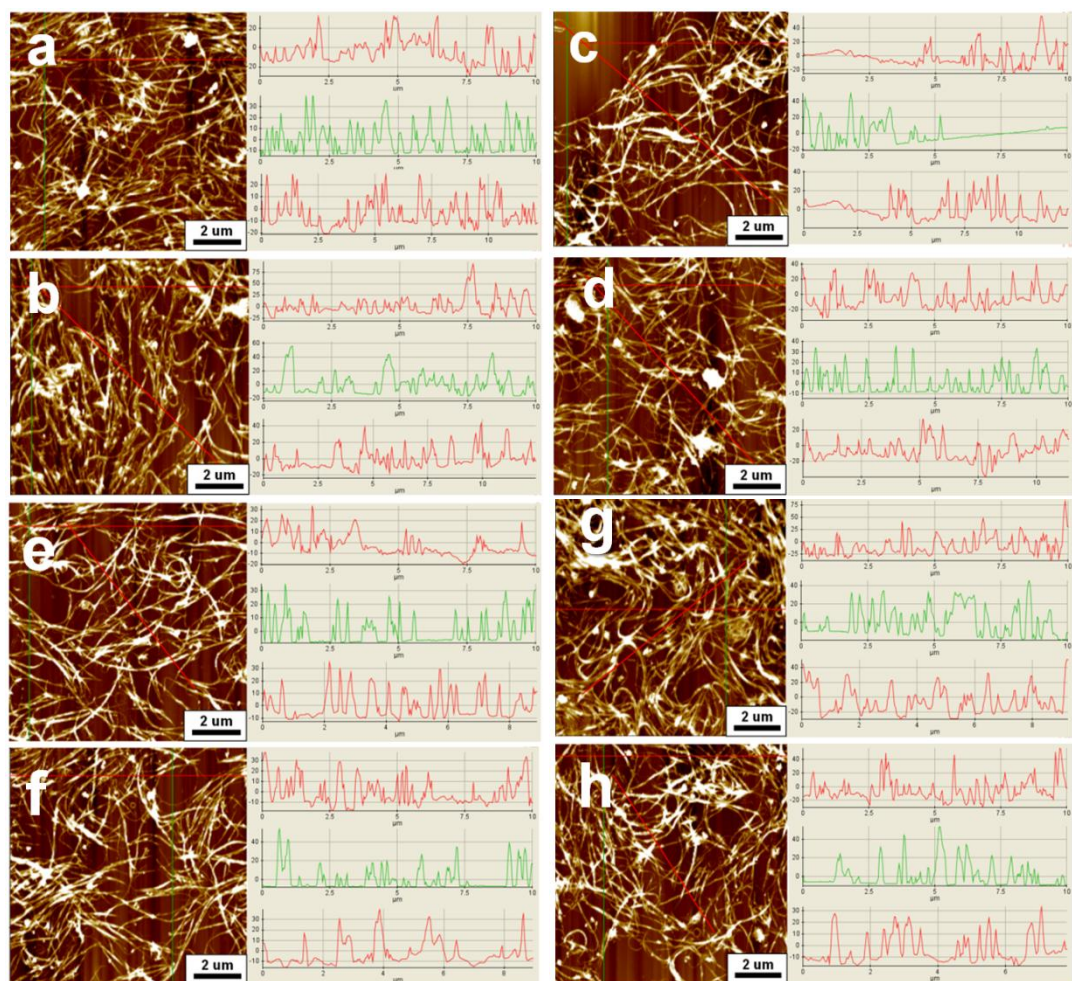
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Materials and Characterization: SWNT (>90%) powders were purchased from Chengdu Organic
10 Chemicals Co., Ltd., Chinese Academy of Sciences. Hydrogen peroxide (H₂O₂, 35%), hydrochloric acid (HCl,
36-38%) and N, N-dimethylformamide (DMF, 99.5%) were purchased from Sinopharm Chemical Reagent Co.,
Ltd. Porous Teflon membrane filters (450 nm pore size, 50 mm diameter) were purchased from Beijing
Zhuoxin Weiye Co., Ltd.

The as-prepared SWNT ultrathin films were characterized by Scanning Electron Microscopy (SEM),
15 Atom Force Microscopy (AFM), Raman spectroscopy, FTIR and UV-vis-near-IR spectrum. SEM images were
obtained using a Hitachi S-4800 field emission scanning electron microscopy at an accelerating voltage of 3.0
kV. The thicknesses and surface morphologies of the ultrathin films were examined by an AFM microscopy
(Park XE-120) in the tapping mode in air. Raman spectra and film transparency measurements were carried
out using Raman spectroscopy (with excitation wavelength of 632.8 nm) and UV-vis-near-IR
20 spectrophotometer (Perkin Elmer Lambda 750), respectively. The sheet resistances of various ultrathin films
were measured by a standard four point probe configuration (Keithley 4200).

Flexible Sensor Fabrication and Measurements: The as-prepared ultrathin film on water surface was
transferred to precleaned PET substrate and dried at 50 °C in air for 30 min. Afterward, conducting Ag paste
was placed on the both sides of SWNT ultrathin film to form electrodes (as show in Figure 4b). Then the
25 flexible sensor is heated to 80 °C under vacuum for at least 2 h to solidify the Ag paste and improve its
conductivity. For NH₃ gas detecting experiments, the flexible sensor was installed onto the inner wall of a gas
tubing, and the resistance of the flexible sensor was continuously monitored with a custom Lab View program

under 1 V DC bias potential. The analytes gas flow through the gas tubing and over the surface of the flexible sensor. The dry air was used as a reference gas, and the different concentrations of NH₃ were achieved by mixing the known concentrations of NH₃ and dry air with different flow rates. The flow rates were regulated by mass flow controllers (Seven star CS200).



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Fig. S1 (a-h) AFM images and height profiles of the eight SWNT ultrathin films.

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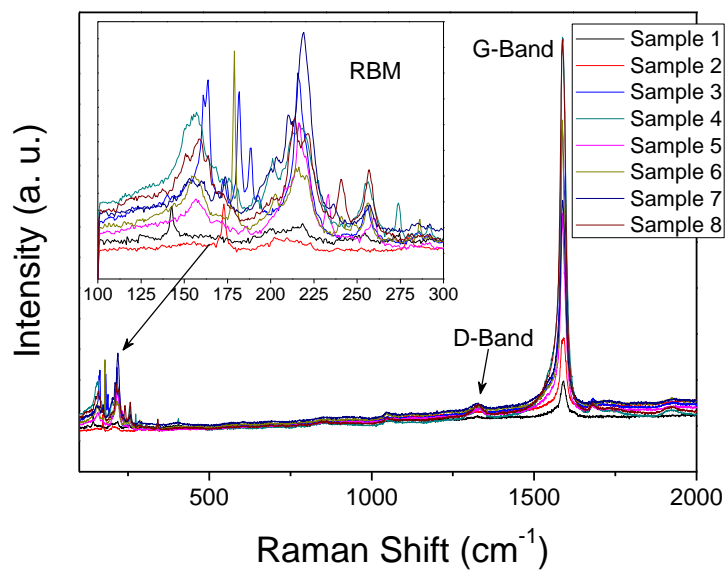
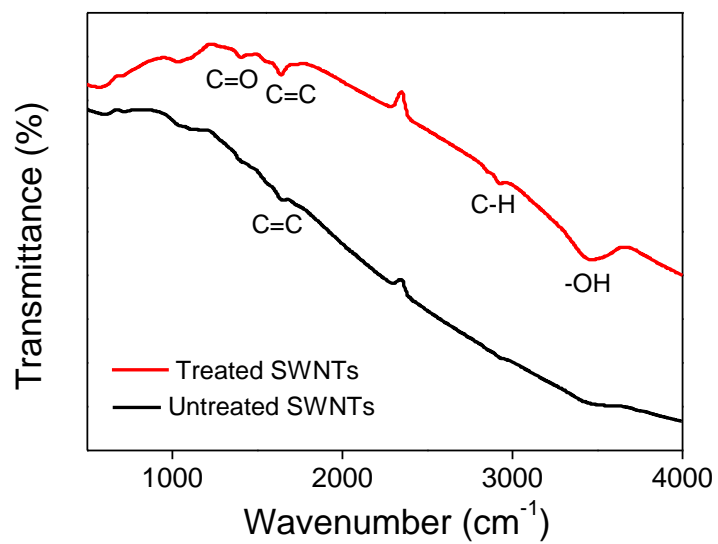


Fig. S2 Raman spectra of the eight samples of SWNT ultrathin films on glass substrates. Insert: the RBM of the as-prepared eight SWNT ultrathin films.

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Fig. S3 FTIR spectra of (black) untreated SWNT and (red) HCl/H₂O₂ treated SWNT.

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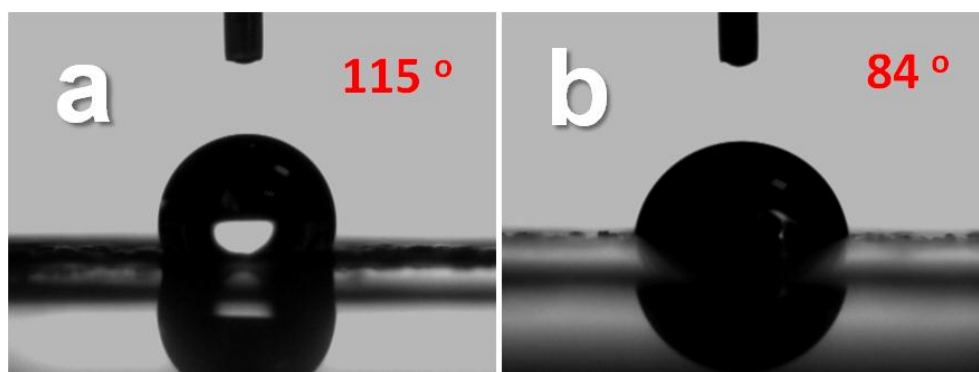


Fig. S4 Contact angles of (a) untreated SWNTs film and (b) HCl/H₂O₂ treated SWNTs ultrathin film.

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