From Membranes to Superconductors: Functional Mesostructured Materials Enabled by In-Situ and High-Throughput SAXS

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The ability to precisely control the properties of materials using atomic structure developed over the last 50 years directly led to revolutionary materials from semiconductors to superalloys. This remarkable control is directly derived from the availability of advanced characterization techniques, e.g. XRD, XAS, neutron diffraction, and HR-TEM. Complex mesoscale structures such as those prepared by block copolymer (BCP) self-assembly may prove to be a powerful tool for tuning the electronic, transport, and other properties of mesostructured materials. Such materials are expected to have substantial impact on demanding applications at the materials-energy-water nexus (e.g. fuel cells, reverse osmosis membranes, and superconducting metamaterials), but few efforts have systematically probed such properties, largely due to a lack of robust characterization tools. This talk will explore three examples of integrated syntheses enabled by new *in situ* and high-throughput scattering techniques

The development of high-performance, low-cost catalysts for electrochemical reactions will form the foundation of future transportation and grid-scale energy devices to meet the needs of a growing population. In the first part, a robust platform for *in situ* SAXS/WAXS/XAS during annealing up to 1200 C in reactive gases is developed and used to enable the first synthesis of ordered mesoporous nitrides from commercially available BCPs. The resulting materials are expected to have improved performance as electrochemical catalysts and are an important first step toward a robust platform for the exploration of the impact of mesoscale ordered structures on correlated electron systems, e.g. superconductors.

To explore these properties more completely, single mesophase crystals are needed to isolate bulk behavior from that at grain boundaries and probe orientation-dependent effects. In the second part, a method for growth of macroscopic (14 mm^2) single mesocrystals of the double gyroid phase in BCP-preceramic polymer blends is demonstrated and optimized using a new single crystal SAXS imaging technique. The resulting maps of crystal orientation and coherent scattering domain size, obtained using approximately 20,000 individual microbeam SAXS patterns, are used to propose a growth mechanism for these crystals. The BCPs used can be readily converted into porous ceramics which exhibit single crystal-like fracture behavior and can be infiltrated with metals to give well-controlled porous superconductors. These superconductors have properties strikingly different from their bulk counterparts including an order of magnitude increase in the critical magnetic field.

Finally, I will outline recent efforts to probe salt localization and pore structures in polyamide membranes. These complex materials are widely used in desalination, yet fundamental questions regarding their pore structure and transport mechanisms remain unanswered, particularly in the presence of salts as encountered in operation. Using a combination of *in situ* GIWAXS, resonant soft x-ray scattering, and SANS, we probe the structural and chemical features of these highly complex materials and provide mechanistic insight.