TiO$_2$ Nanotube Synthesis

Fluoride etching of Ti metal substrate to form TiO$_2$ nanotubes

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


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
   - Electolyte Solution (200mL batch)
     i. Dissolve 0.6686g NH$_4$F in 4mL H$_2$O
     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