

Supplementary Information

First sub-kilogram-scale synthesis of high quality ultrathin tellurium nanowires

Kai Wang,[‡] Yuan Yang,[‡] Hai-Wei Liang,[‡] Jian-Wei Liu, Shu-Hong Yu*

*Division of Nanomaterials & Chemistry, Hefei National Laboratory for Physical Sciences at
Microscale, Collaborative Innovation Center of Suzhou Nano Science and Technology,
Department of Chemistry, University of Science and Technology of China, Hefei 230026, P. R.
China. Fax: +86 0551-63603040; E-mail: shyu@ustc.edu.cn.*

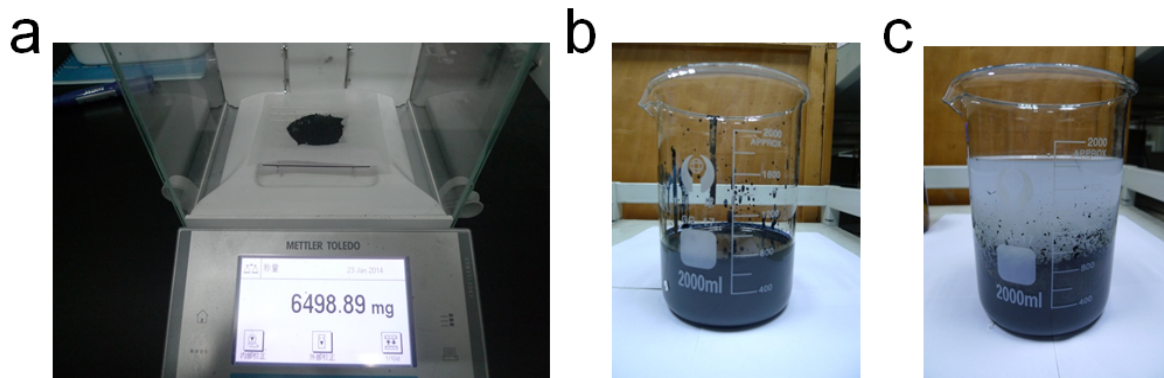


Fig. S1 (a) TeNWs powder sample on the balance; (b) Photograph of the partial sample in a beaker; (c) Photograph of the TeNWs sample extracted by acetone in a beaker.

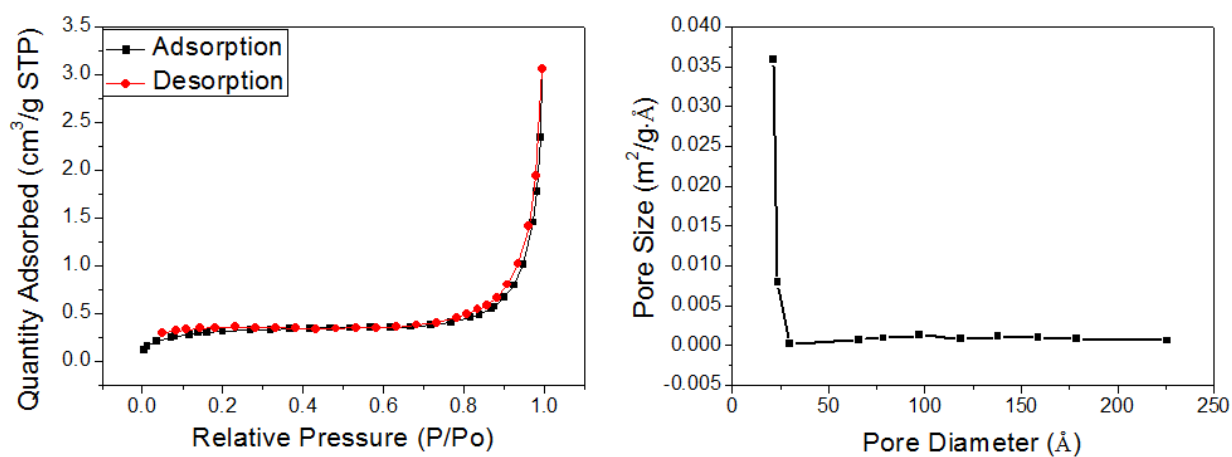


Fig. S2 (a) The BET and (b) the pore size of the as-prepared TeNWs.

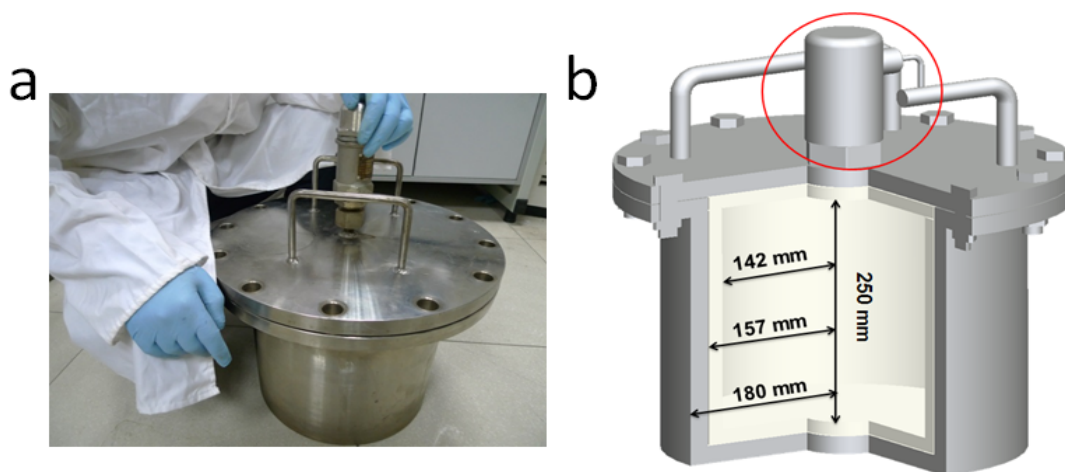


Fig. S3 (a) Photograph of a 16 L Teflon-lined stainless steel autoclave; (b) the diagrammatic drawing of the autoclave.

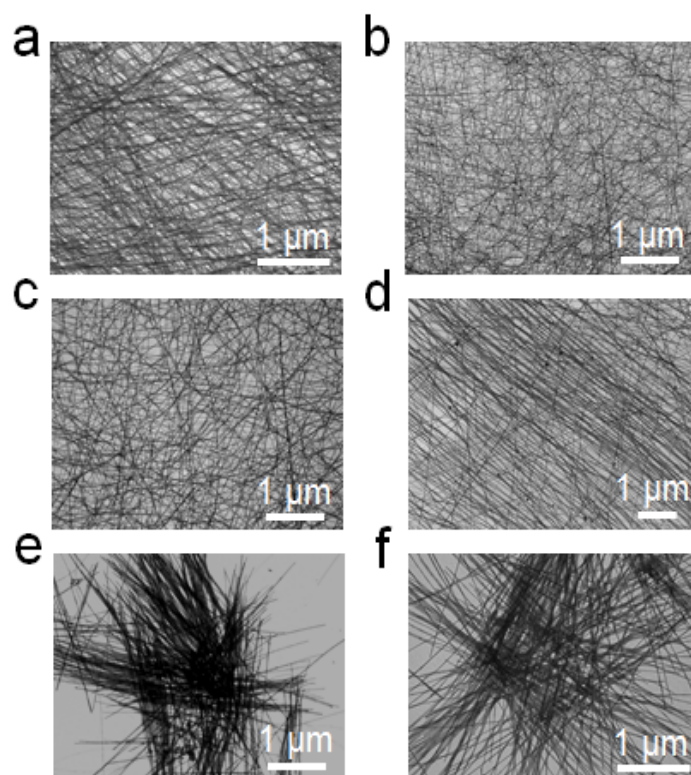


Fig. S4 TEM images of the as-synthesized samples which were prepared in 32 mL double distilled water with 1 g of PVP, 5 mL of aqueous ammonia solution, 3.35 mL of hydrate hydrazine, and with the addition of different concentration of Na_2TeO_3 at 180 °C for 3 h in the 50 mL Teflon-lined stainless steel autoclave, (a) 1.36 mM, (b) 2.26 mM, (c) 3.16 mM, (d) 4.51 mM, (e) 6.78 mM and (f) 9.04 mM.

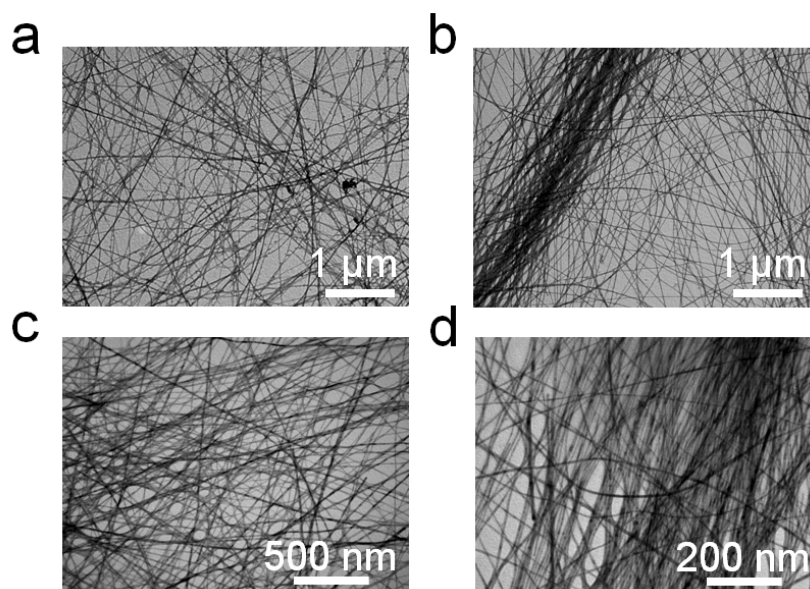


Fig. S5 TEM images of the as-synthesized samples which were prepared in 32 mL double distilled water with 1 g Na_2TeO_3 , 5 mL of aqueous ammonia solution, 3.35 mL of hydrate hydrazine, and with the addition of different concentration of PVP at 180 °C for 3 h in the 50 mL Teflon-lined stainless steel autoclave, (a) 0.3 g, (b) 0.5 g, (c) 0.7 g, (d) 1.5 g.

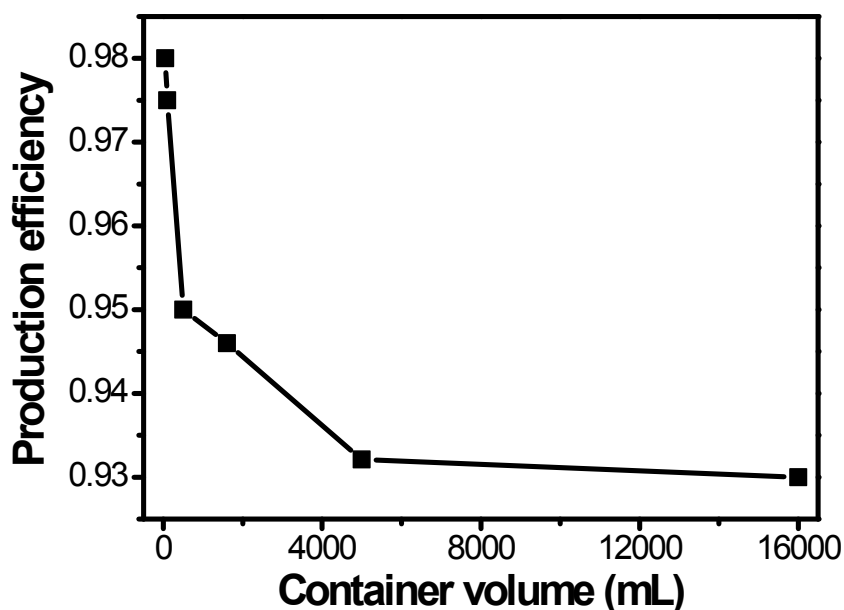


Fig. S6 The graph of the relationship between the production efficiency and the volume of the reaction containers.

Methods

Chemicals. Na_2TeO_3 , poly(vinyl pyrrolidone)(PVP, degree of polymerization: 360°), hydrazine hydrate (85% w/w%), aqueous ammonia solution (25–28% w/w%), ethanol and ethyleneglycol (EG) were purchased from Shanghai Chemical Reagent Co. Ltd. All the chemical reagents used as received without further purification. Doubly distilled deionized water (DIW, 18.2 MΩ) was used for all preparations in the synthesis of TeNWs and Te-based NWs.

Ultra-large-scale synthesis of ultrathin TeNWs.

The up-scale synthesized high-quality uniform ultrathin TeNWs were prepared by improving the synthesis process reported previously²². In a typical synthesis, 1.000 g PVP and 1 g Na_2TeO_3 (4.5 mmol) were firstly dissolved in 32 mL of double distilled water to form a homogeneous solution through vigorous magnetic stirring at room temperature. Then, 3.35 mL of aqueous ammonia solution (25-28%, w/w %) and 5 mL of hydrazine hydrate (85 %, w/w %) were added

into the mixed solution promptly. After that, the final solution was transferred into a Teflon vessel with a total volume of 50 mL. Finally, the container was closed and maintained at 180 °C for 3 h and cooled down to room temperature rapidly by cold tap water. The synthetic procedures of the TeNWs in greater container were similar to the methods that have been mentioned above. As long as expanding the radio of the synthetic raw materials, the TeNWs could be prepared in 100 mL, 500 mL and 1600 mL successfully. To get the high quality ultrathin TeNWs in 5 L and 16 L container, in addition to expand the radio of the raw materials, the reaction time should lengthen to 6 and 10 h, respectively. The TeNWs samples could be precipitated by adding appropriate acetone into the solution and washed with acetone for several times and then dried in vacuum at 60 °C.

Templating Synthesis of Ag₂TeNWs.

The Ag₂Te could be synthesized through the chemical transformation by using the as-prepared TeNWs as the template²⁵. The synthesis method was according to the research work reported previously. 80 mL of the as-synthesized TeNWs was extracted by 30 mL acetone and then was added in 80 mL ethylene glycol (EG) to form a homogenous solution by stirring dramatically at room temperature. 2.455 g of AgNO₃ was first dissolved in 5 mL ethylene glycol. After that, the AgNO₃ solution was added in the as-produced TeNWs solution slowly with the vigorous magnetic stirring. It could be seen that the color of mixed solution changed from dark blue to brown immediately, which indicating the TeNWs have already transferred to the Ag₂TeNWs. The final solution was transferred into a Teflon vessel with a total volume of 100 mL and maintained at 100 °C for 10 h to crystallize the nanowires. The products was centrifuged after natural cooling and washed several times with absolute alcohol and double distilled water, and

then dried in vacuum at 60 °C.

Templating Synthesis of Cu₂TeNWs.

As mentioned in the previous report²⁵, 80 mL of the as-synthesized TeNWs was extracted by 30 mL acetone and then was added in double distilled water with 5.000g of polyvinylpyrrolidone (PVP) to form a homogenous solution by stirring dramatically at room temperature. 3.491 g of Cu(NO₃)₂ was dissolved in 5 mL double distilled water. And then the Cu(NO₃)₂ solution was added in the as-produced TeNWs solution slowly with the vigorous magnetic stirring. The Cu₂TeNWs could be obtained. After crystallization, the products was centrifuged and washed several times with absolute alcohol and double distilled water, and dried in vacuum at 60 °C.

Templating Synthesis of Carbon Nanofiber Hydrogels.

The fabrication of CNF by use the TeNWs as the templates was based on the method that described previously ²⁷. In a typical experiment, 16 mL of the TeNWs solution that prepared above was precipitated by 30 mL of acetone and collected by centrifuging in 6000 rpm. Then, the product was dispersed into 1200 mL glucose solution which contained 80 g glucose with vigorous magnetic stirring at room temperature. After that, the above solution was maintained at 180 oC for 18h using an autoclave with a total volume of 1600 mL resulted in monolithic gel-like product. Finally, the sample could be taken out directly from the Teflon container and soaked successively in ethanol (12 h × 3 times), acidic H₂O₂ solution (HCl: H₂O₂:H₂O = 2:5:23, v/v, 12 h), and double distilled water (12 h × 3 times) at room temperature for removing the impurities and TeNWs templates.

Instruments. X-ray powder diffraction (XRD) patterns were obtained from a Japan RigakuDMax-γA rotation anode X-ray diffractometer equipped with graphite monochromatized

Cu-K radiation ($\lambda = 1.54178 \text{ \AA}$). Scanning electron microscope (SEM) images were taken with a Zeiss Supra 40 scanning electron microscope at an acceleration voltage of 5 kV. Transmission electron microscope (TEM) and high-resolution transmission electron microscope (HRTEM) observations were performed on JEOL-2010F with an acceleration voltage of 200 kV. UV-vis spectra were recorded on UV-2501PC/2550 at room temperature (Shimadzu Corp., Japan).