

TiO₂ Nanotube Synthesis

Fluoride etching of Ti metal substrate to form TiO₂ nanotubes

The procedure described in detail below is taken from the following publications. If this procedure is followed, please cite appropriately.

Meekins, B. H.; Kamat, P. V., Got TiO₂ Nanotubes? Lithium Ion Intercalation can Boost Their Photoelectrochemical Performance Three-Fold. *ACS Nano* **2009**, 3, 3437–3446. NDRL 4821 <http://dx.doi.org/10.1021/nn900897r>

Baker, D. R.; Kamat, P. V., Disassembly, Reassembly and Photoelectrochemistry of Etched TiO₂ Nanotubes. *J. Phys. Chem. C* **2009**, 113, 17967-17972. NDRL 4816 <http://dx.doi.org/10.1021/jp9065357>

Baker, D. R.; Kamat, P. V., Photosensitization of TiO₂ Nanostructures with CdS Quantum Dots. Particulate versus Tubular Support Architectures. *Adv. Funct. Mater.* **2009**, 19, 805-811. NDRL 4771 <http://dx.doi.org/10.1002/adfm.200801173>

1. Sonicate Ti foil (0.8cm x 4.0cm) in 2-propanol for 1 hour, store in acetone until use
2. Fill a 3-arm electrochemical cell with electrolyte solution
 - Electrolyte Solution (200mL batch)
 - i. Dissolve 0.6686g NH₄F in 4mL H₂O
 - ii. Add solution to 196mL ethylene glycol
3. Place Ti metal foil (cathode) and a platinum counter electrode (anode) in electrolyte solution.
4. Slowly increase bias voltage between electrodes from 0V to 60V (about 1 V/s ramp)
 - Bubbles should begin to form off of counter electrode
5. Allow etching to take place for desired amount of time (generally 1-2 hours depending on desired nanotube length)
6. Turn off power supply and remove etched Ti foil. Rinse with EtOH
7. Sonicate in EtOH for 2 seconds (further sonication leads to detachment of the nanotubes from the Ti substrate)
8. Dry in air and sinter at 450°C for 3hrs