

# Synthesis of nanocomposite metallic oxides in a doped low-pressure flame reactor

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## Abstract

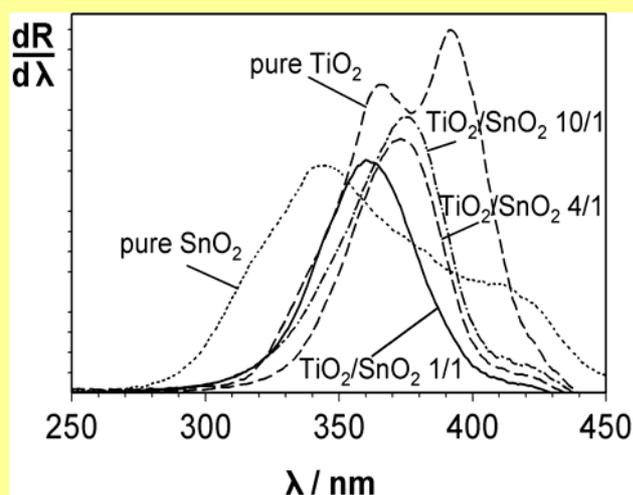
The importance of nanosized material in many contemporary applications is already known. However special properties such as superparamagnetism, semi-conducting and photocatalysis are unique to single metallic oxide. Recent advances in information technology, catalysis, and medicine often require materials that possess a combination of two or more of these properties. Nanocomposite materials present a possibility of having several unique properties within a compact material system. Modern synthesis techniques such as chemical vapour deposition and sol-gel process, becomes seemingly complicated when more than one component is involved. This has necessitated the need to have a synthesis process that is reliable and uncomplicated.

In this study, a  $H_2/O_2/Ar$  flame is presented as a possibility for synthesis of  $TiO_2$  and  $Fe_2O_3$  based nanocomposites. The kinetics of nanocomposite flame synthesis begins with thermal dissociation of the metalorganic precursors due to the high flame temperature. Free radicals resulting from the  $H_2$  oxidation eventually react with precursor material to form primary particles. Growth occurs by collision between primary particles followed by coalescence by either viscous flow mechanism for liquid particles, or by solid state diffusion if the particles are in the solid phase [1]. The final primary particle size depends on the temperature-time history as functions of the surface growth rate and residence time.

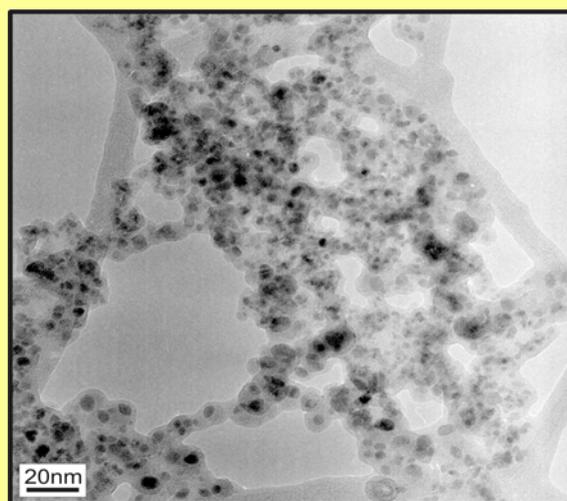
$TiO_2-SnO_2$  mixed oxide nanoparticles  $5\text{ nm} \leq d_p \leq 12\text{ nm}$  were synthesized in a low pressure premixed flame with Titanium tetra iso-propoxide (TTIP) and Tetramethyltin (TMT) as precursors [2]. The effect of varying TTIP and TMT concentrations on the energy band gap of the mixed oxide was investigated. BET, XRD, TEM and UV-VIS were employed to effectively characterize this composite system. It was observed that the particles have a spherical morphology but show strong agglomeration. XRD did not indicate separate peaks of  $TiO_2$  and  $SnO_2$ , hence indicating the absence of a segregated system. New lattice parameters calculated from the XRD indicates varying composition of Ti and Sn within the mixed oxide. It was further deduced that

a shift tendency in the energy band gap reflects the ration between  $\text{TiO}_2$  and  $\text{SnO}_2$  in the structure. Hence this system can be used to tune the energy band gap of  $\text{TiO}_2$ .

Silica embedded iron oxide nanoparticles were also synthesized in a low pressure premix flame using Iron-pentacarbonyl (IPC) and tetramethylsilane (TMS) as precursors. TEM investigation shows that  $\gamma\text{-Fe}_2\text{O}_3$  particles ranging between 3-7 nm were successfully embedded in a surrounding silica matrix of between 9-13 nm [3]. The effects of pressure, concentration, and flow coordinate on the composite size were investigated while keeping flame conditions constant. A silica peak and crystalline peaks corresponding to either  $\gamma\text{-Fe}_2\text{O}_3$  (Maghemite) or  $\text{Fe}_3\text{O}_4$  (magnetite) were observed from XRD Mössbauer spectroscopy was used to confirm the presences of only single phase maghemite. Magnetic properties of this composite system were studied using SQUID. Initial measurement of the magnetization reveals that the particles are superparamagnetic at room temperature. This is due to the segregation of the  $\gamma\text{-Fe}_2\text{O}_3$  core which tends to behave like single magnetic domain particles.



**UV-VIS of nanosized materials**



**TEM micrographs of  $\gamma\text{-Fe}_2\text{O}_3$ /silica particles**

References:

- 1) S.H. Ehrman, S.K. Friedlander, M.R. Zachariah, J.Mater.Res, Vol.14, No.12 (Dec 1999) 4551-4561
- 2) P. Ifeacho, H. Wiggers, P. Roth , Combustion symposium (2004) submitted
- 3) C. Janzen, J. Knipping, B. Rellinghaus, P. Roth, J of Nanoparticle Res 5,(2003) 589-596