In situ combinatorial characterization of catalyst nanoparticles under reactive environment

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In recent years the environmental transmission scanning electron microscope (ESTEM) has been successfully employed to elucidate the structural and chemical changes occurring in the catalyst nanoparticles under reactive environments. While atomic-resolution images and the combination of high spatial and energy resolution is ideally suited to distinguish between active and inactive catalyst particles and identify active surfaces for gas adsorption, unambiguous data can only obtained from the area under observation. This lack of statistical information available from TEM measurements is generally compensated for by using other, ensemble measurement techniques such as x-ray or neutron diffraction, x-ray photoelectron spectroscopy, infrared spectroscopy, Raman spectroscopy etc. However, it is almost impossible to create identical experimental conditions in two separate instruments to make measurements that can be directly compared. Moreover, ambiguities in ESTEM studies may arise from the unknown effects of the incident electron beam and uncertainty of the sample temperature. We have designed and built a unique platform that allows us to concurrently measure atomic-scale and micro-scale changes occurring in samples subjected to identical reactive environmental conditions by incorporating a Raman Spectrometer on the ESTEM. We have used this correlative microscopy platform i) to measure the temperature from 60 µm² area using Raman shifts, ii) to investigate light/matter interactions iii) as a heating source, iii) for concurrent optical and electron spectroscopy such as cathodoluminescence, EELS and Raman. Details of the design, function, and capabilities will be illustrated with results obtained from in situ combinatorial measurements.