ReSe₂ Nanotubes Synthesized From Sacrificial Templates

David B. Seley, Manashi Nath, and B. A. Parkinson^{*} Department of Chemistry Colorado State University Fort Collins, CO 80523.

Supporting Information

Experimental Methods

Se nanotubes:

The Se nanotubes were made by a procedure similar to a reported one.^{S1} In a typical experiment, 40 mg (0.5 mmoles) of elemental Se was dissolved in 20 ml of ethylenediamine by magnetic stirring for several hours until complete dissolution occurs. The yellow-amber solution was then added dropwise to 15 ml of deionized water, which was being ultrasonically treated. The appearance of amorphous selenium was immediately apparent as the ethylene diamine/Se/H₂O mixture became reddish. This solution was ultrasonically treated for about 1 hour or until a metallic looking film appeared on the surface of the solution. The solution was then removed from the ultrasonic bath and allowed to sit overnight in the dark. A dark solid film-like material fell to the bottom of the vial. The supernatant solution was removed carefully and the film-like material was washed with ethanol and air-dried. Large quantities of Se nanotubes were observed in the 25 mg (0.3 mmoles) dark film-like product.

*ReSe*₂ nanotubes by solvothermal method:

40 mg of NH₄ReO₄ (0.15 mmoles) was dissolved in 10 ml of absolute ethanol with sonication and the solution was transferred to a Teflon lined acid digestion bomb. Se nanotubes were then added to this solution and stirred for several minutes. The autoclave was then sealed and placed in an oven at 135 °C for a period of 6 days. Upon removal from the oven, the autoclave was allowed to cool down to ambient conditions. A black precipitate was isolated from the product by centrifugation and was washed several times with water and ethanol. Pxrd and SEM of the black precipitate revealed that it contained the ReSe₂ nanotubes. Small amount of the precipitate was dispersed in ethanol and added on formvar coated holey Cu grids for TEM studies.

Reference S1: Chen, Y-T.; Zhang, W.; Zhang, F-B.; Zhang, Z-X.; Zhou, B-J.; Li, H-L. *Mater. Lett.* **2004**, *58*, 2761.



Fig. S1. Raman spectra of Se nanotubes showing the typical Raman shifts characteristic of Se. Inset shows the Raman spectra obtained from pure, bulk Se.



Fig. S2. EDX analysis of ReSe_2 furry nanotubes. The Re:Se atomic ratio is estimated to be approximately 1:2. The standard deviations were 3.84% for Se and 4.25% for Re. The EDX was collected at 15kV.



Fig. S3. DRS spectra of ReSe₂ furry nanotubes. The bandgap is estimated to be ~ 1.07 eV. Inset shows the DRS spectra obtained from crystalline flakes of bulk ReSe₂, where the bandgap is estimated to be ~ 1.1 eV.



Fig. S4. Elemental mapping of Re and Se across the wall of an open nanotube. Re (green) and Se(red) essentially follow each other indicating that the elemental composition across the nanotube wall is homogeneous.



Fig. S5. High-magnification TEM image of a nanotube wall after annealing. The lattice fringes give a spacing of 5.5 Å.