High-throughput structure/function screening of materials with multiple spectroscopic techniques

Moniek Tromp
University of Southampton, School of Chemistry, Highfield, Southampton, SO17 1BJ, United Kingdom, m.tromp@soton.ac.uk

Combinatorial or high throughput (HTP) synthesis is increasingly applied to generate libraries of inorganic materials. In addition to parallel synthesis techniques obtaining large amounts of new materials in the quest for new, interesting, ones, combinatorial or HTP screening is growing rapidly in its application to optimize the composition of materials to obtain specific physical or chemical properties and (fundamentally) understand their origin.

X-ray diffraction methods (XRD) are now well-established for HTP characterisation of crystalline bulk phases. In comparison, HTP X-ray absorption fine structure (XAFS) spectroscopy is a virtually unexplored technique. XAFS is readily applied in situ and with high time-resolution, providing opportunities for a wide range of HTP applications. With suitable ancillary techniques, both the structure and the function of a wide range of systems can thus be dynamically monitored under process conditions.

At Southampton, we have developed an environmental chamber[1], allowing *in situ* studies on arrays of samples while X-ray absorption fine structure spectroscopy, Raman spectroscopy, X-ray diffraction and/or mass spectrometry can be applied simultaneously to characterize the system under process conditions in a time-resolved manner. The chamber accommodates a diverse range of samples from surface science to materials chemistry to heterogeneous catalysis. Data acquisition and data logging software is developed to handle large quantities of divers but related information. New data logging, processing and analysis procedures and programs are developed which will allow fast structure-function relationships characterization.

In this exemplar study we apply the multi-technique HTP screening approach to understand and optimise/fine-tune the properties of mixed metal oxides (e.g. BiMoVOx $Bi_2V_{1-x}Me_xO_{5.5-\delta}$ materials) suitable as inorganic pigments as well as low temperature oxidation catalysts.[1,2] The chemical and physical properties of these oxides are strongly dependent on their structure and the location and distribution of cations in their structure. The structure and cation distribution in itself is strongly dependent on the pre-treatment procedure, with the temperature being the most important factor. Arrays of these multiple cation oxides (generated efficiently using robotic methodologies) are studied and analysed for metal ion distributions. HTP methodologies are required in order to delineate local structural features rapidly.

1. M. Tromp, et al., AIP, CP882 (858), 2007.
2. S. Russu, et al., AIP, CP882 (535), 2007.