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Exploiting Microreactors for Correlative Studies of Working Catalysts With Electrons And X-Rays

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The development of "closed cell" atmospheric electron microscopy methods have had a substantial impact on the characterization of materials in reactive gaseous environments and increased our understanding of how morphological and chemical changes occur in nanostructured heterogeneous catalysts.[1] These closed-cell microreactors allow the provision of multiple gas streams and temperatures in excess of 1000°C to be applied to the catalysts, concomitant with atomic-resolution imaging and spectroscopy.

In recent years, our group has extended the utility of this microreactor approach to include the incorporation of a range of excitations beyond electrons, including the provision of micro-focused x-rays [2] and infrared radiation.[3]. These studies have demonstrated the ability to understand dynamic changes that can occur in nanoparticle systems during oxidation and reduction [2], to explore how surface speciation affects catalytic activity [3], and determining the structure and associated chemistry of the active phase in bimetallic systems [4].

In this presentation, I will review the entire suite of available correlative techniques and their application to specific problems in heterogeneous catalysis. Particular emphasis will be placed on the latest developments concerning (1) the use of electron energy loss spectroscopy, especially results obtained from the K2-IS detector incorporated into Penn's NEOARM instrument and (2) the development of electron pair distribution function analysis. These two capabilities fill in the final gaps in the overall characterization suite, adding spectroscopic information from light elements and providing sophisticated diffraction analyses.[5]. By providing imaging, spectroscopy and diffraction with x-ray, photons and electrons it becomes possible to describe the structural and chemical features of both catalyst and support over a wide range of descriptors, allowing a deeper understanding of the complex interrelationships that control their reactivity and selectivity.

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