Synthesis and characterization of TiO_2 nano-tubes as anodic material in Lithium-ion batteries

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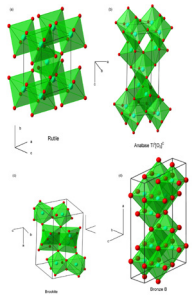
Synthesis of nano-tubular TiO_2

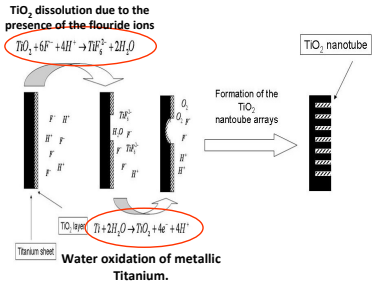
TiO_2 lattice shows more crystalline forms(polymorphs)

Presence and relative amount of different phases greatly influences electrochemical performances

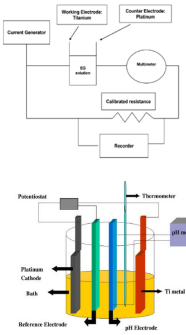
TT main factor investigated in TiO_2 synthesis procedures: phase composition affected



- **Easiness of preparation.**
- **Possibility to control the morphology of the nanotube.**

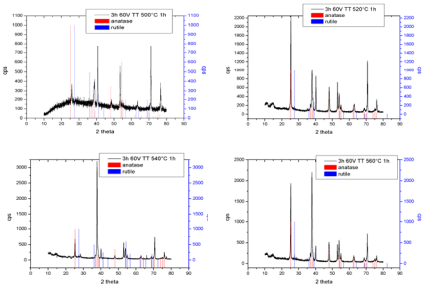


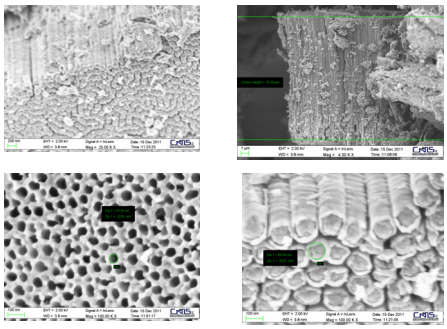
- Substrate cleaning: 3min pickling of Titanium sheets in 1:3 HF/HNO_3 aqueous solution
- Pre-anodizing treatment: surface oxidation via 1mA/cm^2 galvanostatic treatment in 1M KOH aqueous solution
- Anodizing treatment: 3h anodization in NH_4F/H_2O/EG solution at 60V
- Pre-heat treatment: sample vacuum drying
- Heat treatment: calcination in air at 500-600°C(heating rate 1°C/min)

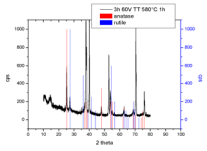


Structural and morfological characterization of the TiO_2 nanotube

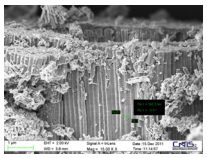
- Presence, relative amount and lattice crystallization of TiO_2 polymorphs vary with calcination temperature(TT)
- No rutile phase observed for TT< 540°C; trace amounts at TT= 540°C
- Higher TT, desto higher amounts of rutile present
- No substantial changes in phase composition after ageing(exposure to air)



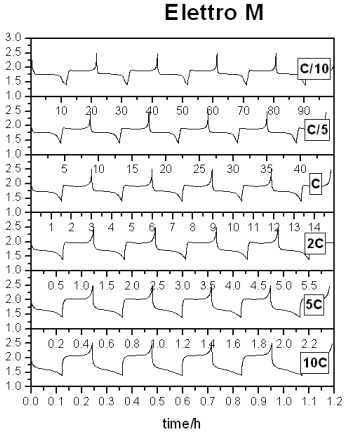


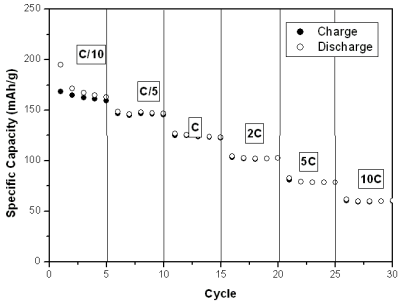


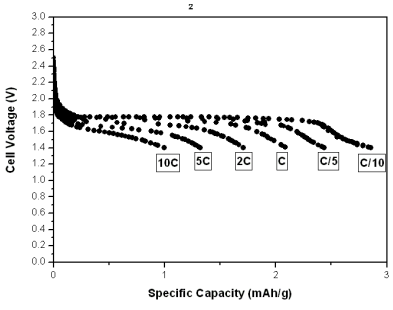
- The nanotube length and the porous diameter is strictly related to the anodization time.
- The voltage and fluoride\water composition ratio influence the rate of the nanotube growth.
- The duration and temperature of the heat treatment determine the crystal structure of nanotube.



Electrochemical behavior of TiO_2







Mura et al., Electrochimica Acta 54 (2009) pp.3794-3798.
Mura et al., Electrochimica Acta 55 (2010) pp. 2246-2251.