This presentation will deal with the development of new materials and operando methods for energy conversion and storage with emphasis on fuel cells and battery materials and technologies. The presentation will begin with a brief overview of the methods employed. Particular emphasis will be placed on the use of X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), X-ray microscopy and tomography and transmission electron microscopy (TEM) under active potential control. The utility of these methods will be illustrated by selected examples including electrocatalysts for the oxygen reduction reaction (ORR), hydrogen oxidation reaction (HOR) and spectroscopic studies of Li/S and Li/Se batteries and Li metal deposition and dendritic growth. The presentation will conclude with an assessment of future directions.