Small-Angle Scattering and Imaging

Microstructure (or nanostructure) control is the key to property and performance optimization for many materials that provide critical functions in advanced technological systems. Achieving this control requires quantitative characterization of microstructure over a contiguous size range from the nanoscale to the mesoscale. This project addresses this need by combining small-angle x-ray and neutron scattering methods with x-ray imaging capabilities to provide a microstructure-characterization-based metrology that links materials processing to performance.

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New technological materials, such as those for fuel cells or thermal barrier coatings, present increasingly difficult microstructure characterization challenges. At the Advanced Photon Source (APS), as part of UNICAT (university/national laboratory/industry collaborative access team), we have made significant advances in the development of x-ray micro-tomography (XMT) and in the application of small-angle x-ray scattering (SAXS) to meet these challenges. The unique capability of the NIST ultra-SAXS (USAXS) instrument to measure absolute intensity now makes it a reference for several SAXS instruments at the APS and positions us for possible development of a SAXS standard reference material. In a continuing effort to more rapidly develop and to expand our suite of synchrotron-radiation-based x-ray characterization tools, we have developed a partnership with the X-ray Operations and Research (XOR) Division at the APS. The XMT capability at XOR serves as a proving ground for the development of nanoscale resolution XMT at UNICAT, and their capability for high-energy SAXS (HESAXS) provides increased sample penetration and spatial resolution that complement the NIST USAXS capability. Several examples of our research efforts are highlighted below.

A multi-component anisotropic-void model for USAXS analyses was used to quantify the complex microstructure of electron beam physical vapor deposited (EBPVD) thermal barrier coatings. These coatings are used in advanced gas turbine engines and are of particular interest to companies such as GE and Siemens–Westinghouse. Measurements of coatings before and after heat treatment reveal that, due to thermal expansion mismatch, a 2 % by volume population of cracks oriented normal to the substrate forms. When combined with thermal and mechanical property measurements performed by collaborators at SUNY Stony Brook, new insight has been gained into coating failure modes.

Electron-beam directed vapor deposited (EB-DVD) coatings are proposed as an alternative to conventional electron beam physical vapor-deposited coatings for high temperature thermal barrier applications. In collaboration with the University of Virginia, HESAXS has been combined with wide-angle x-ray diffraction to study EB-DVD coatings. The same sample volume was measured with both techniques at a spatial resolution of 20 µm. The measurements show that preferred crystallographic growth axes dominate near the top of the coating while void anisotropy, which plays a key role in coating durability and performance, varies throughout the thickness.

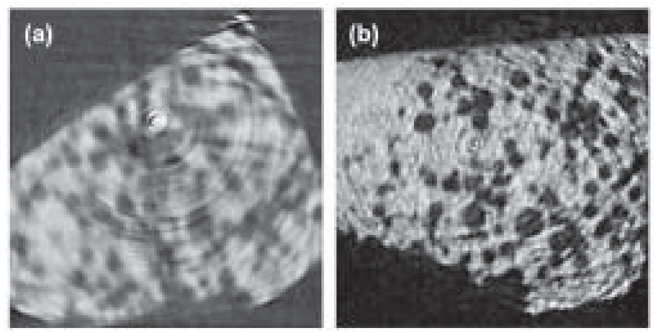

Figure 1: *XMT single-slice images through a SOFC anode layer at (a) 17 keV and (b) 27 keV x-ray energy. The 27 keV image exhibits a strong contrast between the Ni and yttria-stabilized zirconia components.*

In collaboration with the University of Utah, the structure of solid oxide fuel cell (SOFC) materials has been investigated. Measurements of pore structure and phase content have been made within each layer using USAXS, HESAXS (at 5 µm spatial resolution) and XMT. At a spatial resolution of 1.35 µm, XMT shows in 3D, for the first time, both the pore and segregated-phase size and interconnectivity actually present in the membrane layers (see Figure 1). Use of different x-ray energies allows the image contrast for different features to be optimized. This research supports current efforts to join major Department of Energy initiatives in fuel cell research.

Contributors and Collaborators

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