MS37 Advances in Structure determination of new materials by multi-technique approach including imaging techniques

MS37-01

Investigating reaction intermediates under realistic conditions with diffraction, imaging and spectroscopy

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Abstract

Reaction intermediates are complex to characterize as they are often unstable outside of their reaction environment, are metastable, amorphous or poorly crystalline. However, despite these difficulties they are important to the understanding and control of a materials growth mechanism. To investigate these materials, characterization techniques carried out under close to reaction conditions are required, such as in-line, in situ or cryo characterization techniques. These techniques allow access to the material under realistic or simulated conditions, either in solution or under reactive gases. Several techniques such as synchrotron X-ray scattering and diffraction, Raman spectroscopy, infrared spectroscopy and ultraviolet-visible spectroscopy can be used in solution due to the large penetration depth of the technique, where-as transmission electron microscopy (TEM) can be utilized for in-depth analysis, trying to preserve the materials while transferring them into the TEM or generating that form in situ.

In this talk I will detail several examples of reaction intermediates studied by combining electron microscopy techniques with a range of spectroscopic and diffraction techniques under realistic reaction conditions. In particular the materials explored are metastable and form during a reaction, either during growth or under reactive conditions such as corrosion or catalysis. The examples focused on will be materials studied during growth and corrosion to investigate reaction intermediates and reaction kinetics. Nanoparticle intermediates studied during solution synthesis showed the formation of amorphous materials, nanocluster formation and also crystalline structures that rapidly broke down when removed from solution. A range of solution based techniques including cryo electron microscopy were utilized to determine the initial forms of these materials and their growth mechanisms.[1-3] For metallic nanoparticles and their oxides and carbides, reaction dynamics and corrosion mechanisms were explored. A particular example that will be discussed is on the oxidation of iron carbides to iron oxide, utilizing high resolution scanning transmission electron microscopy and electron energy loss spectroscopy to determine the intermediate structures that form and the fate of the carbon in the structure.[4] Finally the growth and passivation of copper indium gallium selenide (CIGSe) thin films will be discussed focusing on reactions at the boundaries between grains and their passivation with alkali metals.

References

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