272d Investigation of the Curing Kinetics in Dip Coated Surfactant / Sol-Gel Silica Films

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When tetraethoxysilane (silica precursor), surfactant, water, acid and ethanol are combined and dip coated under appropriate conditions, we can easily create films with well-defined mesoporous structures. During this process, the silica precursor is hydrolyzed, and undergoes polycondensation while co-assembling with surfactant aggregates. The result is a film with long-range order that mimics the long-range order of a lyotropic liquid crystal, but with rigid silica walls. Removal of the templates (surfactant) creates mesopores which replicate the surfactant mesophase previously present. Both pore size and morphology can be influenced by the choice of surfactant and the processing conditions. Control of the size, shape, and orientation of the pores in ceramic thin films will make them useful in diverse applications, including membrane based separations, models for heterogeneous catalysis, low dielectric constant materials, nanoparticle/wire arrays, and sensor components.

In this work, we investigate the kinetics of silica condensation in surfactant templated, sol-gel dip coated films. Our goal is to understand the kinetics of this process to gain insight into the key factors governing structure evolution, both before and after the film has been deposited. We use transmission Fourier Transformed Infrared spectroscopy (FTIR) and Attenuated Total Reflection FTIR as our major characterization tools. Consistent with the observations of several researchers, we will show that silica condensation actually continues to occur at a measurable rate for a considerable period (between minutes and hours) after the film has been deposited. During this period, the structure of dip coated sol-gel films can be modulated by changing the drying conditions or by bringing the film into contact with functional surfaces. We will describe the influence of key process variables on this post-deposition period, including the age and degree of reaction of the precursor sol, the presence of different types of surfactants (nonionic or cationic), the humidity of the atmosphere present during curing, and the thickness of the coating. We will comment on how these results compare to what is known about homogeneous sol-gel polymerization kinetics.