SYNTHESIS & SUPPORT OF TANTALUM (V) OXIDE FOR INSULATION & PHOTOCATALYTIC APPLICATIONS

Nicholas Muga Ndiege
Department of Chemistry
University of Illinois at Urbana-Champaign, 2008
Richard I. Masel, Advisor

Tantalum (v) oxide is a material that is gaining popularity due to its wide range of application. Ta_2O_5 is appealing for high temperature insulation, photocatalysis, sensory of trace gas quantities, capacitance properties and bone implants just to name a few.

Ta_2O_5 is appealing for high temperature insulation applications because of its high index of refraction, refractory nature, and negligible absorbance in the infrared region. It is a prime candidate for fabrication of photonic bandgap materials that could be used as thin film insulators for miniature devices/reactors. The challenge faced in the realization of such materials is the synthesis of crack-free Ta_2O_5 films whose thickness is on the order of a quarter wavelength of the incident infrared radiation. Fabrication of most transition metal oxide films of over 1μm thickness is plagued by very low growth rates, extensive film cracking, buckling and even catastrophic delamination.

Ta_2O_5 possesses an energy band gap that allows for photoexcitation by ultra violet wavelengths of radiation. It is increasingly becoming a material of great interest in the quest for materials that can efficiently be employed for water purification and energy production via water splitting. Ta_2O_5's conduction band is more cathodic of the O_2/O_2^- and H^+/H_2 redox couples whereas its valence band is more anodic of the $OH^\bullet/OH^-$ and $OH^\bullet/H_2O$ redox couples to ΔE levels that allow for band gap tuning without shifting to

deep UV excitation. This provides an overpotential that facilitates generation of $OH^{\bullet}$ and O_2^- that partake in reactions at greater efficiencies than the unmodified forms of the material in use thus far for pollutant degradation in aqueous environments.

Nanoscale Ta_2O_5 supported on various substrates also offers potential for enhanced reaction rates in photocatalysis and sensory applications. Photocatalytic applications are primarily targeted towards deactivation of organic contaminants, whereas gas detection is primarily focused on H_2, H_2S and NO_2 detection. The enhanced reactivities are due to quantum confinement and substrate-Ta_2O_5 interface effects. Candidate materials include Ta_2O_5 nanoflowers on planar silicon/SiO_2, Ta_2O_5 nanoparticles decorated onto SiO_2 nanoparticles/nanowires, Ta_2O_5 on carbon nanotubes.

This dissertation discusses synthesis techniques of various morphologies of Ta_2O_5 and how these structures are supported on various substrate materials to form composite structures tailored towards the functions introduced above.

A binder assisted solgel synthesis is used to generate thick, dense and crack free Ta_2O_5 films. This technique can be tailored to vary the film microstructure from a fractal type morphology, a packed sphere morphology, a spheroidal morphology to a very dense/glassy microstructure. The technique has also been extended to decorate Ta_2O_5 nanoparticles onto SiO_2 nanospheres. This decoration facilitates valence band tuning of the Ta_2O_5, which coupled with nitrogen doping allows for conduction band tuning as well. Nitrogen doping was done via ammonia flow over Ta_2O_5 under heated conditions and also via solid state mixture of Ta_2O_5 and urea followed by heating within a closed vessel. The nitrogen doped Ta_2O_5 also show photocatalytic activity under visible radiation.

Ta_2O_5 nanoparticles synthesized via the aforementioned solgel technique have also been supported on multiwalled carbon nanotubes and are applicable for enhanced sensory applications.

Ta_2O_5 flowers were also grown on conductive silicon substrate via metallorganic chemical vapour deposition. Substrate heating was done by resistively heating the Si substrate. Continuous heating during deposition generated a dense oxide film. The flower like microstructure was obtained by a pulsed heating of the substrate during deposition. This type of morphology is applicable for porous bed catalytic applications due to its inherent high surface area.

Microwave assisted chemical vapor deposition (MACVD) is also discussed as a new technique for generating dielectric films using inexpensive equipment at deposition rates much higher than conventional growth techniques. A domestic microwave oven was employed in the growth of 50 nm Ta_2O_5 films that are observed to be crystalline and carbon free without any post deposition treatment. Film growth was conducted at ambient conditions with growth rates observed to be in the order of 0.5 μm per second. MACVD was also employed in growth of 110 mm thick Ta_2O_5 films that are dense, stable and crack free.

MACVD was also employed in the growth of silicon films and silicon wires with diameters varying from 20 nm to 3 μm. Silicon films were grown on planar indium tin oxide coated glass substrate material. Silicon nanowires were grown on similar substrate material with the exception that the substrates were decorated with SnO_2 nanoparticles prior to nanowire growth. The nanowires are applicable as low cost catalyst support for the aforementioned solgel derived Ta_2O_5 nanoparticles.