

Template Based Strategies for the Fabrication of  
Macroporous Silicate Films  
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Nanostructured materials are the subject of intense research because of potential applications in the areas of chemical sensors, photonic materials, catalytic supports, and solid-state electrochemical devices. One general procedure used to prepare mesoporous or macroporous materials involves the use of “templates” or “site-directing” agents. In this approach a polymeric network is assembled around a suitable template molecule or structure. Upon removal of the template, diffusional pathways and/or microcavities with a specific size, shape, and/or chemical functionality remain in the crosslinked host.

In this work, polystyrene microspheres have been used as the templating entities to fabricate macroporous silicate films with controllable pore size and geometry. This procedure involves (1) preparation of the silica sol using standard sol-gel techniques, (2) doping of the sol with sulfate stabilized polystyrene microspheres of diameter 100 – 1000 nm, (3) casting of the silica sol on a conducting substrate (i.e., glassy carbon, ITO) to form a thin film, and (4) removal of the spheres by soaking the film in either toluene or chloroform. The size, number density, and distribution of pores produced in a dense silicate host can be varied by judiciously choosing the sol-gel processing conditions and template structure and size. The macroporous materials thus produced have been thoroughly examined and characterized via a combination of electrochemical, microscopic, and spectroscopic techniques.

Atomic force microscopy (AFM) reveals that the cavities are formed in the film after template removal. The shape of the cavities mimic the shape of the lower half of the polystyrene particle. When relatively low amounts of surfactant are added to the sol prior to spin casting, the cavities are randomly scattered across the surface of the substrate. In contrast, 2-D ordered arrays of cavities are produced when the concentration of surfactant is relatively high. It is believed that the surfactant wets the glassy carbon substrate and provides a more hydrophilic surface for the template to adhere to.

The inner diameter of the cavity can be tailored by changing the concentration of the silica in the sol. When the concentration of silica is small, the films are thin and only a relatively small portion of the polystyrene particle is coated with silica. This yields cavities that are not very deep, not very wide, and have thin walls. In contrast, at high concentrations of silica in the sol are used, more than half the polystyrene template is coated thus producing cavities that are deeper and have thicker walls. Further manipulation of the cavity shape, depth, and distance between them can be achieved through variations in the size of the template doped into the sol.

In this presentation, these results as well as other approaches to molecular templating and the formation of nanostructured materials will be presented and discussed.