Evolution of Nanocrystallinity in Periodic Mesoporous Anatase thin Films

Sung Yeun Choi[a], Marc Mamak[b], Scott Speakman[c], Naveen Chopra[d], Geoffrey A. Ozin[a], [a]*Department of Chmistry, University of Toronto.* [b]*Center for Nanostructure Imaging (CNI) University of Toronto, On., Canada.* [c]*Oak Ridge National Laboratory (ORNL), TN, USA.* [d]*Xerox Research Centre of Canada,On. Canada.* E-mail: gozin@chem.utoronto.ca

Within the last few years, many periodic mesostructured forms of titania denoted meso-TiO_2 have been produced based upon the self-assembly method. Besides the usual benefits of the self-assembly method including high surface area and uniform pore size and shape, the crystallinity and crystallite size of the anatase composing the channel walls of meso-TiO_2 are an equally important factor since potential applications rely upon the intrinsic properties of titania governed by the extent and nature of its crystalline phase. Although crystallite growth, during the film calcination step, within the mesostructured titania framework should be considered the critical step in the formation of meso-TiO_2 thin films, the issue of crystallite growth has yet to be identified as a major determining factor with respect to the properties of meso-TiO_2 thin films and their applicability to electroactive and photoactive devices.

Herein we report the first kinetic study of the intrachannel wall phase-transition of amorphous titania to nanocrystalline anatase for periodic mesoporous titania thin films, monitored by time-resolved *in-situ* high temperature X-ray diffraction (HTXRD).[1] Structural transformations associated with the phase transition are further probed by high-resolution scanning electron microscopy (HRSEM) and transmission electron microscopy (HRTEM). The model found to be most consistent with the kinetic data involves 1-D diffusion controlled growth of nanocrystalline anatase within the spatial confines of the channel walls of the mesostructure. The observation of anisotropic, rod-shaped anatase nanocrystals preferentially aligned along the channel axis implies that the framework of the liquid crystal templated mesostructure guides the crystal growth.

[1] Choi S.Y., Mamak M., Speakman S., Chopra N., Ozin G.A., S*mall*, 2005, **1**, 226.

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