

## An *in situ* XRD study of the synthesis of LiFePO<sub>4</sub>

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In recent years, LiFePO<sub>4</sub> has emerged as a prime Li-ion battery cathode material, due largely to its potential cheapness, high theoretical capacity (170 mAh/g) and the flatness of its discharge/charge curve at ca. 3.5V vs. Li/Li<sup>+</sup> [1,2]. The only serious problem has been its exceptionally low electronic conductivity, but the adverse effects of this can now be combated successfully through the use of various types of carbon coating [3-5], in combination with lower particle-size.

In this context, we have followed the solid-state synthesis of LiFePO<sub>4</sub> by *in situ* XRD to probe the effects of sintering-temperature, -time, etc. on the properties of the final product. Our starting materials were: FeC<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O, Li<sub>2</sub>O<sub>3</sub> and NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>. After mixing and grinding, the mixture was first decomposed in a tube-furnace under a N<sub>2</sub>-gas flow. It was then packed into capillaries in an Ar-filled glovebox, and XRD diffractograms collected for a range of temperature profiles, using a STOE HT furnace attachment mounted on a STOE position-sensitive X-ray powder diffractometer. The effects on the resulting LiFePO<sub>4</sub> were monitored in terms of both its physical and electrochemical properties.

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