Characterization Techniques for Nickel, Manganese and Cobalt containing Gradient Composition Materials

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A logical step forward in cathode development is gradient and core-shell particle compositions. Gradient and core-shell materials can provide improved performance by protecting compositions that have undesired surface interactions with a coating of a material that has superior stability at its surface [1]. It is important to verify that the desired gradients and/or core-shell particles have been properly synthesized, and to determine how the gradients change during lithiation and heat treatment.

Verification of gradient compositions is often accomplished by energy dispersive x-ray spectroscopy (EDS) or similar techniques that sample a cross section of a particle [2], this process samples only a single particle at a time. Although X-Ray Diffraction (XRD) is commonly used to determine compositions in homogeneous samples, little effort has been done in analyzing XRD patterns obtained for gradient materials. Here, we use a simple model assuming spherical particles with a constant growth rate to fit XRD patterns of Ni0.15Mn0.85(OH)2 with a continuous composition gradient.

This study explores various gradients of Ni, Mn and/or Co hydroxide precursors and their respective lithiated oxides. The hydroxide precursors were developed using a constantly stirring tank reactor (CSTR) [3] with a computer controller adjusting the flow rate of inputted metal sulfate solutions to develop the gradient compositions. Figure 1B shows that the solution added to the tank was initially entirely manganese sulfate, while at the end it was entirely nickel sulfate.

Figure 1A shows that the XRD pattern changes dramatically as reaction proceeds. This is caused by the presence of primary particles with a wide range of lattice constant values over the entire Ni0.15Mn0.85(OH)2 solid solution. The wide range of the lattice constants is particularly evident in the 55-65° range where two peaks, the 110 and 111 broaden to the point that they overlap to give a small apparent peak at 56° (Figure 1A, 9-10 h).

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